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South Florida Ecosystem Assessment: Phase I/II (Technical Report) – Everglades Stressor Interactions: Hydropatterns, Eutrophication, Habitat Alteration, and Mercury Contamination

Monitoring for Adaptive Management: Implications for Ecosystem Restoration

September 30, 2001

The South Florida Ecosystem Assessment was conducted by the United States Environmental Protection Agency Region 4 in partnership with the Florida International University Southeast Environmental Research Center, FTN Associates Ltd., and Battelle Marine Sciences Laboratory. Additional cooperating agencies include the National Park Service, the United States Fish and Wildlife Service, the United States Geological Survey, The Florida Department of Environmental Protection, the South Florida Water Management District and the Florida Fish and Wildlife Conservation Commission. The Miccosukee Tribe of Indians of Florida and the Seminole Tribe of Indians allowed sampling to take place on their federal reservations within the Everglades.

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SOUTH FLORIDA ECOSYSTEM ASSESSMENT:

Phase I/II (Technical Report) – Everglades Stressor Interactions: Hydropatterns, Eutrophication, Habitat Alteration, and Mercury Contamination

by

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LIST OF ABBREVIATIONS

ac-ft = acre feet
ACME = Aquatic Cycling of Mercury in the Florida Everglades
AFDW = ash free dry weight
APTMD = Air, Pesticides, and Toxics Management Division
BAF = bioaccumulation factor
BASS = Bioaccumulation and Aquatic System Simulator Model
BMP = Best Management Practices
CERP = Comprehensive Everglades Restoration Program
cm = centimeter
culm = the stem of a grass-like plant
EAA = Everglades Agricultural Area
EMAP = Environmental Monitoring and Assessment Program
ENP = Everglades National Park
FDEP = Florida Department of Environmental Protection
FIU SERC = Florida International University--Southeastern Environmental Research Center
Floc = particulate organic matter suspended in the water column above the soil surface
GIS = geographic information system
Hg = mercury
Hg0 = elemental mercury
HgII = inorganic mercury
kg/yr = kilogram per year
Kg = kilogram
km = kilometer
LOX = Loxahatchee National Wildlife Refuge
m = meter
MeHg = methylmercury
mg/kg = parts per million (ppm)
mg/L = milligram per liter (ppm)
mi = mile
NAWQA = National Water-Quality Assessment Program
NERL – Athens = National Exposure Research Laboratory - Athens, Ga
ng/L = nanogram per liter (ppt)
NHEERL - RTP = National Health and Environmental Exposure Research Laboratory - Research Triangle Park, NC
NPS = National Park Service
ORC = Office of Regional Counsel
% OM = percent organic matter
peri = periphyton
ppb = parts per billion (ug/L)
ppm = parts per million (mg/L)
ppt = parts per trillion (ng/L)
PS = periphyton (soil)
PU = periphyton (*Utricularia*)
REMAP = Regional Environmental Monitoring and Assessment Program
S^{2-} = sulfide
SESD = Science and Ecosystem Support Division
SFWMD = South Florida Water Management District
SFWMM = South Florida Water Management Model
SRB = sulfate reducing bacteria
STAs = Storm water Treatment Areas
TP = total phosphorus
ug/kg = parts per billion (ppb)
ug/m^2 = microgram per meter squared
ug/L = microgram per liter (ppb)
uMol/hr = micromoles per hour
UPGMA = unweighted pair group mean averaging
US EPA = United States Environmental Protection Agency
USGS = United States Geological Survey
WCA = Water Conservation Area

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1.0 INTRODUCTION

1.1 Purpose

The US Environmental Protection Agency (EPA) Region 4 South Florida Ecosystem Assessment Project is an innovative, long-term research, monitoring and assessment project. Phase I of the Project was conducted from 1992 through 1998 and was discussed in two previous reports (Stober et al. 1996, 1998). Phase II sampling was conducted during 1999. This report describes the Phase II Project results.

The ultimate Project goal is to provide decision-makers with sound, scientific information for environmental decisions related to the South Florida Everglade ecosystem restoration.

Project purposes are to:

1. Contribute to the South Florida Interagency Everglades Restoration Program by monitoring the status and trends in the condition of the South Florida Everglades ecosystem.

2. Assess the effects and potential risks of mercury contamination on the South Florida ecosystem, specifically the processes and pathways from inorganic mercury to prey fish mercury contamination.

3. Assess the effects and potential risks of other environmental stressors such as hydropattern modification, habitat alteration, and total phosphorus loading, as well as their interaction with mercury contamination.

4. Improve monitoring design and environmental reporting for the South Florida ecosystem, and

5. Provide interim information on a regular basis that contributes to environmental decisions on Everglades restoration issues.

1.2 EPA Region 4 South Florida Ecosystem Assessment Project

The EPA Region 4 South Florida Ecosystem Assessment Project - Phase II continued the Phase I monitoring that was initiated in 1994, but modified the monitoring design, indicators, and media that were sampled. These modifications are described in subsequent chapters of this report. The Phase II Project maintained the focus on relative risk and was guided by the same seven policy-relevant assessment questions raised in Phase I:

1. **Magnitude** - What is the magnitude of the problem(s) in the South Florida ecosystem?

2. **Extent** - What is the extent of the problem(s)?

3. **Trend** - Is the problem(s) getting better, worse, or staying the same?

4. **Cause** - What factors are associated with or contribute to the problem(s)?

5. **Source** - What are the source(s) and what is the contribution and importance of each source to the problem(s)?

6. **Risk** - What are the risks to different ecological systems and species from the stressors or factors causing the problem(s)?

7. **Solutions** - What management alternatives are available to ameliorate or eliminate the problem(s)?

These policy-relevant questions are applicable to each major issue identified by the Science Subgroup as impacting the South Florida ecosystem (i.e., hydropattern modification, mercury contamination, eutrophication, habitat alteration, and exotic species invasions). Conceptual models and testable hypotheses were developed around these key issues and policy-relevant questions.

Unlike other studies in support of the South Florida Everglades restoration effort, the South Florida Ecosystem Assessment Project is unique in a number of ways:

1. Scale - The South Florida Ecosystem Assessment Project is a multimedia study being conducted on over 5,800 km^2 (2,250 mi^2) in South Florida extending from the Everglades Agricultural Area (EAA) in the north to the Florida Bay in the south (Figure 1.1). Few ecological studies have been conducted at this scale. This large-scale, multimedia approach provides the ability to assess patterns in individual resources throughout the whole Everglades ecosystem and the interactions among these resources and patterns.

2. Study Design - This Project uses a unique probability-based, statistical survey design to select sample locations throughout the Everglades marsh and canals. This sampling design permits the development of unbiased population estimates of resource condition with known confidence. Furthermore, this design permits spatial analyses and associations that provide insight into fundamental relationships among observed ecological effects and multiple stressors.

3. Risk Based Approach - The South Florida Ecosystem Assessment Project evaluates multiple impacts and stressors on the Everglades ecosystem simultaneously using an ecological risk based approach. By using a risk based approach, issues that are critical to the restoration efforts and the interaction among these issues and stressors can be identified for decision makers.

4. Complementary Interagency Efforts - This Project was designed to address critical policy-relevant questions complementary to the approaches being used by other agencies and studies. This Project contributes directly to the Interagency Task Force on South Florida Ecosystem Restoration and provides the benchmark against which restoration practices can be evaluated. This Project has been conducted by U S Environmental Protection Agency, Region 4, Science and Ecosystem Support Division in partnership with Florida International University Southeast Environmental Research Center, FTN Associates Ltd., and Battelle Marine Sciences Laboratory. Additional cooperating agencies include the US National Park Service Everglades National Park, US Fish & Wildlife Service, US Geological Survey, The Florida Department of Environmental Protection, the South Florida Water Management District and the Florida Fish & Wildlife Conservation Commission.

1.3 Mercury Contamination

The Project was originally designed to specifically address the mercury contamination problem that exists in South Florida while also providing information useful for restoration. The Phase I report, in conjunction with the largemouth bass monitoring by Florida Fish and Wildlife Consservation Commission and the wading bird studies being conducted by the University of Florida, clearly documented the extent of the mercury contamination problem in South Florida. One of the major conclusions of Phase I was that there were no apparent point sources of mercury to South Florida (Stober et al. 1998). Atmospheric deposition was contributing about 35-40 times the mercury coming from the EAA discharges (Stober et al., 1998). In addition, there was no “smoking gun”, clearly indicating that local emission sources were responsible for the atmospheric contributions.

Phase I sampling found that only 4 surface water samples out of over 500 collected had total mercury concentrations that exceeded the current mercury water quality standard (i.e., 12 ng/L); yet over 2 million acres are under fish consumption advisories because of mercury. Revised mercury standards are needed, but these standards need to consider both the mercury form (methylmercury rather than total mercury) and bioaccumulation/biomagnification of mercury through the food chain.

The Phase I Project also indicated the greatest risk to the South Florida ecosystem is to assume that the environmental issues (e.g., hydropattern modification, eutrophication, mercury contamination) are independent and can be managed independently. Mercury contamination is influenced by hydropattern, nutrient status, habitat and the specific biological organisms. The interactions of these additional factors must be considered when proposing and evaluating various management practices for restoring the Everglades. Additional insight into the interactions among these environmental problems is provided in this Phase II report.

1.4 Everglades Ecosystem Restoration

Many of the problems with declining Everglades ecosystem health revolve around four interrelated factors: water quantity, quality, timing, and distribution. Consequently, the major goal of restoration is to deliver the right amount of water that is clean enough to the right places and at the right time. Since water largely defined the natural system, it is expected that the natural system will respond to water management improvements. The Water Resources Development Acts of 1992 and 1996 directed the U. S. Army Corps of Engineers to review the Central and Southern Florida Project and develop a comprehensive plan to restore and preserve south Florida's natural ecosystem, while providing for other water-related needs of the region including urban and agricultural water supply and flood protection. The result is the Comprehensive Everglades Restoration Plan (CERP, or the Plan). The development of the Plan was led by the Army Corps of Engineers and the South Florida Water Management District and was accomplished by a team of more than 100 ecologists, hydrologists, engineers and other professionals from over 30 federal, state, tribal, and local agencies. The Plan includes: water storage areas; man-made wetlands to treat urban or agricultural runoff; wastewater reuse; extensive aquifer storage and recovery; water management operational changes; and structural changes to improve how and when water is delivered to the Everglades, including removal of some canals or levees that prevent natural overland sheet flow. The entire Plan is projected to take over 30 years and cost about $8 billion to implement, with the cost split equally by Florida and the federal government. If nothing is done, the health of the Everglades will continue to decline, water quality will degrade further, some plant and animal populations will become stressed further, water shortages for urban and agricultural users will increase, and the ability to

protect people and their property from flooding will be compromised. (USAE & SFWMD, 1998, 1999).

A series of ecological success criteria have been defined that will gauge the success of ecosystem restoration efforts. Some example ecosystem restoration success indicators: (Science subgroup 1997)

Problem	**Success Indicators**
Water Management	Reinstate system-wide natural hydropatterns and sheet flow
Habitat Alteration	Increased spatial extent of habitat and wildlife corridors
Eutrophication	Reduced phosphorus loading
Mercury Contamination	Reduced top carnivore mercury body burden
Endangered Species	Recover of threatened endangered species
Soil Loss	Restore natural soil formation processes and rates

1.5 Long-term Monitoring and Adaptive Assessment

The attention and funding devoted toward Everglades ecosystem restoration is unprecedented. It is imperative that ecosystem health is assessed in a cost-effective, quantitative manner such that baseline, pre-restoration conditions are documented. Such an assessment identifies resource restoration needs. Continued assessment allows one to determine the effectiveness of restoration efforts. A major defining feature of the Everglades is its large spatial area; hence, to monitor restoration it is essential to determine the area of the current Everglades that is subject to various human impacts. This study employs a scientifically rigorous way of accomplishing this, using probability-based sampling. This project uses a statistical, probability-based sampling strategy to select sites for sampling. Samples were collected from the freshwater wetland portion of the Everglades and Big Cypress. The study area extended from Lake Okeechobee southward to the mangrove fringe on Florida Bay and from the ridge along the urban, eastern coast westward into Big Cypress National Preserve. This study permits a consistent, synoptic look at indicators of the ecological condition in the entire freshwater canal and marsh system in South Florida from Lake Okeechobee to the Florida mangrove system. This large-scale perspective is critical to understanding the impacts of different factors (such as phosphorus and mercury distributions throughout the canals and marsh, habitat alteration, or

hydropattern modification) on the entire system rather than at individual locations or in small areas. Looking only at isolated sites in any given area and extrapolating to South Florida can give a distorted perspective. This study is unique to South Florida: its extensive spatial coverage and sampling intensity are unprecedented; its probability-based sampling approach permits quantitative statements about ecosystem health.

A key advantage to this study's probability-based statistical sampling approach is that it allows one to estimate, with known confidence and without bias, the current status and extent of indicators for the condition of ecological resources (Thornton et al., 1994; Stevens, 1997). Indicators of pollutant exposure and habitat condition also can be used to identify associations between human-induced stresses and ecological condition. This design has been reviewed by the National Academy of Sciences, and the USEPA has applied it to lakes, rivers, streams, wetlands, estuaries, forests, arid ecosystems and agro-ecosystems throughout the United States. (Olsen et al. 1999; EPA 1995).

Parameters measured at each site can be used to answer questions on multiple environmental problems threatening the Everglades, including water management, soil loss, eutrophication, habitat alteration and mercury contamination.

- Water management (e.g., water depth at all sites)
- Water quality and eutrophication (e.g., phosphorus concentrations in water and soil, cattail distribution)
- Habitat alteration (e.g., wet prairie, sawgrass marsh plant community distribution)
- Mercury contamination (e.g., mercury in water, soil, algae, and preyfish)

Specific questions related to Everglades restoration goals that this study answers include:

- How much of the marsh or canal system has a total phosphorus concentration greater than 50 parts per billion, the Phase I phosphorus control goal, or 10 parts per billion, the approximate natural marsh background concentration?
- How much of the marsh is dominated by sawgrass? Wet prairie? Cattail?
- How much of the marsh still has the natural oligotrophic periphyton mat?
- How much of the marsh area is dry, and where?

- How much of the marsh has prey fish with mercury levels that present increased risk to top predators such as wading birds?

- What water quality conditions are associated with marsh zones of high mercury bioaccumulation ?

Data from this study have been used by a variety of scientists and agencies for many purposes:

- Input to models that predict the Everglades' response to water management changes.

- Input to models that predict periphyton or vegetation changes in response to phosphorus enrichment.

- Developing empirical models in order to better understand interrelationships among mercury, phosphorus, sulfur and carbon.

- Developing water quality standards to protect human health and fish and wildlife. Understanding the relative risks of phosphorus and mercury.

Monitoring is important for determining ecosystem condition, identifying threats, and evaluating environmental restoration efforts. As portions of the Comprehensive Everglades Restoration Plan are implemented, a system-wide monitoring program is needed. Monitoring objectives include:

- Documenting status and trends;
- Determining baseline variability;
- Detecting responses to management actions; and
- Improving the understanding of cause and effect relationships.

This South Florida Ecosystem Assessment Project provides such information system-wide for the freshwater Everglades marsh as of 1995-1996 and 1999. All reports and data for the study are available on the internet at <http://www.epa.gov/region4/sesd/sesdpub.html>.

1.6 Report Organization

This report builds on the Phase I report (Stober et al., 1998) and provides additional information on the status of the South Florida ecosystem. The report is organized into nine chapters and 12 appendices. Chapter 2 discusses the Phase II design modifications made to the

Phase I statistical survey frame. Revised and new Phase II materials and methods are described in Chapter 3. Chapters 4 and 5 present information on macrophyte and periphyton distributions, respectively. Plants integrate physicochemical factors and provide insight into large-scale responses to these factors. One of the major attributes of the large-scale survey design and sampling is that it can describe landscape patterns for the entire South Florida Everglades ecosystem, which are patterns that were not apparent before this Project was conducted. These landscape patterns are described in Chapter 6. Based on these landscape patterns and complementary process studies being conducted in other programs by other agencies, a series of conceptual models were developed to describe the interactions among constituents. These conceptual models formed the foundation for a series of structural equations used to perform path analysis to assess mercury risks. These conceptual models and path analyses are described in Chapter 7. Chapter 8 provides the foundation for a probabilistic ecological risk assessment of mercury that was conducted on wading birds in the Everglades ecosystem by the South Florida Water Management District. These analyses couple the physical-chemical factors with mercury bioaccumulation through the food chain to the mosquitofish. Mosquitofish is a prey species for sunfish and largemouth bass, which are major components of wading bird diets. The probabilistic ecological risk assessment model starts with sunfish and largemouth bass and models the upper portion of the food chain to wading birds. Linking both these studies provides a management tool that can be used to assess the effects of changing water depth, total phosphorus, total mercury and other constituent concentrations on mercury methylation and bioaccumulation through the food chain to largemouth bass and ultimately on wading birds. Management implications from this Phase II Project are presented in Chapter 9. Future directions for this Project are discussed in Chapter 10. References cited are contained in Chapter 11. There are a number of appendices that contain additional information on the Quality Assurance Project Plan (QAPP), methods, materials, and other Project features.

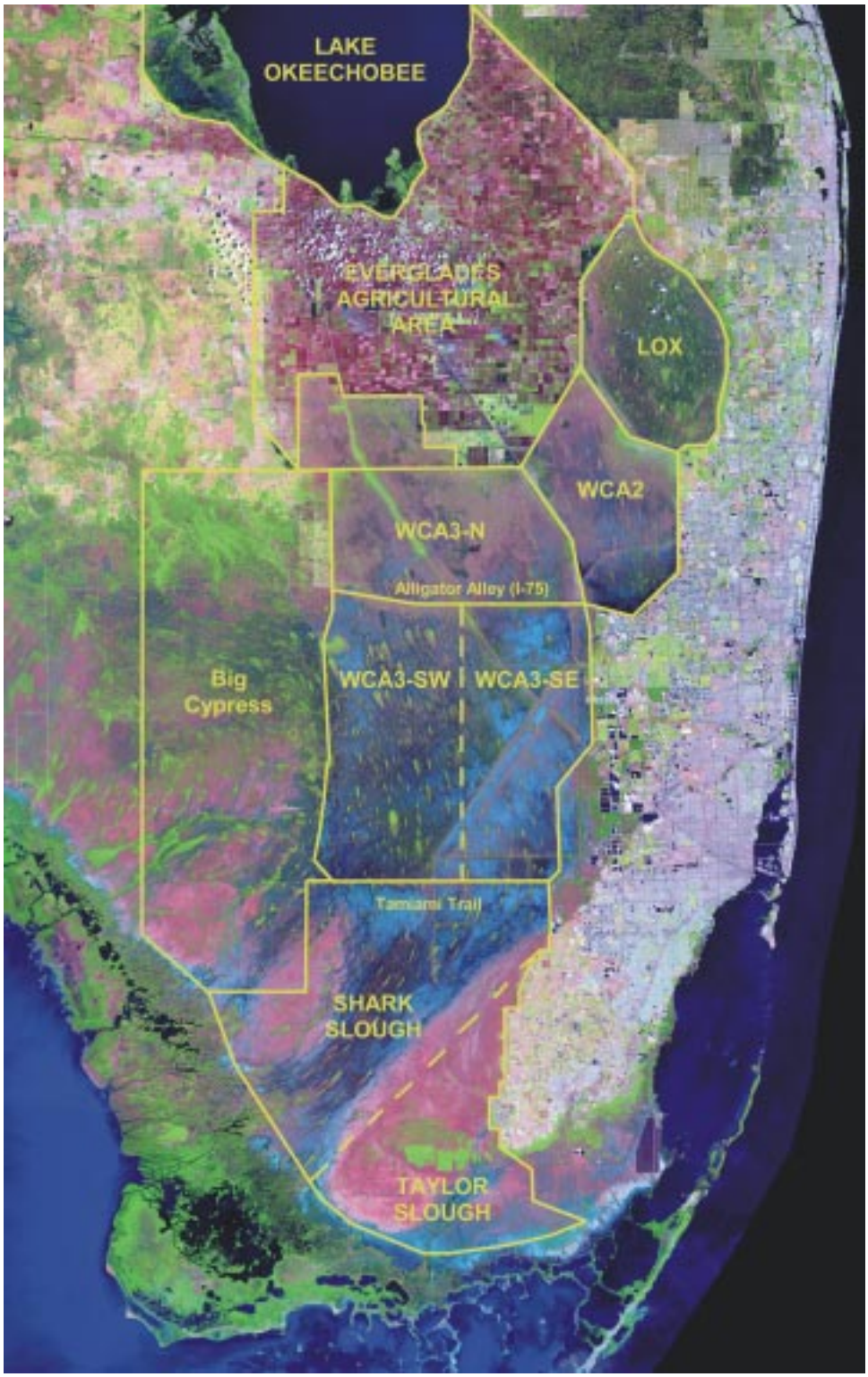


Figure 1.1. USGS satellite image of South Florida: light areas on the east indicate urban areas; dark areas in the center are the remnant Everglades; the red area at the top is the Everglades Agricultural Area and the western part of the image is Big Cypress National Preserve.

2.0 STUDY DESIGN

The EPA Region 4 South Florida Ecosystem Assessment Project Phase II design was modified from the Phase I design to improve the efficiency of sampling and focus on the portion of the system at greatest risk from mercury contamination. These modifications are presented in this chapter.

2.1 Phase I Design

The Phase I design, described in the South Florida Ecosystem Assessment Phase I Report, selected sites in both the canals and the marsh, including Big Cypress National Preserve for sampling (Stober et al. 1998). The Phase I design was a probability-based synoptic survey that systematically sampled randomly selected sites during both the dry and wet seasons. The formal name for the Phase I design is a random tessellation stratified (RTS) design (Bellhouse 1977, Overton and Stehman 1993, Stevens 1997). Because it is based on probability sampling, the RTS design samples the marsh resource in direct proportion to the occurrence of its attributes (e.g., soil type, phosphorus concentration, water mercury concentrations, plant species, etc.). This means that population estimates can be made of the proportion of the area or total acreage, with known confidence, for any particular system attribute. For example, the proportion of the marsh area that has soil total phosphorus concentrations exceeding some threshold value, or the total area of the marsh that has fish mercury concentrations exceeding the USFWS proposed predator prey mercury criteria can be estimated with known confidence. In addition, the systematic approach to sampling (i.e., a grid) captures the spatial or landscape context of the sites. The design is particularly suited for spatially displaying data and detecting large scale patterns in the ecosystem if these patterns are present, because there is an almost uniform distribution of sites distributed on the landscape.

During Phase I, approximately 50 canal sites and 125 marsh sites were sampled during each season. Canals were sampled from 1993 to 1995 while the marsh was sampled from 1995 to 1996. A total of about 200 canal sites and 500 marsh sites were sampled during the Phase I project.

The Phase I assessment determined that fish in the canals and Big Cypress National Preserve had lower mercury concentrations than fish collected from the South Florida marsh

sites. The Phase II design, therefore, was modified to increase its effectiveness in sampling areas with elevated fish mercury contamination as well as other areas important to the Everglades Restoration programs.

2.2 Phase II Design Modifications

The Phase II sampling design focused on the area from Lake Okeechobee in the North to Florida Bay in the South and from the edge of the urban area on the East to the edge of Big Cypress National Preserve on the West (Figure 1.1). This included Loxahatchee National Wildlife Refuge, Water Conservation Areas 2 and 3 and Everglades National Park (Figure 1.1). A probability-based synoptic survey also was used to systematically select 126 marsh sites for sampling during both the dry (May - Cycle 4) and wet (September - Cycle 5) seasons in 1999 (Figure 2.1). The canals and the Big Cypress National Preserve were not sampled during Phase II. The total number of marsh sampling sites for both Phases I and II are shown in Figure 2.2

An analysis of variance (ANOVA) was conducted for selected, critical variables to compare the proportion of the total variance accounted by within versus among site variance (Table 2.1). A goal established in the EPA Office of Research and Development Environmental Monitoring and Assessment Program (EMAP) was to have within site variance be about 10% of the among site variance so that large scale constituent gradients and patterns could be adequately detected. During Phase I, for example, five mosquitofish were collected and individually analyzed from each site. The ANOVA indicated that the within site variance was about 12%. It was estimated that collecting two additional fish at each site during Phase II should reduce the within site variance to about 9% of the among site variance. Because mosquitofish mercury concentration was a critical variable in the study, two additional fish were collected at each Phase II site so that seven individual fish were analyzed at each Phase II site. This resulted in the within site variance being 9% of the among site variance in Phase II. The within site compared to among site variance in other constituents are also shown in Table 2.1. During Phase II, within versus among site variance was greater than 10% of total phosphorus and total nitrogen in water, and total phosphorus and methylmercury in soil. Because Phase I sampling occurred over two years and Phase II sampling occurred over only one year, the Phase II sample size is approximately one half that of the Phase I sample size, which does influence the variance

estimates.

The Phase II sampling design also incorporated vegetation (i.e., macrophyte) transect sampling in conjunction with the probability sites. The purpose of the vegetation transect sampling was to assess the relationship between plant responses and large scale gradients in nutrient concentrations and hydrologic variables. Estimating plant biomass was not a Project objective. The plant transect sampling is described in Chapter 3, Methods and Materials.

Table 2.1. Comparison of within versus among site variance.

	1996			1999		
Constituent	**Within***	**Among***	**% W/A+**	**Within***	**Among***	**% W/A+**
Surface Water						
Total Phosphorus	26	916	3	23	102	22
Total Nitrogen	0.02	1.09	2	0.67	4.99	13
TOC	1.45	173	1	5.72	205	3
Sulfate	0.28	569	<0.1	0.20	196	0.1
Total Mercury	0.53	3.83	14	0.11	1.74	6
Methyl Mercury	0.02	0.34	6	.03	0.57	5
Soil						
Total Mercury	498	9,518	5	280	8,132	3
Total Phosphorus	3,211	51,651	6	34,022	64,074	53
Methyl Mercury	0.14	1.24	11	6.75	27.7	24
AFDW	22	2,143	1	49	848	6
Mosquito Fish						
Total Mercury	9,673	80,126	12	4,441	47,000	9

* Mean square error (MSE)
+ W/A = within MSE/among MSE

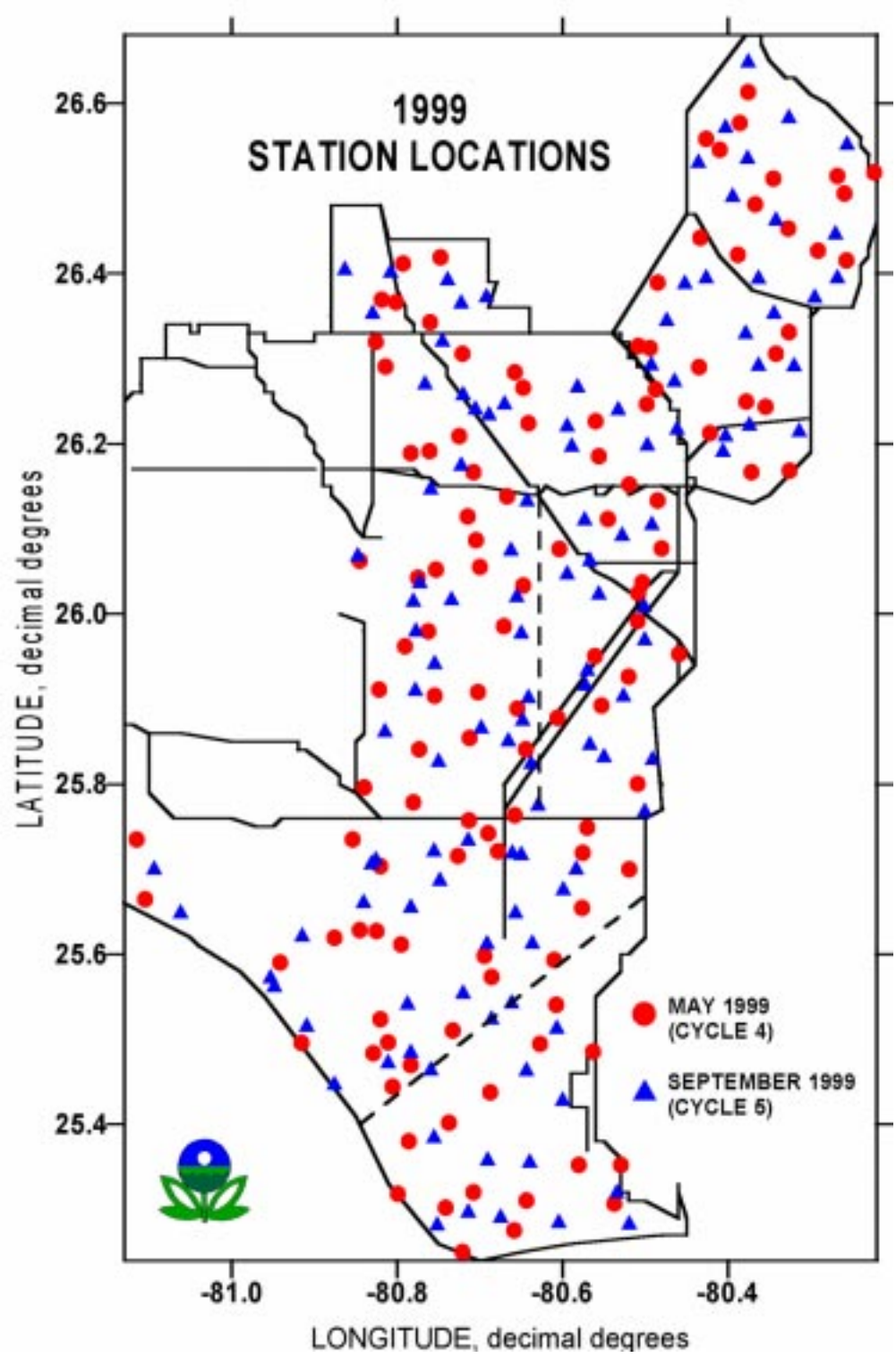


Figure 2.1. Site locations for May (Cycle 4) and September (Cycle 5) 1999 sampling.

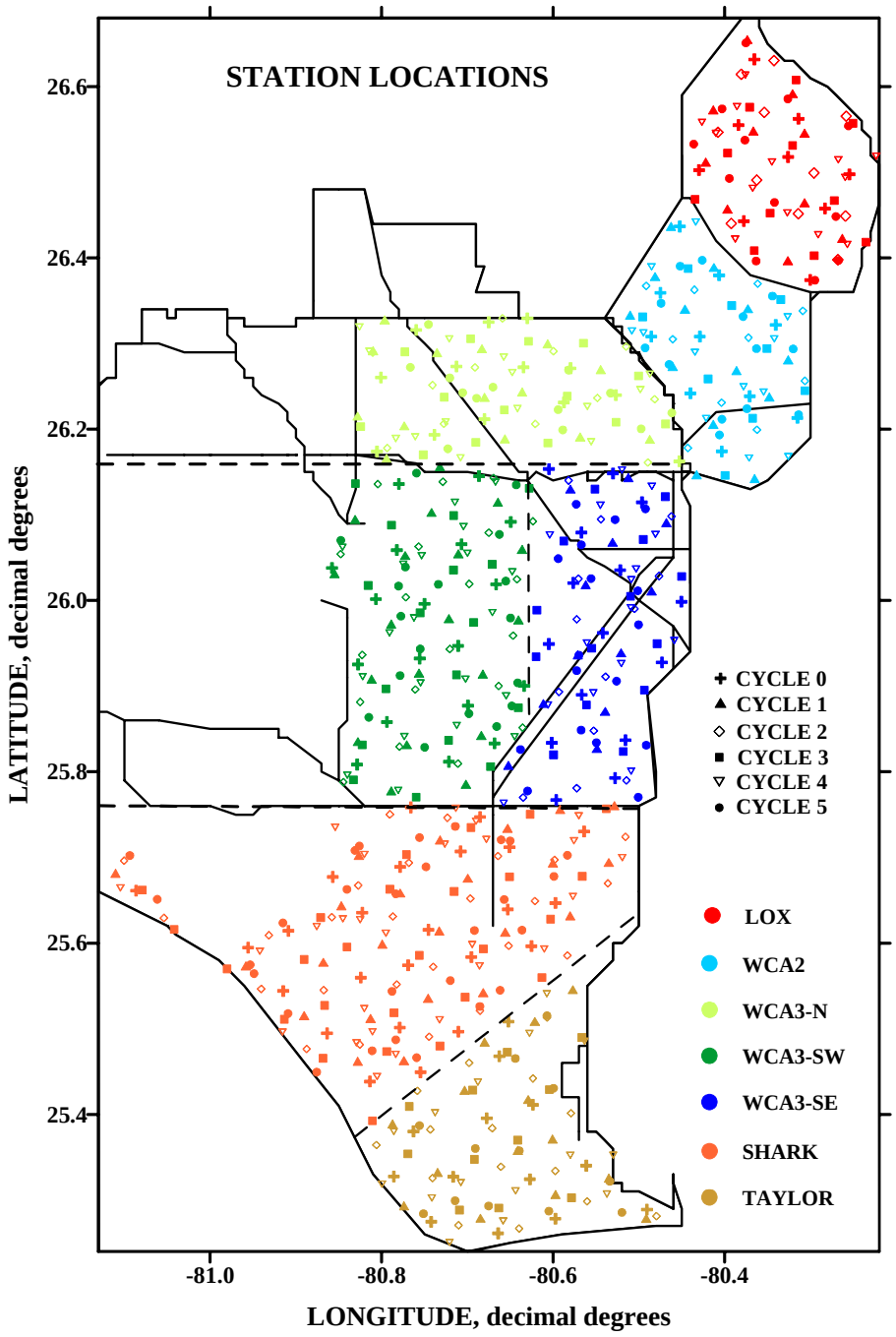


Figure 2.2. 750 sampling sites are located in over 2 million marsh acres.

3.0 MATERIALS AND METHODS

3.1 Field Procedures and Methods

3.1.1 Logistical Rationale and Needs

The large spatial scale of this study required field sampling with helicopters (Bell Jet Ranger four-passenger with floats) to make the sampling as efficient and rapid as possible. All stations were located with handheld global positioning system (GPS) equipment (Trimble® Pathfinder Pro) corrected to within ±1 m. A synoptic sample over the entire marsh ecosystem proceeding from south to north was completed in an 8-day period (125 stations). Phase II biogeochemical sampling was conducted near the end of the spring dry season, from May 4 to 13, 1999 (Cycle 4) and during the summer wet season from September 22 to 30, 1999 (Cycle 5). The sampling time at each site was approximately one hour with helicopter shut down during sampling. The marsh grid was sampled with two helicopters and a team of 6 samplers (each team worked two days on and one day off). A two-person sampling team was used in each aircraft and all gear and sample containers were designed to fit in the fourth seat and the aft storage compartment. For safety, each flight was monitored with ENP Dispatch.

3.1.2 Sampling Apparatus and Procedures

The variables and media sampled during Phase II are listed in Tables 3.1 and 3.2. Table 3.1 is a list of critical variables while Table 3.2 is a list of non-critical variables. The basis for the selection of critical vs. noncritical measurements was that measurements thought to have regulatory implications or usage for setting regulatory criteria/standards were considered "critical" measurements. All other measurements collected during the project were considered noncritical and useable for research purposes.

3.1.2.1 Surface Water

A Hydrolab Scout 2 Water Quality Data System™ (Hydrolab) was used to measure water temperature (°C), DO (mg/L), specific conductivity (mS/cm), pH (su), and Eh (mV). The data sonde was suspended in the water column at mid-depth and the DO probe was allowed to equilibrate prior to recording the readings on the field data sheet. The Hydrolab calibration

procedure defined in the EPA Science and Ecosystem Support Division (SESD) Standard Operating Procedure (SOP) was executed in the laboratory prior to entering and leaving the field each day.

The sampling procedure was initiated at each station by placing a 2-L polypropylene bottle in the vacuum chamber and filling the bottle about 25% full. This water was used to rinse the bottle and discarded. The bottle was then filled to 75% capacity and aliquots were poured into appropriate containers for TP, TOC, TN, turbidity, alkaline phosphatase (AP), anions, (SO_4^{2-}, Cl^-, Br^-, F^-, NO_2^-, NO_3^-), ortho-P, and TKN. The critical and noncritical variables are listed in Tables 3.1 and 3.2, respectively. The number of 125-ml polypropylene containers filled varied with the number of duplicate stations, laboratory splits and nutrient filtration/preservation methods used for QA/QC. At least 10 % of all samples collected were for QA/QC, including field duplicates, blanks and splits for each media (Tables 3.1 and 3.2).

The development and application of clean mercury sampling methods has been of primary importance in both the Phase I and Phase II projects. A hand-operated vacuum water sampling chamber was developed and used to consistently collect a screened (105 Fm replaceable Nitex screen) ultra trace level water sample. Specifications and pictures of this sampling equipment were presented in Stober et al. (1998). The initial Phase I study found that it was important to prevent the intake of large particulates into the samples. However, the samples were not filtered to permit quantification of total constituent concentration, which can be ecologically significant.

A trace level mercury sample was taken immediately following the collection of water for conventional water quality variables by placing a 2-L Teflon® bottle into the chamber and pumping it full with no headspace. The bottle was filled in about 5 minutes with about 380 mm (15 inches) of vacuum. The bottle was labeled, its number recorded, inserted into a Fisher® ziploc plastic bag and placed in a cooler inside a black plastic bag. The sampling sequence flushed the device twice before each clean low level Hg sample was collected at each station. During this procedure, the operator was gloved with PVC gloves covered with shoulder length polyethylene gloves and clothed in chest waders and/or a flight suit. Water samples were collected near the helicopter at about one foot below the surface when sampling in deep water and at mid-depth when sampling shallow water less than one foot. Water field blanks of Hg free

deionized water were taken into the field with each crew each day. Additional surface water sampling details can be found in Appendix A, Attachment 3.

Sulfide in surface water at each site was sampled with two 60 ml plastic syringes with leur-loc tips connected to a three-way valve. One syringe was previously prepared with zinc acetate/6N sodium hydroxide preservative solution and the other was used to evacuate the air from the system prior to drawing the sample into the syringe with the preservative while holding the sampler underwater.

Sulfide in porewater was sampled with the same double syringe triple valve system with the addition of a hollow stainless steel insertion tool developed to penetrate the soil and facilitate the insertion of a filter (nominal porosity = 60 Fm) (Porex 6810, Interstate Specialty Products) attached to 3.5 mm OD Tygon tube approximately 65 cm in length with a leur-loc attachment to the three-way valve. The insertion tool fitted with a stainless steel push rod was used to insert the filter to a maximum depth of 10 cm into the soft soil. The system was voided by one syringe followed by drawing the sample into the other syringe containing the preservative.

Filtered nutrient samples (NH_4^+, NO_2^-, NO_3^-, PO_4^{2-}) were collected for both surface and porewater by filtering 60 ml of surface water and 30 ml of porewater through a GF/F 0.8 Fm filter attached to a syringe. Additional details regarding design, development, and operation can be found in Appendix A, Attachment 3.

3.1.2.2 Soil Sampling

A stainless steel extension rod graduated in tenths of feet was used to measure the surface water depth and soil depth to bedrock at each station. An in situ Eh probe (Stober et al. 1998) was deployed in the soil at each station near the helicopter and five measurements recorded at soil depths of 2.5 to 20 cm were recorded following a 15-minute equilibration period.

Soil sampling was conducted with a 3-inch diameter clear polycarbonate coring tube (see Stober et al. 1998 for design specifications) to sample the top 10 cm. Three cores were composited per station and placed in a sealed 1-gallon plastic container for transport to the laboratory. During soil sample collection, the slurry (floc) of particulate matter and water captured on top of the soil core was poured off into an Imhoff cone to concentrate the particulate matter which was then placed in a separate 500-ml polyethylene container. In addition, whenever

a layer of periphyton mat was present on the top of the soil core it was separated in the field and placed in a separate container for analysis. Soil samples, therefore, were limited to the material remaining with large roots, rocks and coarse debris removed.

3.1.2.3 Mosquitofish

Mosquitofish (*Gambusia holbrooki*) were collected with a Turtox Indestructable dipnet (800 x 900-mm multifilament nylon net) with a 40-inch wooden handle. The sampler used the net in an aggressive manner in an attempt to capture a complete size range of the fishes in the area near the helicopter. When necessary, both crew members used the same technique to collect the required number of mosquitofish to shorten the time at each sampling station. The fish captured with each swipe of the net were handled with latex gloves and placed in a 5 x 8-inch Fisher® plastic bag and labeled according to station number (place) and documented on the field data sheet. A minimum of 15 individuals was collected at each site, when available, for THg, QA/QC, and stable isotopes analyses. Twenty individuals were collected and preserved in formalin for stomach content analysis. When fish were scarce and extremely hard to catch, the priority samples were for THg and gut analysis. The fish in plastic bags were held on ice in the field and frozen immediately upon return to the FIU laboratory.

3.1.2.4 Mosquitofish Food Habits Data Collection

Mosquitofish were collected for stomach analysis in September 1996 and 1999 and May 1999. Fish were collected from 101 locations in September 1996, 35 locations in May 1999, and 120 locations in September of 1999. The low water conditions of May 1999 limited the spatial extent of aquatic habitat available for fish sampling at that time. Upon collection, the fish were preserved in a jar with 10% formalin and transported to the laboratory. Twelve to 14 specimens were obtained from each collection site for analysis of stomach contents. Additional logistical, planning aids, step-wise sampling protocols, and methods details can be found in Appendix A, Attachment 3.

3.1.2.5 Macrophytes

Macrophyte Census

A census of macrophytes was conducted at all sites previously sampled by the biogeochemistry team. Sampling sites, which had been temporarily flagged with tape marker by the biogeochemistry team, were accessed by helicopter. GPS coordinates and siting of the marker were used to land the helicopter. One hundred twenty sites were sampled from May 12 to May 19, 1999, during Cycle 4, and 120 sites were sampled from September 30 to October 7, 1999, during Cycle 5. Data was collected by a 3-person team that included 2 EPA wetland scientists and 1 FIU botanist or ecologist.

In the spring (Cycle 4) one or two transects were established at each site. The number of transects established depended on the homogeneity of the site. If a single wetland community type was present in the area, only a single transect was sampled. If two communities were present, a second transect was sampled from the other community type. In Cycle 5 if only a single community type was present in the area, 2 transects were sampled from that community, with the second transect established at 90E from the far end of the first transect. A total of 178 transects were sampled in Cycle 4 and 240 transects were sampled in Cycle 5.

Each transect was 10 m long, as defined by a rope marked off in 1-m intervals. Data on species presence and periphyton % cover were taken from five 1-m^2 quadrats laid out at 2-m intervals on alternate sides of the transect. Quadrats consisted of two 0.5 x 1-m rectangles made of PVC pipe that had been filled with styrofoam to maintain flotation. Each rectangle was halved with string. Two rectangles were laid out side-by-side to make a 1-m^2 quadrat subdivided into four 0.25-m^2 quadrats. Data on species presence was recorded from each 0.25-m^2 quadrat, for a total of twenty 0.25-m^2 quadrats per transect. If an unknown species was present in a quadrat, it was recorded with a number, collected, labelled, and brought back to FIU for identification. Specimen nomenclature followed Wunderlin (1998).

At each site, the helicopter GPS coordinates, time, and water depth were recorded. A picture was taken of the first transect and of the site from the air as the helicopter left the site.

Morphometric and Tissue C:N:P Data

Morphometric data were collected on two species to document plant responses to changes in physicochemical conditions in sawgrass and wet prairie communities and to evaluate their potential as indicator species. Sawgrass *(Cladium jamaicense)* and lance-leaf arrowhead *(Sagittaria lancifolia)* were chosen based on their morphological variability and presence across the entire ecosystem. Morphological measurements and tissue nutrient analyses were made on individuals collected from the same sites as plant census and biogeochemical data.

Sawgrass culm number was counted in the third 1-m^2 quadrat of every transect. At sites where sawgrass was present, a single sawgrass plant was collected from each of the five 1-m^2 plant census quadrats and brought back to the lab for morphometric measurements and C:N:P analyses. Each specimen included the main shoot apex, mature leaves and rhizome. At 188 sites (95 in Cycle 4, 94 in Cycle 5) distributed throughout the study area, 5 plants were collected from a single transect. At 52 sites (38 in Cycle 4, 14 in Cycle 5), an additional set of 5 was collected from the second transect (= 10 total plants/site). In the May 1999 sample 8 sites were re-sampled and an additional 5 plants were collected from these sites (= 15 total/site); for 3 of these sites both transects were re-sampled, for an additional 10 plants (= 20 plants/site). A total of 1,129 plants (606 in Cycle 4, 523 in Cycle 5) were sampled.

At sites where it was present, up to 5 plants of *Sagittaria lancifolia* on or adjacent to at least one transect per site were harvested and brought back to the lab for morphometric measurements and C:N:P analyses. The rhizomes, main shoot apex, and attached leaves were harvested. A total of 648 plants (338 during Cycle 4, 305 during Cycle 5) were sampled from 140 transects (76 from Cycle 4, 64 from Cycle 5) at 122 sites (62 in Cycle 4, 60 in Cycle 5) throughout the study area. Fourteen sites in Cycle 4 and four sites in Cycle 5 had plants harvested from more than one transect.

Plant Tissue Mercury Concentrations

The Cycle 4 (May 1999) synoptic sampling also included the collection of five whole leaves from sawgrass and cattail (*Typha domingensis*) plants at every site where each of these species occurred. The whole leaves were placed in ziploc plastic bags for transport to the laboratory for THg and stable isotope analyses.

3.1.2.6 Periphyton

Periphyton cover was estimated to the nearest 10% from each of the five 1-m^2 quadrats established for macrophyte sampling. Periphyton volume was measured from a 8-inch diameter stove-pipe corer used to core the periphyton and water column adjacent to each transect at each site. To measure periphyton volume the periphyton material inside the corer was transferred to a 2,000-ml graduated cylinder with holes drilled in it, the water allowed to drain, then the volume of the remaining material was recorded. A qualitative subsample of this material was removed and later frozen for periphyton constituent and diatom analysis.

Three types of periphyton collected in the field were identified as either floating mat (floating), soil mat (lying on the soil surface), or epiphytic (associated with *Utricularia*). These three designations were most quickly determined in the field and were associated with the field sampling procedures. These designations are not intended to denote ecological significance because the periphyton mat can be found anywhere in the water column depending on stage of growth or time of day. These designations were based on location at the time of field sampling. The floating periphyton mat was sampled with a 3-inch diameter serrated edge cylinder to obtain a comparable surface area and volume collected with the soil core sampler. These samples were placed in a 4-oz. cup for volume to weight ratio analysis. To ensure enough additional sample material was collected for the variety of analyses to be conducted, a 32-oz container was filled in the field with each individual periphyton type available.

3.2 Sample Preparation Procedures and Laboratory Analyses

The measurement parameters and associated analytical methods utilized in Phase II are listed in Table 3.3. Twenty surface water parameters, 11 porewater parameters, 13 soil parameters, four parameters on three types of periphyton, and total mercury in sawgrass, cattails, and mosquitofish were analyzed throughout the South Florida ecosystem. Surface water parameters added in Phase II are indicated by asterisk and include filtered NH_4^+, NO_2^-, NO_3^-, SRP, SO_4^{2-} and unfiltered S^{2-}. All the porewater parameters were added including TP, TN, Br^-, Cl^-, F^-, SO_4^{2-}, S^{2-} and filtered NH_4^+, NO_2^-, NO_3^-, and SRP. Soil parameters added included CH_4 and CO_2. Diatom counts and biomass estimates of periphyton also were included. Total mercury in sawgrass and cattails was measured only during the May 1999 dry cycle.

Atomic fluorescence-based analytical and preparation methods were developed for measuring ultratrace levels of inorganic and organic mercury in environmental (water, soil, sediment, floc) and biological (tissue–fish, periphyton, macrophyte) samples (Jones et al. 1994). For the analysis of total Hg in soil, sediment and fish the samples are digested with concentrated nitric acid in sealed glass ampules, and subsequently autoclaved. Water samples are digested using standard brominating procedures. A Merlin Plus, PS Analytical atomic fluorescence spectrometer (AFS) system equipped with an autosampler, vapor generator, fluorescence detector and a PC based integrator package is used in the determination of total Hg. The determination of organic Hg species in water, without pre-derivitization, involves adsorbent pre-concentration of the organomercurials onto sulfydryl-cotton fiber. The organic Hg compounds are eluted with a small volume of acidic KBr and $CuSO^4$ and extracted into dichloromethane. Sediment, soil and tissue samples are homogenized and the organomercurials first released from the sample by the combined action of acidic KBr and $CuSO^4$ and extracted into dichloromethane. The initial extracts are subjected to thiosulfate clean-up and the organomercury species are isolated as their chloride derivatives by cupric chloride addition and subsequent extraction into a small volume of dichloromethane. Analysis of organic Hg compounds was accomplished by capillary column chromatography coupled with atomic fluorescence detection. Additional refinements of these methods can be found in Cai et al. 1996; Cai et al. 1997; Cai et al. 1997; and Cai et al. 1998. Additional details can be found in Appendix A, Attachment 3.

3.2.1 Water and Pore Water

Acidification of the Hg samples was made the same day following return to the clean laboratory on the Florida International University (FIU) campus, where 5 ml of trace metal grade HCl per 1,000 ml of sample was added to each Hg sample. Water field blanks of Hg free deionized water were taken into the field with each crew each day and analyzed for ultra trace level THg before and after transport to the field. Additional details can be found in Appendix A, Attachment 3.

3.2.2 Soil

In the laboratory, the soil cores were further processed by mixing and removing additional debris before placement in a 500-ml HDPE blender jar. A known amount of deionized water was added to dry soil samples to achieve a slurry. Homogenization of the sample was obtained in 30 to 60 seconds. This was a departure from the Phase I procedures where the soil samples were not blended. The homogenized sample was then poured into multiple 4-oz cups for THg, MeHg, TP, AP, CH_4, CO_2, AFDW, BD, mineral content, SO_4^{2-}, stable isotopes, and QA/QC analyses. Following blending and splitting, all samples were frozen for later analysis except for a set held at room temperature for enzyme and gas analysis.

3.2.3 Floc

The floc sample was blended in the laboratory and split into multiple 4-oz cups for THg, MeHg, AP, CH_4, CO_2, TP, AFDW, BD, mineral content, stable isotopes, and QA/QC analyses. Following blending and splitting, all samples were frozen for later analysis except for a set held at room temperature for enzyme and gas analysis.

3.2.4 Mosquitofish

Individual mosquitofish were dissected and their gut contents removed and separated into six categories: plant matter (pooling algae, vascular plant, and detritus), cladocera, aquatic mites, chironomid larvae (midge larvae), adult midges, and other (primarily spiders, ants, aquatic beetles, and fish). Counts of the number of items in all animal categories were recorded for each mosquitofish, along with their sex and standard length. Males could be identified readily by the presence of a gonopodium, and females were identified by presence of mature ovaries or by standard length exceeding 18-mm. Juveniles were all fish below the 18-mm standard length lacking a gonopodium. The presence or absence of plant matter was recorded for each specimen, and if no food was present this was also noted. All food items for the fish from a single population sample were pooled and the mass of each food category was determined. The sum of these masses provided an estimate of the total mass of food consumed by that sample of fish.

3.2.5 Macrophytes

3.2.5.1 Morphometric and Tissue C:N:P Analyses

Sawgrass plants were measured within 24-hr of being returned to the laboratory. Each plant was washed, roots were removed, and the number of mature, living leaves were recorded. Leaves were considered mature when they had attained the gray-green color and general height of the bulk of the leaves on the plant. Leaves which were more than 1/2 brown were considered dead leaves. Leaf parameters measured for sawgrass were length of the longest mature leaf, taken from its tip to the point of its attachment to the rhizome, and width half-way along the length. Horizontal rhizome diameter was measured just behind the main apex.

In Cycle 5 the rhizome length and fresh weight were also measured and leaves were harvested for C:N:P analysis. Rhizome length was measured from the angle where the apex turned up to the distal cut end of the rhizome. Fresh weight was recorded after plants had been washed, roots and dead material removed, and leaves clipped to 20 cm from the rhizome base. The three most recently matured leaves per rhizome were sampled from each sawgrass plant and dried to ambient temperature and humidity in a dehumidified room for tissue nutrient analysis. A subsample of these leaves was processed for C:N:P analysis. To obtain the subsample, the sample area was subdivided into six latitudinal strata and four to six sites were randomly chosen from these strata for analysis. The five samples from these sites were further dried for 24 hr in an 80EC oven, then ground in a Wiley mill. One mg from each individual plant was bulked to make a site sample for analysis. From two sites, one at the northern end and one at the southern end of the study area, the five individuals per site were analyzed separately in order to examine within-site variation. Samples were analyzed for % C, N, and P by the FIU SERC lab.

For *Sagittaria lancifolia* Cycle 4 plants were stored individually in plastic bags with water and left in the greenhouse. Measurements on these plants were made over the course of two weeks, in contrast to the Cycle 5 plants, which were measured within 24 hr of harvest. Prior to measurement, soil, dead leaves, roots, and all leaves older than the third most recently matured leaf were removed from the plants. In Cycle 4 measurements were made on the three most recently matured leaves, while in Cycle 5, measurements were made only on the most recently matured leaf. Leaf parameters measured for *Sagittaria* were leaf base length (from insertion on the rhizome to where the leaf sheath unites), petiole length (leaf base tip to lamina base), petiole diameter (adaxial to abaxial in the middle of the petiole), lamina width at its broadest, and lamina length. Rhizome parameters measured for *Sagittaria* were rhizome length

from the cut end to where the rhizome curved orthotropically and horizontal rhizome diameter in the region of attachment of the recently matured leaves. Fresh weight of the rhizome was measured after the three most recently matured leaves had been removed above 20 cm from the rhizome base. The entire leaves were not removed in order to protect the shoot apical meristem, as the rhizomes were used in other experiments. After measuring, the leaves were dried to ambient temperature and humidity in a dehumidified room. Leaves from all plants from Cycle 4 were further dried in an 80EC oven, ground in a Wiley mill, and analyzed for % C, N, and P by the FIU SERC lab.

3.2.5.2 Plant Tissue Mercury Concentrations

In the laboratory Cycle 4 leaves collected from sawgrass and cattail plants were subsampled by folding each individual leaf into halves again and again until the leaf bundle was about 6 inches long. Thin cross-sectional slices of each leaf were cut off both ends of the bundle with a stainless steel blade and placed in a 4-oz. cup. A composite of all five leaves was placed in a series of 4-oz containers for THg, stable isotopes, and QA/QC samples. The tissue in all containers was frozen.

3.2.6 Periphyton and Diatoms

3.2.6.1 Periphyton

In the laboratory, periphyton samples were prepared for analysis by removing any large particulate matter, adding a known amount of deionized water to dry samples and blending to achieve homogenization of the sample. An assortment of 4-oz cups were filled with the homogenate for THg, MeHg, diatom composition, pigment analysis, and stable isotopes. A volume to weight ratio for floating and soil biomass “cookies” was determined as well as AFDW. A volume to weight ratio for epiphytic periphyton was determined by comparing the volume in a cup to the subsequent dried weight. All samples were frozen following preparation for later analysis.

3.2.6.2 Diatoms

Subsamples of frozen periphyton material were thawed for diatom extraction. Diatoms were cleaned of calcite and organics by oxidation with concentrated sulfuric acid, potassium permangenate and oxalic acid. Once oxidized, samples were repeatedly washed with distilled water and decanted until a neutral pH was achieved. A concentrated subsample was then removed and dried onto a #1 coverslip. Coverslips were permanently mounted to microscope slides using Naphrax® mounting medium. Five hundred diatoms were counted and identified on random transects at 1008 X magnification on a Zeiss Axioskop microscope equipped with Nomarski DIC optics. To ensure taxonomic consistency, a photograph of each taxon was taken with a high resolution CCD digital camera equipped to the microscope.

3.3 QA/QC

Three analytical laboratories were utilized for QA/QC purposes as well as to process the large volume of samples collected. Tables 3.1 and 3.2 indicate Project Laboratory responsibilities (primary, primary QA/QC or secondary QA/QC), desired method detection limits (MDLs), holding times and the anticipated sample numbers. A detailed Quality Assurance Project Plan (QAPP) (Appendix A, Attachments 1 through 6) was prepared for this Project, which can be referenced for additional details on the analytical methods and standard operating procedures.

QA/QC has been an integral part of this project since its inception in Phase I and has continued throughout Phase II. Numerous QA/QC comparisons in water, soil, sediment, and tissue were conducted among the Project laboratories during Phase I. The addition of new parameters in Phase II presented the need for additional comparisons. Because differences in methods could produce differences in results, every effort was made to achieve agreement, whenever possible, even though common methods could not always be required. The goal with many of the parameters was to achieve the lowest consistent detection levels possible in order to provide the greatest amount of useful information to the Project.

To streamline the QA/QC process in Phase II, a detailed Quality Assurance Project Plan (QAPP) was developed during a pilot study to lay out data package requirements. (Appendix A). The data quality requirements and validation was specified in seven areas: accuracy and bias, precision, comparability, completeness, representativeness, tolerable background levels and data

quality objectives (Stanley and Verner,1985; Smith et.al., 1988). Method detection limits were specified based on the Phase I REMAP monitoring. Several detection limits were lowered where lower detection levels were needed and could be achieved. The validation process considered each of the following components using a statistically appropriate method.

3.3.l Accuracy and Bias

Accuracy is the degree to which a measured value or property agrees with an accepted "true" value (Taylor, 1988). Accuracy was estimated by measuring a sample with a known reference value. Bias is the systematic error inherent in a method or caused by some artifact or idiosyncrasy of the measurement system. Accuracy and bias were estimated by interlaboratory comparison of performance evaluation (PE) samples. In addition to the PE samples, internal standards developed by the laboratory were used to assess accuracy (bias) and matrix spikes were evaluated to assess matrix interferences with the analytical procedure.

3.3.2 Precision

Precision is a measure of the scatter among independent repeated observations or measures of the same property made under prescribed conditions (Taylor 1988). Precision was estimated at several points in the data collection process in order to estimate the effects of different sources of error. Precision can be partitioned into analytical and measurement system precision. Analytical precision refers to precision of the analysis performed by analytical instruments. It is estimated by laboratory replication, including replicates of performance audit samples. Measurement system precision refers to the precision of the sampling process, including sample collection, storage, transport, preparation and analysis. Colocated field duplicates were used to estimate precision of the entire measurement system, and laboratory splits were used to estimate the precision of sample processing after the sample had been received in the laboratory. Independent sets of spatially distributed duplicates and splits amounting to 10% of the data were analyzed for this purpose. Percent relative standard deviation estimates were one of the statistics calculated for precision estimation.

Precision and bias are estimates of random and systematic error in a measurement process (Kirchner 1983, Hunt and Wilson 1986). Collectively, they provide an estimate of the

total error or uncertainty associated with an individual measurement, or set of measurements. Estimates of the various error components was determined primarily by replicate sampling. The statistical design and sampling plan minimized systematic errors in all components except measurement error by using documented methodologies and standardized procedures (QAPP). The use of more sensitive methods achieving minimum detection levels and the associated analytical modifications were supported with additional documentation in the QAPP as the process moved toward standardization. In addition, standard PE samples were included in the laboratory and subjected to the entire measurement process. Variance components of the collection and measurement process (e.g., among analytical laboratories) were estimated after the pilot study and at the completion of each cycle so the QA efforts could be allocated to control major sources of error.

3.3.3 Comparability

Comparability is defined as "the confidence with which one data set can be compared to another" (Stanley and Verner 1985, Smith et al. 1988). Comparability studies were routinely conducted among the cooperating laboratories. Analysis and interpretation of the Phase I and II data was careful to evaluate change in the data making certain that changes, when they occur, were not due to analytical modifications because this is all important in the development of trend information. Typically, standard methods were used to assist with comparability, but there were no standard methods for mercury when this program began and this is a program to develop and refine analytical methods.

3.3.4 Completeness

Completeness requirements for this monitoring effort were that 90 percent of all proposed samples were collected and analyzed. This goal was achieved, however, it does not include sites where no samples could be obtained because the site was dry or located on private land.

3.3.5 Representativeness

Representativeness is defined as "the degree to which the data accurately and precisely represent a characteristic of a population parameter, a variation of a property, a process characteristic, or an operation condition" (Stanley and Verner 1985; Smith et al. 1988). The statistical survey, sampling periods and sample locations were selected to ensure representative samples. By following the statistical survey design which ensured probability samples were collected, by definition, the sample was representative of the specific known proportion of the population.

3.3.6 Tolerable Background Levels

Background is operationally defined as the amount of contamination due to collection, handling, processing and measurement. It is particularly relevant to the measurement of trace concentrations of mercury species. Background levels have not been tolerated due to the use of "clean sampling and analytical techniques" and if detected, the source was isolated and eliminated however, none was detected. Field and laboratory blank samples were added to each day's samples and used to control and eliminate background contamination, assess background levels and establish minimum detection limits and quantitation limits.

3.3.7 Data Quality Objectives

The assessment of Data Quality Objectives (Appendix A, Attachment 2) followed the guidance provided in EPA QA/G-4 (EPA 1994). DQO's developed during Phase I were used for comparison with QA results. Range checks were conducted for each constituent. Data were plotted on control charts to ensure data are within the DQO specifications (e.g., ±3 standard deviations, etc.) This assessment of the data was compared after the pilot study and each cycle of spatial sampling for conformance to the Phase I results. The data were flagged, as appropriate, if QC checks did not satisfy QA requirements. Additional QC analyses were conducted as part of the statistical analysis of the data. Deviations with Phase I results were investigated and the most probable explanation developed. The overall goal of maintaining consistency in the database between Phase I and II is most important to provide an accurate basis for trend assessments.

Specific Data Package Requirements for all laboratories participating in this study followed the guidance entitled "Laboratory Documentation and Quality Control Requirements

for Data Validation" (EPA 1998). The QA/QC data review package with the associated evaluations by FTN was presented to EPA Region 4 SESD OQA for final review (Appendix C). The baseline data developed during Phases I and II has a very high degree of internal consistency and future monitoring should endeavor to continue this consistency and comparability to minimize the introduction of artifacts into the baseline that has been established.

3.4 Database Exploration and Analyses

3.4.1 Data Verification and Validation

Data verification and validation analysis were conducted on the data, both for QA/QC and to establish the database for statistical and spatial analyses. This data set, with associated meta data, can be obtained from EPA Region 4 SESD, Athens, GA. The Phase I technical report complete with appendices and database are posted on the Region 4 website.

A number of statistical analyses were performed on these validated/verified data. These analyses are briefly discussed below.

3.4.2 Statistical Analyses

3.4.2.1 Descriptive Statistics

Descriptive statistics, including the range, mean, median, standard deviation, and quartiles for each constituent, by media, sampling cycle, and system type, were computed for various subpopulations (whole ecosystem, 7 geographic subareas (Figure 1.1), area north of Alligator Alley, etc.). These descriptive statistics provided initial insight into the structure and attributes of these subpopulations in the South Florida Everglades ecosystem. Box and whisker plots were computed and displayed by constituent, media, and subpopulations to provide a visual image of the subpopulation attributes.

Cumulative distributions were computed for each constituent, by media, cycle, and subpopulation to characterize the structure of subpopulations and to provide initial insight into any data transformations that might be required for parametric statistical analyses. Constituent information was sorted by latitude and longitude to determine if there might be north to south or east to west gradients that could provide insight into possible ecological interactions or indicate

other factors that might be contributing to the elevated Hg concentrations measured in the Everglades ecosystem.

3.4.2.2 Exploratory Analyses

A number of exploratory analyses were conducted on the data to gain greater insight into the structure and attributes of various subpopulations of interest. These exploratory analyses included scatter plots and scatter plot matrices, principal component, regression tree, and cluster analyses. These analyses identified several factors or principal components that contributed to the distribution of Hg in various media throughout the Everglades.

3.4.2.3 Inferential Statistics

Once the population and subpopulation attributes were described, statistical tests were performed to test various hypotheses about differences among subpopulation characteristics. These test included the Cramer von Mises test (Kiefer 1959) for differences among cumulative distributions, and analyses of variance and covariance to determine if various constituent combinations were contributing to differences among subpopulations. General linear models also were used to determine the proportion of the variance in fish Hg concentrations accounted for by a suite of other factors and constituents. Structural equation models or path analyses were used to test the strength of the data in supporting a series of risk hypotheses or conceptual models. Frequency tables were used to evaluate possible differences among the distribution of selected constituents.

3.4.2.4 Structural Equation Models and Path Analysis

Structural equation modeling is a general, but powerful multivariate technique used to investigate hypothesized relationships among variables and test causal models with a linear equation system. Structural equation modeling uses additive and multiplicative transformations on lists of numbers to evaluate the relation of the data to the conceptual or causal model. For example, for the list of numbers 1, 2, 3, the mean is 2 and the standard deviation is 1. If each number in this list is multiplied by the constant 4, the mean becomes 8 and the standard deviation becomes 4, or the variance becomes 16. If a relationship exists between a set of

numbers, X, and another set of numbers, Y, such that Y = 4X, then the variance of Y must be 16 times that of X. This relationship permits testing the hypothesis that X and Y are related by the equation, Y = 4X indirectly by comparing the variances of the Y and X variables (StatSoft, Inc. 2000). This is the underlying principle for path analysis. It is assumed there are sets of linear relationships among multiple variables, as developed in the conceptual or causal models, and that these relationships can be tested by examining the variances and covariances among variables.

Structural equation models are linear approximations, so similar to any regression equation, the fit will not be perfect. The purpose of these analyses are to describe the system of relationships that are supported by the underlying data, and assist in understanding the processes and pathways that are contributing to the system responses. Structural equation models are useful because they permit a full evaluation of the conceptual models, including relations among dependent variables in general linear models.

Path analyses or structural equation models are described in Allen (1997), Bollen (1989), Bollen and Long (1993), Duncan (1975), Everitt and Dunn (1983), Hoyle (1995), James et al. (1982), and in the journal, *Structural Equation Modeling*. Structural equation models have been used extensively in the social and psychological sciences, and are starting to be used more in the natural sciences.

3.4.2.5 Spatial Statistics

Kriging was used to characterize the spatial patterns of constituent concentrations throughout the marsh ecosystems. Kriging predictors are obtained at a fine grid of sites (here, every 0.1E latitude and longitude), from which a contour map of predicted values was obtained. The contour map of predicted constituent concentrations was obtained using Surfer® for Windows, version 9 (Golden Software, Inc. 2000). A linear kriging model was used consistently across all plots.

3.4.3 Media Specific Statistical Analyses

3.4.3.1 Mosquitofish Food Habitat Statistical Analyses

The percentage of each food category in the diet of fishes from each population sample was calculated from the mass data. These percentages were analyzed in analyses of covariance by grouping populations into geographic regions of the study area using two schemes. First, populations were grouped according to the water management region where they were found: WCA-1, WCA-2, WCA-3, Everglades National Park (ENP), and Big Cypress National Wildlife Preserve (Big Cypress). There are general north-south gradients in productivity across the Everglades following patterns of nutrient enrichment from agricultural runoff (Davis 1994, Stober 1996). The effects of this pattern were examined by grouping the populations into seven subareas by latitude and longitude. The average standard length of fish from each collection was retained as a covariate in these analyses. In all cases, data were examined for consistency with the assumptions of standard statistical procedures such as normality, and transformations were applied as needed to fulfill the assumptions of analyses (Zar 1984).

The trophic position of fishes (τ) from each sample were estimated by the sum of the trophic scores of their food items, multiplied by the proportion of the diet comprised by each food type (Adams et al. 1983). Literature values were used to classify the invertebrate prey into trophic groups. Adams et al. (1983; see also Winemiller 1990) provided the following equation to estimate trophic levels:

(1) $$\tau_i = 1.0 + \Sigma\, \tau_j\, (F_{ij}),$$

where τ_i is τ of fish species *i*,

τ_j is τ of food item *j* and,

F_{ij} is the proportion of the food volume for species *i* comprised by item *j*.

Thus, an herbivore consuming entirely plant material received a τ of 1.0. Detritus was given a τ of 0.2 because of the associated microbes inhabiting detrital particles. Diet breadth was estimated based on the proportion of volumetric contributions attributable to each food type by Levin's (1968) niche breadth formula:

(2) $B = 1/ \tau \rho_j^2$

where B is Levin's niche-breadth measure, and ρ_j = proportion of volume contributed by resource state j, for all species and size classes within each of those combinations. B ranges from 1, when one resource is used exclusively, to n, the number of resource states.

3.4.3.2 Aerial Photograph Interpretation

Long-term monitoring of plant community distributions as indicators of biogeochemical changes over broad areas such as the Everglades ecosystem can be implemented using remote sensing and geographic information system (GIS) techniques.

The study area also was subdivided into latitudinal zones by the EPA. Depicted in Figure 3.1 the boundaries between latitudinal zones correspond (from north to south) to 26.68E, 26.36E, 26.16E, 25.95E, 25.76E, 25.56E and 25.24Enorth latitudes. Within these latitudinal zones, the following monitoring sites were randomly located for EPA field data collection:
1) 132 stations for the Cycle 4 dry-season field survey conducted in April, 1999; and 2) 126 stations for the Cycle 5 wet-season field survey conducted in September, 1999. Eight of these monitoring sites fell outside of the EPA South Florida Ecosystem Assessment Project study area and were subsequently dropped from the analysis. The UGA Center for Remote Sensing and Mapping Science (CRMS) defined a 1 km^2 area around each of the remaining 250 monitoring sites for characterization of vegetation communities using remote sensing and GIS techniques.

Latitude and longitude values for all monitoring sites were used to create two Arc/Info coverages, one containing Cycle 4 sites, the other Cycle 5 sites (see Figure 3.1). Six sites from Cycle 4 and one site from Cycle 5 were selected for use in a pilot study designed to establish appropriate field techniques and statistical analysis methods before the project fieldwork began in April 1999. Eight sites provided to the CRMS fell outside both ENP and SFWMD boundaries and were disregarded, leaving 128 Cycle 4 sites and 122 Cycle 5 sites – a total of 250 monitoring sites.

Detailed vegetation databases previously compiled by the CRMS, NPS, and SFWMD from 1:40,000- and 1:24,000-scale color infrared (CIR) aerial photographs recorded in 1994/1995 were the primary data sources employed in this project. In each of these databases,

the vegetation was photointerpreted and vegetation boundaries rectified to the Universal Transverse Mercator (UTM) ground coordinate system tied to the North American Datum of 1983 (NAD 83) to within a root mean square error (RMSE) of approximately ±5 to 10 m. The minimum mapping unit was one hectare. Details on the mapping procedures, ground truthing and database development can be found in Welch et al. (1999) and Rutchey and Vilchek (1999). These data sets provided consistent and detailed information on vegetation communities for 117 of the 250 EPA monitoring sites

Vegetation patterns for the remaining 133 monitoring sites were interpreted using USGS CIR Digital Orthophoto Quarter Quads (DOQQs) covering WCA 1, WCA 2, EAA and a portion of WCA 3. The DOQQs of Florida were derived from USGS NAPP aerial photographs (the same 1994/1995 NAPP photographs used in the CRMS/NPS mapping project). They are reported by the USGS to meet planimetric accuracy standards of about ±3 m. Approximately 86 DOQQs were required to interpret the vegetation for those sites not included in the original CRMS/NPS/SFWMD databases.

For each site, a 1 km^2 plot centered on the monitoring site was created in Arc/Info coverage format (Figure 3.2). Vegetation data from the CRMS/NPS or SFWMD was clipped from the corresponding area in the vegetation databases. Where no vegetation data existed, the plot was digitally overlaid on the DOQQ and used as a template to interpret vegetation communities and create a new vegetation map centered on the monitoring site.

Vegetation classes delineated within the 1 km^2 plots followed the Everglades Vegetation Classification System developed by the CRMS, NPS and SFWMD (Madden et al. 1999; Welch et al. 1999). In this hierarchical system, 89 classes can be used to identify Everglades vegetation to the plant community, association and species levels. These classes also can be used in combination with numeric modifiers indicating factors affecting vegetation growth, (e.g., evidence of abandoned agriculture or altered drainage), information about the vegetation distribution (e.g., scattered individuals) and important environmental characteristics (e.g., abundant periphyton). Figure 3.3 provides a description of the Everglades Vegetation Classification System.

In order to accommodate the complex vegetation patterns found in the Everglades, a three-tiered scheme was developed for attributing vegetation polygons (Welch et al. 1995;

Obeysekera and Rutchey 1997). Using this scheme, interpreters were able to annotate each polygon with a dominant vegetation class accounting for more than 50 percent of the vegetation in the polygon. Secondary and tertiary vegetation classes were added as required to describe mixed plant communities within the polygon. This three-tiered scheme, as well as the hierarchical organization of the classification system, permits classes to be collapsed and generalized as required to examine trends over space and time.

The digital data sets for 250 sites were used to create hardcopy maps and to generate summary statistics of total area and percent cover for vegetation classes. To enable the efficient production of hardcopy map products, an automated mapping interface was developed. The interface allows each 1 km^2 map to be plotted using a standardized map collar, which included the EPA monitoring station name, cycle number, locator map, UTM grid, scale bar and legend. Detailed plant community information is included as text labels within each polygon. Tabular summary data of area and percent for each vegetation classification found in the 1 km^2 map, are automatically generated when the map is plotted and included in each map legend. The CRMS provided a total of 250 page-size (8.5 x 11 in.) paper maps prior to the intended field survey dates that included all monitoring sites in both Cycles 4 and 5.

3.4.3.3 Macrophyte Analysis

For both sawgrass and *Sagittaria* geographical trends in the data were initially analyzed by plotting individual parameters against site and analyzing responses across geographical areas. Covariance among parameters was analyzed using principle components analysis. Variation in response to biogeochemical parameters and tissue nutrient concentration was examined by regression/correlation of the first principle component(s) to the physical parameters. Variation in parameters among plants and sites was analyzed with a nested analysis of variance for sawgrass, while variation among leaves on individual plants, plants and sites was analyzed for *Sagittaria lancifolia*.

Cluster Analysis

To quantitatively identify the major plant communities in south Florida wetlands, a cluster analysis was performed on the species frequency data for all transects sampled during

Cycles 4 and 5. A cluster analysis requires the selection of a distance metric, which measures the similarity in species composition of each pair of transects, and the selection of a method for defining clusters. Sorenson's distance was used to measure the similarity of each pair of sites *i* and *j* .

$$Sij = \frac{\sum_{k=1}^{p} \left| n_{ik} - n_{jk} \right|}{\sum_{k=1}^{p} n_{ik} + \sum_{k=1}^{p} n_{jk}}$$

The sums are over each of the species encountered in the survey, and n_{ik} and n_{jk} are the frequencies of species *k* in sites *i* and *j*, respectively. Unweighted pair group mean averaging (UPGMA) was used to form the clusters. This agglomerative method starts with each transect forming a singleton. The pair of transects with the smallest value of Sorenson's distance form the first cluster. During each iteration of the algorithm, the distance between each pair of clusters (either singletons or clusters of multiple transects) are computed. For UPGMA the distance between a pair of clusters is defined to be equal to the average distance between each pair of transects comprising the two clusters, one from each component cluster. Then the pair of clusters with the smallest distance are combined to form the next cluster. This procedure is continued until all transects are combined into a single cluster.

The results of the cluster analysis were summarized in a dendrogram with branches occurring at heights corresponding to the distances between each pair of clusters. This dendrogram was used to classify the transects by designating a distance above which all branches were taken to correspond to distinct community types. The specification of this distance involved a trade-off between having more community types than could be easily interpreted and having so few that they did not correspond to homogeneous assemblages. The percent of transects containing each species was computed for each proposed community and ranked from highest to lowest. Each proposed community was than identified by the species that were found in 100 % or close to 100 % of the transects. If a proposed community was not dominated by one or more species, then its cluster was further partitioned until a homogeneous assemblage was obtained.

Logistic Regression

Logistic regression was used to investigate the relationship between the frequencies of common macrophyte species and geochemical variables. Let n_i denote the frequency of a given species out of the N = 20 quadrats in transect *i*, and x_i is the corresponding value of a geochemical variable. Assume that this frequency is binomially distributed; that is, the probability that the frequency is equal to n_i is given by

$$\Pr(n_i) = \left(\frac{N}{n_i}\right) p_i^{n_i} (1 - p_i)^{N - n_i}$$

where p_i is the probability that an arbitrary quadrat from transect i contains the species. The latter probability is modeled by

$$\rho i = \frac{\exp\{\beta_0 + \beta_1 x_i + \beta_2 x_i^2\}}{1 + \exp\{\beta_0 + \beta_1 x_i + \beta_2 x_i^2\}}$$

where β_0, β_1, and β_2 are the parameters of the model. Maximum likelihood estimators of these parameters were obtained using the generalized linear model procedure of SAS (SAS Institute 1999). Under the fitted model, a plot of ρ against χ yields a bell-shaped curve with a peak at $\chi \; ' \; \beta_1 \div 2\beta_2$ (assuming that the estimate of β_2 is negative), indicating the value of χ that is optimal for the given species. The breadth of the curve gives the range of conditions tolerated by that species. By plotting the curves for the various species on the same graph, the ranges of distribution of the various species were explored.

3.4.3.4 Diatom Analysis

Five hundred diatoms were counted and identified microscopically on random transects through the slide. Counts were relativized and patterns in species abundances analyzed by non-metric multidimensional scaling ordination with PC-ORD software. Species with significant distributional patterns were plotted on spatial maps and their relationship to environmental parameters analyzed by linear regression.

3.5 Mass Estimates

Mass estimates for THg, MeHg, and TP were calculated for the study area. Periphyton and fish Hg concentrations were measured and biotic densities estimated from the literature. Water, floc, and soil Hg and TP concentrations were measured and the mass estimates can be based on the spatial weighting factors associated with each probability sample. The spatial weighting factors for a cycle in Phase I and Phase II were 51.7857 and 48.3333, respectively. If the results of a variable were added to increase the sample size then the weight was divided by the number of cycles added. However, due to the variance in the number of sample sites per subarea from cycle to cycle final estimates were based on a constant area obtained by GIS for each subarea to remove this source of variance in the mass estimates.

3.6 Ecological Risk Assessment

The EPA ecological risk assessment framework and guidelines for ecological risk assessment (EPA 1992, 1998) have been the foundations for the South Florida Ecosystem Assessment project since its inception. These approaches were used to help guide a relative, comparative risk assessment of mercury in the South Florida Everglades ecosystem.

Table 3.1. REMAP Phase II critical parameters by cycle.

Parameter	Primary Lab	Primary QA/QC	Secondary QA/QC	Primary Lab MDL	Holding times	Site No. Per Cycle	Samp No.
SURFACE WATER							
DO	SESD	SESD-SOP		0.2 mg/L	in-situ	129	129
pH	SESD	SESD-SOP		0.1 s.u.	in-situ	129	129
Conductivity	SESD	SESD-SOP		1.0 uS	in-situ	129	129
Turbidity	SESD	SESD		0.1 NTU	48 hrs	129	155
Total Phosphorus	FIU	SESD		0.6 ug/L	28 days (1)	129	155
Total Nitrogen	FIU	SESD		0.03 mg/L	14 days (1)	129	155
Total Organic Carbon	FIU	SESD		0.12 mg/L	28 days	129	155
Sulfate	SESD	SESD		0.05 mg/L	28 days	129	155
Total Mercury	FIU	Battelle	SESD	0.3 ng/L	28 days	129	187
Methyl Mercury	FIU	Battelle		0.02 ng/L	28 days	129	187
SOIL/SEDIMENT							
Total Mercury	FIU	SESD	Battelle	4.3 Fg/kg	28 days	129	15
Methyl Mercury	FIU	Battelle		0.2 Fg/kg	28 days	129	155
Total Phosphorus	FIU	SESD		0.06 mg/kg	28 days	129	155
Ash Free Dry Weight	FIU			0.02 mg/kg		129	155
Bulk Density	FIU			0.001 g/cc		129	155
MOSQUITO-FISH							
Total Mercury	FIU	SESD	Battelle	3.2 ug/kg	28 days	129	1043
Length	FIU			0.1 mm	14 days (1)	129	993
Weight	FIU			0.05 g	14 days (1)	129	993

THg in water = 129 sites, 16 field blanks, 13 duplicates, 16 equip. blanks, 13 splits = 187
Porewater (nutrients/anions) = 129 sites, 13 dups, 16 equip blanks, 13 splits = 171
THg in soil = 129 sites, 13 dups, 13 splits = 155
THg in fish = 129 sites @ 7 fish/site = 903, 90 dups, 50 stand. tissue = 1,043

(1) Holding time goals

Table 3.2. REMAP Phase II noncritical parameters by cycle.

Parameter	Primary Lab	Primary QA/QC	Secondary QA/QC	Primary Lab MDL**	Holding Times	Site No. Per Cycle	Samp No.
SURFACE WATER							
(Eh) Redox Potential	SESD	SESD-SOP		1 m V	in-situ	129	129
Depth	SESD	SESD-SOP		1 cm	in-situ	129	129
Sulfide	SESD	SESD		0.01 mg/L	7 days (1)	129	155
(APA) Alkaline Phosphate	SESD	FIU		0.01 FM/h	24 hrs (1)	129	155
Temperature	SESD	SESD-SOP		0.15 C	in-situ	129	129
Chlorophyll a	FIU	FIU		0.1 Fg/L	14 days (1)	30	33
Sulfate (filtered-0.8)*	SESD	SESD		0.5 mg/l	28 days	129	155
Filtered (0.8) Nutrients (NH_4,NO_2, NO_3, PO_4)*	FIU	SESD		NO3-0.7 Fg/L NO2-0.3 Fg/L NH4-0.8 Fg/L SRP-0.6 Fg/L	48 hrs (1)	129	155
SOIL/SEDIMENT							
Type	SESD				14 days (1)	129	129
Thickness	SESD			1 cm	14 days (1)	129	129
pH	SESD				in-situ	129	129
(Eh in situ) Redox Potential	SESD			1 m V	in-situ	129	129
(Eh lab) Redox Potential	SESD			1 m V	48 hrs (1)	129	129
Sulfate	SESD			0.05 Fg/kg	28 days (1)	129	155
Mineral Content	FIU			3%	14 days (1)	129	155
(CH_4) Methane*	FIU				48 hrs (1)	129	155
(CO_2) Carbon Dioxide*	FIU				48 hrs (1)	129	155
(APA) Alkaline Phosphate	FIU					129	155
MOSQUITOFISH							
Sex	FIU				14 days (1)	129	993
Food Habits Analysis	FIU					129	993
PORE WATER*							
Total Phosphorus*	FIU			0.6 Fg/L	28 days (1)	129	171(a)
Total Nitrogen*	FIU			0.3 mg/L	14 days (1)	129	155
Filtered (0.8) Nutrients (NH_4, NO_2, NO_3, PO_4)*	FIU			NO3-0.7 Fg/L NO2-0.3 Fg/L NH4-0.8 Fg/L SRP-0.6 Fg/L	48 hrs (1)	129	155
Anions (Br, Cl, Fl, NO_2, NO_3, SRP, SO_4)*	SESD			ion chrom.	14 days (1)	129	155
Sulfate*	SESD			0.05 mg/L	28 days (1)	129	171
Sulfide*	SESD			0.01 mg/L	7 days (1)	129	171
PERIPHYTON - Utricularia							
Total Mercury	FIU	Battelle		4.3 Fg/kg	28 days (1)	100	110

Table 3.2. Continued.

Parameter	Primary Lab	Primary QA/QC	Secondary QA/QC	Primary Lab MDL**	Holding Times	Site No. Per Cycle	Samp No.
Methyl Mercury	FIU	Battelle		0.2 Fg/kg	28 days (1)	100	110
Diatoms*	FIU				14 days (1)	30	33
Pigments*	FIU				14 days (1)	30	33
PERIPHYTON - Soil							
Total Mercury	FIU	Battelle		4.3 Fg/kg	28 days (1)	100	110
Methyl Mercury	FIU	Battelle		0.2 Fg/kg	28 days (1)	100	110
Biomass*	SESD			1 g	14 days (1)	100	110
Diatoms*	FIU				14 days (1)	30	33
Pigments	FIU				14 days (1)	30	33
PERIPHYTON - Floating							
Total Mercury	FIU	Battelle		4.3 Fg/kg	28 days (1)	100	110
Methyl Mercury	FIU	Battelle		0.2 Fg/kg	28 days (1)	100	110
Biomass*	SESD			1 g	14 days (1)	100	110
Diatoms*	FIU				14 days (1)	30	33
Pigments	FIU				14 days (1)	30	33
SAWGRASS							
Total Mercury	FIU	Battelle		4.3 Fg/ku	28 days (1)	65	72
Surface Area (% cover)	UGA					65	
CATTAILS							
Total Mercury	FIU	Battelle		4.3 Fg/ku	28 days (1)	40	44
Surface Area (% cover)	UGA					40	
HABITAT EVALUATION							
Food Habits Analysis*	FIU					129	129
Periphyton*	FIU					129	129
Macrophyte Analysis*	FIU					129	129
Aerial Photo Interpretation	UGA					129	129

* Parameter added for the Phase II analysis

** minimum reportable quantities

(a) Porewater (nutrients/anions) = 129 sites, 13 dups, 16 equip blanks, 13 splits = 171

(1) Holding time goals

Table 3.3. Measurement and analytical methods for Phase II laboratories.

Media/Parameter	SERC (FIU)	SESD/ESAT	Battelle
Surface Water			
Dissolved Oxygen	--	EPA 360.1	--
pH	--	EPA 150.1	--
Temperature	--	EPA 170.1	--
Conductivity	--	EPA 120.1	--
Redox Potential	--	Voltage Meter	--
Water Depth	--	Calibrated Extensive Rod	--
Turbidity	--	EPA 180.1	--
Total Phosphorus	EPA 365.1(modified)	EPA 365.1	--
Total Nitrogen	Antek 7000N Analyzer	EPA 351.1 + (EPA 300 or 353.2) [(1)]	--
Ammonium-N (filtered-0.8)	EPA 350.1	EPA 350.1	--
Nitrite-N (filtered)	EPA 353.2	EPA 353.2 or EPA 300	--
Nitrate-N (filtered)	EPA 353.2	EPA 353.2 or EPA 300	--
Soluble Reactive Phosphate	EPA 365.1	EPA 365.1 or EPA 300	--
Total Organic Carbon	EPA 415.1 (modified)	EPA 415.2	--
Sulfate	--	EPA 300.0	--
Sulfate (filtered - 0.8)	EPA 300.0	EPA 300.0	
Sulfide	--	Hach	--
Alkaline Phosphatase	Experimental Methodology	--	--
Total Mercury	CVAF	CVAF	CVAF
Methyl Mercury	CVAF	--	CVAF
Pore Water			
Total Phosphorus	EPA 365.1	--	--
Total Nitrogen	Antek 7000N Analyzer	--	--
Ammonium-N (filtered)	EPA 350.1	--	--
Nitrite-N (filtered)	EPA 353.2	--	--
Nitrate-N (filtered)	EPA 353.2	--	--
Soluble Reactive Phosphate	EPA 365.1	--	--
Bromide		EPA 300.0	--
Chloride		EPA 300.0	--
Fluoride		EPA 300.0	--
Sulfate (ion)		EPA 300.0	--
Sulfide	--	Hach	--
Soil/Sediment			
Type	--	Visual Classification	--
Thickness	--	Visual Classification	--
Redox Potential (insitu)	--	Voltage Meter	--
Total Mercury	CVAF	CVAF	CVAF
Methyl Mercury	CVAF	--	CVAF
Sulfate	--	EPA 300.0	--
Total Phosphorus	EPA 365.1	--	--
Ash Free Dry Weight	ASTM D2974-87	--	--
Bulk Density	ASTM D4531-86	--	--
Mineral Content	ASTM D 2974-87	--	--

Table 3.3. Continued.

Media/Parameter	SERC (FIU)	SESD/ESAT	Battelle
Methane	ASTM D 2974-87	--	--
Carbon Dioxide	ASTM D 2974-87	--	--
Alkaline Phosphatase	Experimental Analytical Methodology	--	--
Periphyton - Utricularia			
Total Mercury	CVAF	CVAF	CVAF
Methyl Mercury	CVAF	--	CVAF
Diatoms	ASTM D 2974-87	--	--
Pigments	ASTM D 2974-87	--	--
Periphyton - Floating			
Total Mercury	CVAF	CVAF	CVAF
Methyl Mercury	CVAF	--	CVAF
Biomass*	--	--	--
Diatoms	ASTM D 2974-87	--	--
Pigments	ASTM D 2974-87	--	--
Media: Periphyton - Soil			
Total Mercury	CVAF	CVAF	CVAF
Methyl Mercury	CVAF	–	CVAF
Biomass		--	--
Diatoms	ASTM D 2974-87	--	--
Pigments	ASTM D 2974-87	--	--
Media: Sawgrass			
Total Mercury	CVAF	CVAF	CVAF
Media: Cattails			
Total Mercury	CVAF	CVAF	CVAF
Media: Mosquitofish			
Total Mercury	CVAF	CVAF	CVAF
Length	Measurement	--	--
Weight	Measurement	--	--
Sex	Visual	--	--
Gut Contents	Visual	--	--
Habitat Evaluation			
Food Habits Analysis	Visual	--	--
Periphyton*	Experimental	--	--
Microphyton*	Experimental	Experimental	--
Aerial Photo Interpretation*	–	Experimental (UGA)	–

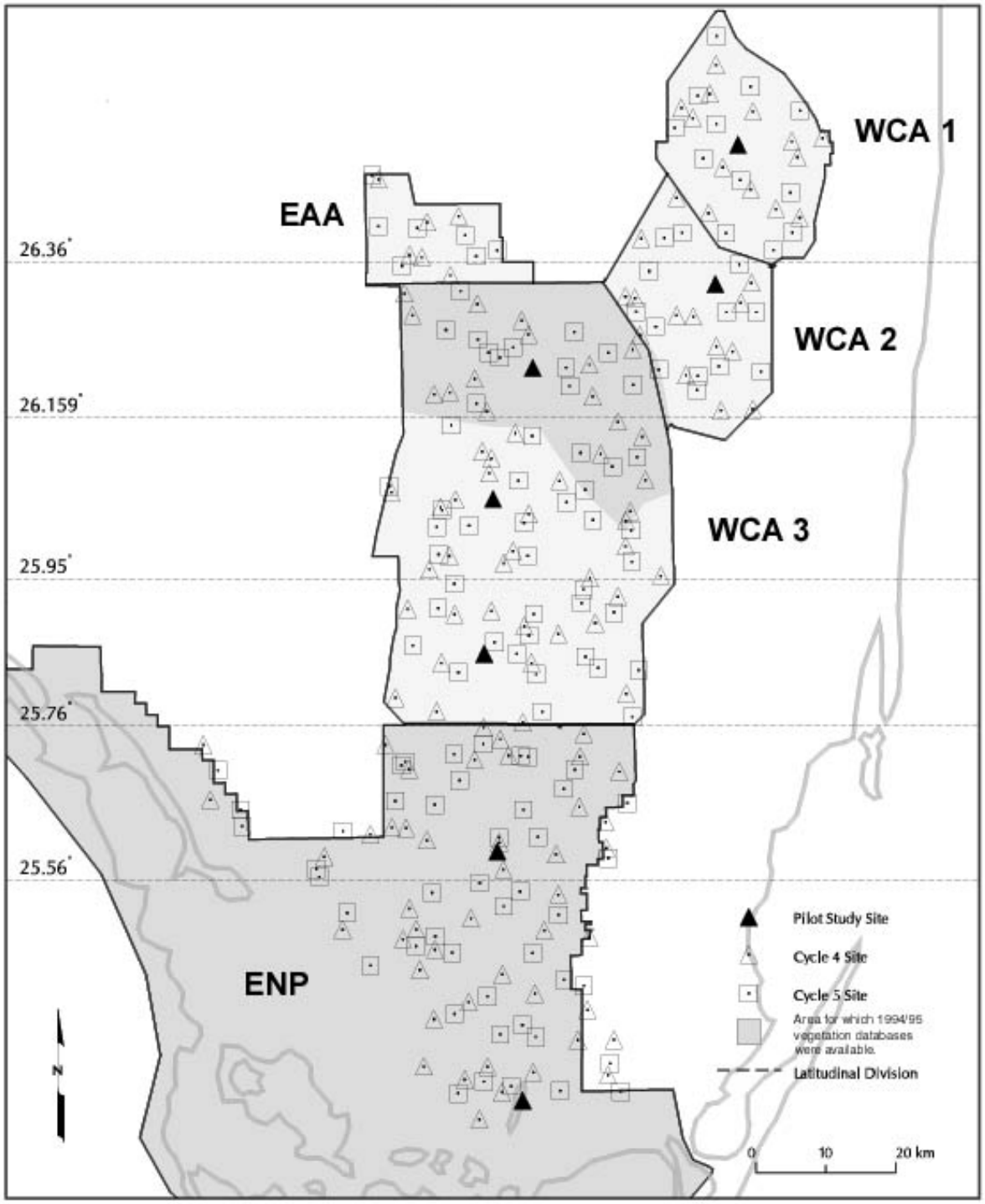


Figure 3.1. EPA South Florida Ecosystem Assessment Project study area and locations of pilot study, Cycle 4 and 5 monitoring sites.

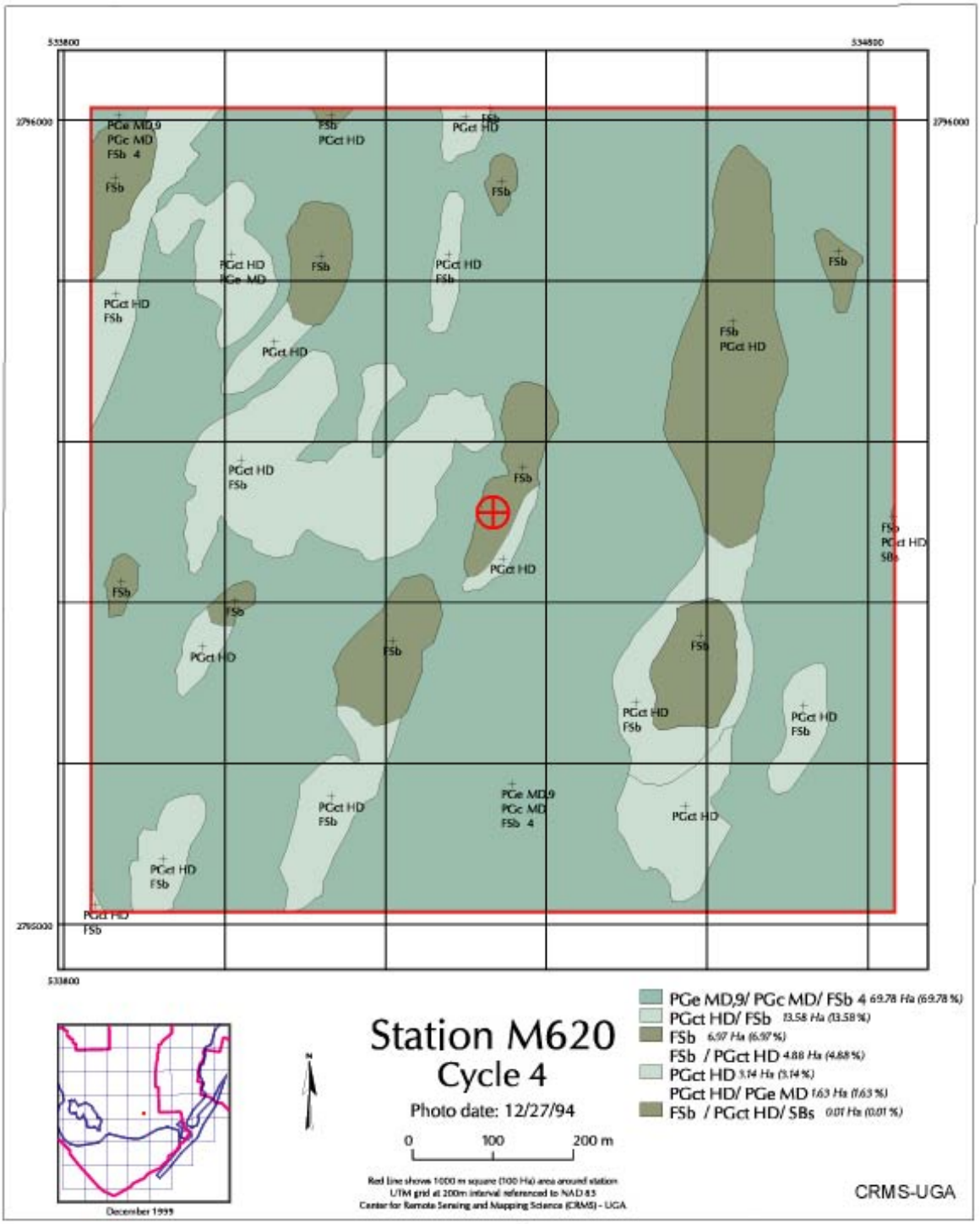


Figure 3.2. Sample vegetation map for a 1 km^2 plot surrounding a single EPA monitoring site.

Figure 3.3. Everglades Classification System legend.

4.0 MACROPHYTES

This chapter presents the results of the 3 macrophyte studies completed in the South Florida Ecosystem in 1999. Section 4.1 presents the results of the aerial photo vegetation assessment; Section 4.2 presents the results of the plant census study, and Section 4.3 presents the results of the macrophyte morphometric and landscape parameter analysis.

4.1 Aerial Photo Vegetation Assessment

Remote sensing and GIS techniques were used to assess vegetation patterns in the EPA South Florida Ecosystem Assessment Project. Tables 4.1 and 4.2 list the percent cover of major vegetation classes; cattail, sawgrass, wet prairie and other, for all 250 1-km^2 maps organized by region (Table 4.1) and latitudinal zone (Table 4.2), respectively (Welch and Madden 2000).

By region, cattail is most abundant in WCA2, covering nearly 25%, while only 1 percent of ENP contains cattail (Figure 4.1). Sawgrass covers approximately 40% of most regions with the highest coverage (55%) in ENP. Wet prairie ranges between 15 and 29% cover in all regions except ENP where wet prairie covers only 11%. Other vegetation is most abundant in the EAA and ENP, covering 45 and 33%, respectively.

The distribution of vegetation summary statistics by latitudinal zones is shown in Table 4.2 and Figure 4.2. Ranging from north to south (left to right on the table and graph), cattail coverage decreases steadily from 12 and 17% in the northern most zones to 1.5 and 0.4% in the southern most zones. Sawgrass coverage is fairly constant among northern zones (40 to 35%) and peaks at 68 and 44% cover in the most southern zones. Wet prairie decreases considerably at the northern border of ENP (25.76E), most likely due to the blockage of water flow by Tamiami Trail (US Highway 41) running east-west at this location. Other vegetation cover is distributed fairly evenly across latitudinal zones with the highest coverage in the southern most zone made up mainly of mangrove scrub and forest vegetation.

The spatial distribution of the four major vegetation classes over the entire study area is shown in Figure 4.3. The proportion of vegetation cover in each monitoring site is represented by a pie chart and the slices of the pie chart represent the relative areas of the four major vegetation classes within the 1-km^2 plots. Pie charts representing Cycle 4 monitoring sites are outlined in blue, while those representing Cycle 5 sites are outlined in red. Sites in which

periphyton existed in greater than 25% of the 1-km^2 plots are indicated with an asterisk placed at the center of the pie chart. It should be noted that given the difficulties in consistently identifying periphyton, as well as its transitory/seasonal nature, periphyton identification should not be considered definitive but rather indicative of potential areas of excessive periphyton growth.

The graphs depicted on the map represent histograms of dominant and secondary vegetation types, generalized into the four major vegetation classes. The smaller histograms summarize the total area included in each generalized class by region, namely: LOX, WCA2, WCA 3, Rotenberger/Holey Land EAA and ENP. Background colors in these histograms correspond to the colors of the region that is represented. The larger histograms, with white backgrounds, summarize the total area included in each generalized class by latitudinal zone.

In addition to representing major vegetation cover at each monitoring site, Figure 4.3 also provides spatial information on vegetation trends and characteristics by region and by latitudinal zone. For example, pie charts colored more than one half in dark blue and denoting monitoring sites dominated by wet prairie, are clustered within LOX, in the lower two-thirds of WCA 3 and within two particular areas of ENP. The distribution of predominantly wet prairie monitoring sites in the WCAs can be correlated with man-made structures such as canals and roadways that restrict hydrologic flow and tend to pool water. The two clusters of wet prairie sites in ENP occur within natural features, namely, Shark River Slough and Taylor Slough. The distribution of sites containing considerable proportions of cattail (colored red) are also grouped within WCA2, the north and east portions of WCA3 and the northeastern section of ENP. These sites appear to coincide with canals and may warrant further investigation of spatial correlations with nutrient levels within the system.

In order to determine if the proportion of vegetation types and areal coverage within the monitoring sites is representative of vegetation distributions over the entire Everglades study area, a comparison was made between the percent cover of 10 general vegetation classes as mapped within a subset of the monitoring sites and within the corresponding area in existing databases. Figure 4.4 depicts 30 monitoring sites in the northern portion of WCA3 (WCA3-N) that correspond with the existing WCA3 vegetation database (shaded in grey). Likewise, 44 monitoring sites corresponded with the northern portion of the ENP (ENP-N) vegetation database. The percent cover of vegetation was tallied for ten general classes defined by the EPA as sawgrass, wet prairie, muhly grass, cattail, mixed graminoid, non-graminoid emergent,

bayhead, pine/hardwood, water and other vegetation (Table 4.3). Results show that there is a high degree of correspondence between the percent cover of vegetation types in the monitoring sites of both WCA3-N and ENP-N with the percent cover derived from the existing databases. The greatest difference was only 7.1% for sawgrass in ENP-N, and the difference for all other vegetation types was less than 4%. The average difference in percent cover for vegetation types in ENP-N was 1.5% and the average for WCA3-N was 0.4%.

Figures 4.5 through 4.8 depict isolines representing predicted percentages of cover across the study area for each of the four major vegetation classes. Figure 4.5 illustrates relatively high proportions of cattail in WCA 2, the northeast section of WCA 3, and the border of ENP and WCA3 for the combined Cycle 4 and 5 monitoring.

Relatively even percentages of sawgrass were interpolated throughout the study area (Figure 4.6), while wet prairie isolines (Figure 4.7) reveal higher percentages within WCA 2, the lower two-thirds of WCA3 and slough areas of ENP. As expected, the highest levels of "other" vegetation are inside the Rotenberger/Holey Land EAA, largely due to abandoned agriculture in the EAA and the higher elevation pinelands area in ENP (Figure 4.8). These results illustrate the possibility of extrapolating information gathered within sample sites to the greater Everglades Ecosystem study area using spatial data analysis techniques such as kriging interpolation.

These patterns of major vegetation distributions over the entire study area were depicted in a map specially designed to visualize general trends in areal summary statistics. In addition, a comparison of areal statistics for monitoring sites with statistics derived from full-coverage vegetation databases confirmed randomly selected 1-km^2 plots adequately represented vegetation cover in the South Florida Ecosystem Assessment Project study area. Spatial interpolation of vegetation cover between monitoring sites also demonstrated the possibility of extrapolating sampled vegetation data to the broader landscape.

The 1994/1995 vegetation distributions documented in this study are now a baseline against which changes can be measured. It is anticipated that these methodologies can be used to efficiently monitor future vegetation and spatially analyze change as an indicator of biogeochemical fluctuations in the Everglades Ecosystem.

4.2 Plant Community Census

One hundred and sixty-one taxa were collected during the macrophyte census study. One hundred twenty eight of these taxa were identified to the species level and eight to the genus level, for a total of 136 identified taxa. Twenty-five plants could not be identified from the material collected.

4.2.1 Species Frequency Among Transects

The 136 species that were identified are listed in Table 4.4. Approximately one third (54 species or 34%) of all taxa were found in only a single transect (Table 4.5). Ninety-one percent (146 taxa) were found in fewer than 10% of all transects. Fifteen species occurred in more than 10% of the transects (43 – 309 transects) (Table 4.5), with the most common species found being sawgrass (Table 4.6).

The majority of species identified were dicotyledons (59%), followed by monocotyledons (33%), then ferns (6%) (Table 4.7). The most well represented families were the *Cyperaceae* (18 species), Poaceae (16 species), and Asteraceae (16 species) (Table 4.4). Only five exotic species (*Alternanthera philoxeroides, Ludwigia peruviana, Lygodium japonicum, Melaleuca quinquinervia*, and *Panicum repens*) were encountered on the transects. We found six endemics and an additional 121 native species (Table 4.4).

The number of live species per transect ranged from 0 (site 605, where all species were dead) to 30 (Table 4.8, Figure 4.9). The median and modal number of species per transect was 5, and the number of species per transect did not differ between the spring (Cycle 4) and fall (Cycle 5) sampling events. Only 8 transects (<2%) distributed among 6 sites had more than 15 species per transect.

4.2.2 Unidentified Species

Fifteen of the 25 unidentified taxa were collected during Cycle 4, while 10 were collected during Cycle 5 (Table 4.9). Nine of the Cycle 4 unidentified species came from a unique site, site 604, in the rocky glades area of ENP. In general the unidentified species were infrequent at the sites at which they were found, occurring in only one or two of the 20 quadrats sampled per transect (Table 4.9).

4.2.3 Cluster Analysis Results

After reviewing the results of defining 2 to 30 clusters, we used a grouping of 8 clusters to define communities, analyze their spatial distributions, and relate their occurrence to abiotic parameters. The dendrogram for these clusters is diagrammed in Figure 4.10.

The 8 clusters consisted of 4 relatively large clusters, each having more than 15 transects, and 4 small clusters, each with only 1 to 3 transects (Figure 4.10, Tables 4.10 and 4.11). The frequency of the 5 most common species in each large cluster is given in Table 4.12.

The distribution of clusters across the study area is given in Figure 4.11, while subsets of the clusters are mapped in Figure 4.12 and 4.13. There were two *Typha* clusters dominated by the southern cattail, *Typha domingensis*—a relatively large one with 18 transects and a smaller *Typha-Sagittaria* cluster with 2 transects (Figure 4.10). In the dendrogram these 2 clusters formed a branch distinct from the rest of the clusters (Figure 4.10).

The other large clusters (i.e., *Nymphaea-Utricularia, Eleocharis cellulosa*, and *Cladium* clusters) correspond to major south Florida communities recognized by other researchers (Davis 1943; Loveless 1959; Gunderson 1994, Doren et al. 1996, Jordan, Jelks and Kitchens 1997, Olmstead and Armentano 1997), whereas the small clusters (i.e., M707,the *Rynchospora tracyi* cluster, and M604) define several unique associations. The following sections describe the 4 large clusters, then the 4 smaller ones.

The sawgrass (*Cladium jamaicense*) cluster included 55% of the transects (Tables 4.6, 4.10 and 4.11). This cluster was defined by the presence of sawgrass, which occurred in all of its transects (Table 4.12). The next most frequent species, *Utricularia purpurea*, occurred in 26% of the transects. Transects belonging to this cluster occurred throughout the study area (Figures 4.11 and 4.13).

The cattail (*Typha domingensis*) cluster had 4% of the transects (Table 4.12). This cluster was defined by the presence of cattails, which were found in all of its transects. The next most frequently associated species, *Sagittaria lancifolia*, occurred in 44% of the cluster's transects. Transects belonging to this cluster were concentrated in the northern part of the study area (Figures 4.11, 4.12).

A water lily-purple bladderwort (*Nymphaea odorata-Utricularia purpurea*) cluster, which comprised 17% of the transects (Tables 4.10 through 4.12, Figures 4.11 and 4.12), was defined by an aggregation of species, none of which occurred in all of the transects belonging to

this cluster. The most common species in the cluster were *N. odorata*, found in 87% of the cluster transects, and *U. purpurea*, found in 78% of the transects. Four other species (*Eleocharis elongata, Panicum hemitomon, Utricularia foliosa and Utricularia gibba*), were found in 52% to 57% of the transects (Tables 4.10 and 4.12). Eight additional species occurred in more than 10% of the transects (Table 4.10). This cluster was common in the LOX subarea and in the central part of the study area, following the Shark Slough drainage (Figures 4.11 and 4.12).

An *Eleocharis cellulosa* (spikerush) cluster included 22% of the transects (Table 4.12). *E. cellulosa* occurred in all transects of this cluster, but *Utricularia purpurea*, which was found in 72% of the transects, and sawgrass, which was found in 60% of the transects, were also common (Tables 4.10 through 4.12). An additional 12 species were found in more than 10% of the transects (Table 4.10). This cluster was common in the central and southern part of the system but was lacking from the northern areas and from the region of Everglades National Park that separates Shark Slough from Taylor Slough (Figure 4.12).

The 4 small clusters included sites with the greatest species diversity. These clusters are described in the following section.

A small *Typha* cluster that had 2 transects from 2 sites (Tables 4.10 and 4.11) co-occurred with the larger *Typha* cluster (Figures 4.11 and 4.13). This small cluster differed from the larger *Typha* cluster in the consistent presence of *Sagittaria lancifolia* (Table 4.10 and 4.12).

A single site in the rocky glades area of ENP formed a unique cluster. This site, M604, occurred on the eastern edge of ENP and had a single transect (Figure 4.13). M604 had the highest species diversity in the study. Many of its taxa were typical south Florida pineland species (Table 4.10), and pinelands typically have the greatest species diversity in south Florida communities (Gunderson 1994).

The third small cluster had 3 transects from 3 sites where *Rhynchospora tracyi*, Tracy's beakrush, occurred in all transects. Two of the three transects came from adjacent sites in the LOX subarea, whereas the third was found in the northeast region of ENP (Table 4.10, Figure 4.12). These 3 transects were the only cluster where *R. tracyi* was the defining species. This is in contrast to previous descriptions of south Florida plant communities (Loveless 1959, Goodrick 1984, Gunderson 1994), which have recognized a prominent beakrush community. *R. tracyi* was one of the 15 most common species on our transects, especially in LOX and ENP (see Section 4.2.5), occurring in 17 to 24% of the transects in the sawgrass, *Nymphaea-*

Utricularia, and *Eleocharis* clusters. This species did not, however, form a distinct community in our analysis.

A cluster of 2 transects that had a high frequency of grasses, such as *Panicum tenerum* and *Eragrostis elliottii,* came from a single site, M707, in the northwestern part of ENP (Table 4.10, Figure 4.13). With 22 and 24 species per transect this site, which was a wetland site, had the second highest species diversity in our study. We need more information on the distribution of this unique community.

Designation of more clusters did not substantially alter community composition. If the data set was aggregated into 30 clusters, there were 8 large clusters that had 12 or more transects each. These 8 accounted for 86% of the transects and consisted of 3 sawgrass clusters (205 transects), 2 *Eleocharis cellulosa* clusters (91 transects), a *Utricularia purpurea-Nymphaea odorata* cluster (41 transects), an *Eleocharis elongata* cluster (12 transects) and a cattail cluster (12 transects).

The median number of species/transect differed among clusters. Two of the small clusters, the rocky glades cluster (M604, Figure 4.10) and the wet prairie grass cluster (M707, Figure 4.10) had the highest species numbers of any transects in the study, as described above. When the unknowns for these sites were included, the rocky glades cluster had 30 species on its single transect, while the wet prairie grass cluster had 22 and 24 species on its two transects. The *Nymphaea-Utricularia* cluster had a median of 7 species/transect, the *Eleocharis* cluster had a median of 6, the sawgrass cluster had a median of 5, and the *Typha* cluster had a median of 4.

4.2.4 Analysis of Clusters in Subareas

The subareas differed in the total number of species found. WCA2 had the fewest species, while LOX, Shark River and Taylor Sloughs had the most species (Table 4.13). When transects within subareas were clustered, the resulting groups generally resembled those found in the larger data set, although some refinements of the major groups also emerged. Not all of the clusters or even all of the major clusters found in the overall analysis were present in each subarea, and the frequency of clusters that were present varied among subareas (Figure 4.12 and 4.13).

The LOX subarea had 41 transects with 48 species. These transects aggregated into 2 large clusters and 1 small one (Figure 4.14, Table 4.14). The large clusters were a *Nymphaea*

odorata cluster (22 transects), which also had *Utricularia purpurea* and *Eleocharis elongata* at high frequencies, and a sawgrass cluster (17 transects). The small cluster had 2 transects, which were the 2 *Rhynchospora tracyi* transects recognized in the analysis of the entire dataset (Figures 4.12 and 4.14).

WCA2 had 41 transects with 23 species grouped into 3 major clusters (Figure 4.15, Table 4.15). The largest of these was a sawgrass cluster that had 30 transects. The other two were *a Nymphaea odorata-Utricularia purpurea-Eleocharis cellulosa* cluster with 5 transects and a cattail cluster with 6 transects. The cattail transects were from the periphery of WCA2, while the *Nymphaea* transects occurred on the southern edge (Figure 4.15). The sawgrass cluster occurred throughout (Figure 4.15).

WCA3-N had 43 transects with 49 species. These transects aggregated into a major sawgrass cluster with 33 transects and 2 small clusters (Figure 4.16, Table 4.16). One of the small clusters was a cattail group with 7 transects. The other was a small group of 3 transects from 2 sites west of the Miami Canal. *Paspalidium geminatum* was the most common species shared among these transects.

WCA3-SE had 49 transects with 18 species. A 3-cluster partition identified a large sawgrass cluster with 28 transects, a cluster of 14 transects dominated by *N. odorata* and a small group of 7 transects defined by *E. cellulosa* (Figure 4.17, Table 4.17). The *Nymphaea* transects were found northwest of the L-67 canal, while the *Eleocharis* transects were in the eastern half of the subarea (Figure 4.17). The sawgrass transects were distributed throughout. A 4-cluster partition subdivided the sawgrass group into a 9 transect cluster that was primarily sawgrass and a more diverse 19 transect cluster that also had *U. purpurea* present in all transects (Table 4.17).

WCA3-SW had 76 transects with 36 species. Aggregating the transects into four clusters produced a singleton cluster (M573), a small cluster of 5 transects dominated by *E. cellulosa*, and two large clusters (Figure 4.18, Table 4.18). One of the large clusters was a sawgrass cluster with 38 transects, while the other was a cluster of 34 transects dominated by *N. odorata*, *Utricularia purpurea*, and *Panicum hemitomon*. Increasing the number of clusters in this region to six identified an additional singleton cluster (M552) and subdivided the *N. odorata* cluster. The two new clusters were a group of 15 transects dominated by *P. hemitomon* and *Paspalidium geminatum* and a second group of 18 dominated by *N. odorata* and *U. purpurea* (Figure 4.19; Table 4.18). A *P. hemitomon -Paspalidium geminatum* cluster was not recognized in the overall

data set. The *Nymphaea-Utricularia* cluster dominated the central part of WCA3-SW, especially in the south, while the *Panicum-Paspalidium* cluster was more common in the northwest part of the subarea (Figure 4.19).

The Shark River Slough subarea had 98 transects with 66 species. One site, M605, had dead cattails but no living species present. This site was removed from the analysis, leaving 97 transects. If these transects were aggregated into 2 clusters, 1 of the 2 had 2 transects from a single site, M707. This cluster, which had the second highest species diversity in the study, was also identified in the clusters from the entire data set (Figure 4.13).

Clustering the Shark River Slough transects into 5 groups identified a singleton cluster (M587), 2 small clusters and 2 large clusters (Figure 4.20, Table 4.19). M587 was one of the three transects that formed the *Rhynchospora tracyi* cluster in the entire data set. The small clusters were the M707 site and a small group of 7 transects dominated by *E. elongata* but also having *P. hemitomon*, *R. tracyi*, and *U. purpurea* in high proportions. One of the two large clusters was a sawgrass cluster with 33 transects, which also had *Bacopa caroliniana*, *E. elongata*, and *R. tracyi*. The second large cluster had 53 transects. It was dominated by *E. cellulosa* but had high percentages of sawgrass and *U. purpurea* (Figure 4.20, Table 4.19). Defining six clusters did not make major divisions of these larger clusters, but when seven clusters were recognized, the sawgrass cluster was subdivided into a cluster of 21 transects that had sawgrass with *Eleocharis elongata* in 71% of the transects, and a cluster of 12 transects that had sawgrass and *R. tracyi* in all of the transects (Table 4.19).

The Taylor Slough (TS) subarea had 51 transects with 81 species. Initial clustering identified the single transect from site M604 as unique, as recognized in the analysis of the entire dataset. Removing this site from the analysis left 50 transects with 71 species. These clustered into 1 large and 2 small groups (Figure 4.21, Table 4.20). The large group was a sawgrass cluster of 35 transects that also had *R. tracyi* and *U. gibba* in more than 40% of the transects (Table 4.20). The two smaller groups were a cluster of 7 transects dominated by *E. cellulosa* and *R. tracyi* and a cluster of 8 transects that had sawgrass but also *P. hemitomon* (88%), *Cassytha filiformis* (75%), *R. tracyi* (75%), as well as other species. The sawgrass and sawgrass-*Panicum* + clusters were intermixed throughout the subarea, but the *E. cellulosa* cluster was found only in the southern part of the subarea (Figure 4.21).

4.2.5 Individual Species Distributions

Many of the common species occur in more than one cluster. Figures 4.22 through 4.61 map the distributions and provide the frequency per transect and percent occurrence by subarea for the 15 most common species.

Cladium jamaicense, sawgrass, was present in 74% of all transects (Table 4.6). Sawgrass was distributed throughout the system and was abundant where it occurred (Figures 4.22 and 4.23). The sawgrass cluster had 229 transects, but this species was also present in an additional 80 transects (309 of the 418 transects surveyed). Sawgrass was common along the transects, occurring in all 20 quadrats of 157 (51%) of the transects in which it was found (Figure 4.24). It was not evenly distributed throughout the system, however, being most frequent in the north, except for LOX, and south. Sawgrass was less common in the central areas (Figures 4.22, 4.25, and 4.26).

For transects in which sawgrass was present in the third 1-m^2 quadrat, the number of culms per m^2 ranged from 1 to 113 with a median number of 18. Density varied spatially in a manner similar to frequency among transects, so that the densest populations occurred in areas that had the most transects with sawgrass (Figures 4.25 and 4.26). Sawgrass morphology, however, had a different pattern. The largest sawgrass plants with long leaves and thick rhizomes were found in WCA3-SE and WCA3-SW (Figure 4.27), where sawgrass was least dense and was less common among transects (Figures 4.25 and 4.26). The smallest sawgrass plants, which had shorter leaves and narrower rhizomes, were found in Shark and Taylor Sloughs (Figure 4.27), where sawgrass had high frequencies per transect and intermediate to high culm densities (Figures 4.25 and 4.26). Transects in WCA2 and WCA3-N had high sawgrass densities, high frequency per transect, and large sawgrass plants (Figures 4.25 through 4.27). In the literature sawgrass communities have been characterized as dense vs. sparse and tall vs. intermediate or short, as well as dense vs. short. The asymmetry of the relationships among frequency, density, and size reported here quantify the reason for this confusion in the characterization of these communities.

Three species of bladderworts, *Utricularia purpurea*, *U. foliosa* and *U. gibba,* were among the 15 most frequent species (Table 4.6). *U. purpurea* was the second most common species, being present in 44% of all transects (Table 4.6). *U. foliosa* and *U. gibba* were approximately half as abundant; both occurred in 23% of the transects (Table 4.6).

None of the *Utricularia* species were evenly distributed throughout the Everglades (Figures 4.28 and 4.31). Both *U. purpurea* and *U. foliosa* were more common in LOX and in the southern part of WCA3 and Shark River Slough, tracking the longer hydroperiod parts of the system (Figures 4.28 through 4.30). Both species had their lowest frequencies per transect in WCA2, WCA3-N, Rotenberger-Holeyland, and Taylor Slough (Figure 4.29).

Because *Utricularia gibba* is a small plant that grows as a free-floating filament or embedded in the soil, it is a less obvious component of communities than *U. purpurea* and *U. foliosa*. It was, however, as common in the transects as the latter species (Table 4.6). *U. gibba* had a pattern of distribution similar to the other two species (Figures 4.31 through 4.33), with the exception that it was relatively more common in Taylor Slough (Figures 4.31 and 4.33).

Three species of spikerush, *Eleocharis cellulosa, E. elongata*, and *E. interstincta,* appeared in the transects. *E. cellulosa* was most common, occurring in 36% of all transects, followed by *E. elongata* (19% of all transects; Table 4.6), while *E. interstincta* was found in only 3 transects—2 in LOX and 1 in WCA3-SE. *E. interstincta* occurred in only 1 quadrat of each transect where it was found.

E. cellulosa and *E. elongata* were not evenly distributed throughout the ecosystem and had somewhat different frequencies and distributions (Figures 4.34 through 4.36). *E. cellulosa* was rare in the northern part of the system, while *E. elongata* was common in LOX but sparse in WCA2 and the northern part of WCA3-N (Figures 4.34 and 4.36). LOX was the only subarea where *E. elongata* was more commonly found than *E. cellulosa* (Figure 4.35). Both species were absent in samples from the area of ENP that separates Shark River Slough from Taylor Slough (Figure 4.34).

Maidencane, *Panicum hemitomon,* was rare in WCA2 and the Rotenberger-Holeyland tract but was found throughout the rest of the ecosystem (Figures 4.37 and 4.40). This species had both the greatest frequency per transect and was most common on the western side of WCA3-SW, followed by LOX (Figures 4.37, 4.39 and 4.40).

Although *Paspalidium geminatum,* Egyptian paspalidium, occurred in approximately half as many transects as *P. hemitomon* (17% vs. 32%, Table 4.6), *P. geminatum*'s distribution was similar to *Panicum hemitomon*'s (Figures 4.37, 4.38 and 4.40). In addition to being less abundant, *P. geminatum* was generally less frequent along a transect than *P. hemitomon,* especially in LOX and WCA3-SW (Figure 4.39). The exception to the relative abundances of

these two species was in the Taylor Slough region of ENP, where *Paspalidium geminatum* had a greater frequency per transect than *Panicum hemitomon* (Figure 4.39).

Sagittaria lancifolia, the lance-leaf arrowhead, was found in 27% of all transects (Table 4.6). *S. lancifolia* was widespread, although it was infrequent in the interiors of LOX, WCA2, and the central part of Shark River Slough (Figures 4.41 and 4.43). It was most common in WCA3-N and in the Rotenberger-Holeyland tract (Figure 4.41), where it occurred in over 70% of the transects sampled (Figure 4.43). When it was present, the species tended to occur in two to three 0.25-m^2 quadrats per transect, except in WCA3-N, where it was more frequent (Figure 4.42).

Twenty-four percent of the transects had water hyssops, *Bacopa caroliniana* . This species was most common in the western part of the ecosystem, although it was also found at lower frequencies per transect in LOX (Figures 4.44 through 4.46). It was infrequent in WCA2, WCA3-SE, and in Taylor Slough (Figure 4.44). *B. caroliniana* was most abundant and had the greatest frequencies per transect in the western parts of WCA3 and in Shark River Slough (Figures 4.44 through 4.46).

Nymphaea odorata, the white water lily, was found in 23% of all transects (Table 4.6). It was most common in LOX, WCA2, and WCA3-SE and WCA3-SW (Figures 4.47 and 4.49). Although the *Nymphaea-Utricularia* cluster from the total dataset extended to the southern part of Shark River Slough (Figure 4.12), *N. odorata* was rare or absent from our samples in ENP (Figures 4.47 and 4.49). Waterlilies had the greatest frequency per transect in subareas where it was also most common among transects (Figure 4.48).

Rhynchospora tracyi, Tracy's beakrush, was present in 19% of all transects (Table 4.6). It was found in LOX, Shark River Slough and Taylor Slough (Figures 4.50 and 4.52). This species was absent or rare in the intervening regions (Figures 4.50 and 4.52). *R. tracyi* had the highest frequency per transect in LOX and Taylor Slough (Figure 4.51).

Cattails, *Typha domingensis*, were found in 13% of all transects (Table 4.6). Transects with cattails occurred primarily in the northern part of the system, although this species was less frequent per transect and less common in LOX (Figures 4.53 through 4.55). *T. domingensis* was absent from WCA3-SW and occurred at only two sites in ENP, one in Shark River Slough and one in Taylor Slough (Figures 4.53 and 4.55). Cattails had the highest frequencies per transect

and were most common in the Rotenberger-Holeyland tract, WCA2, WCA3-N, and the northern part of WCA3-SE (Figures 4.53 through 4.55).

Peltandra virginica, green arrow arum, was found in 11% of all transects (Table 4.6). It was most common in LOX, where it occurred in 41% of the transects and achieved its highest frequencies per transect (Figures 4.56 through 4.58). This species was absent from transects in WCA2 and occurred in 13% or fewer of the transects in other parts of the system (Figures 4.57 and 4.58).

Hymenocallis latifolia , spiderlily, occurred in 10% of all transects (Table 4.6). This species was absent from transects in LOX, WCA2, and the Rotenberger-Holeyland tract and was present at low frequencies per transect in the rest of the system (Figures 4.59 through 4.61). In the central and southern regions *H. latifolia* was absent from central Shark River Slough (Figure 4.59).

4.2.6 Association of Clusters with Nutrients and Hydroperiod

Table 4.21 gives the means for nutrient, soil, and hydroperiod parameters at sites characterized by the four larger clusters derived from the total dataset. Since some sites had two transects that belonged to different clusters, only data from sites where both transects belonged to a single cluster were used to calculate values for Table 4.21.

Sites supporting different species clusters had distinct suites of abiotic parameters. The *T. domingensis* cluster occurred at sites that had the highest surface water nutrient values, lowest AP values, and highest soil TP of any of the clusters (Table 4.21).

Sites where the *Nymphaea-Utricularia* cluster was found were distinguished by the deepest soil, highest soil AFDW, and lowest soil bulk density (Table 4.21). This cluster also occurred at sites with the longest hydroperiods and deepest water (Table 4.21). The sites inhabited by this cluster had medium to high surface water nutrient values and soil TP (Table 4.21).

The sawgrass and *Eleocharis* clusters tended to have intermediate to low surface water nutrient values, although the *Eleocharis* sites had the highest surface water AP means (Table 4.21). These two sites differed in their soil characteristics. The *Eleocharis* sites had shallower soils with lower AFDW and lower soil TP (Table 4.21), suggesting that this cluster was found on soils with greater marl content. Both annual average hydroperiod classes and water

depth measured during the wet season sampling suggest that the sawgrass cluster occurs in shallower, shorter hydroperiod sites than the *Eleocharis* cluster (Table 4.21). A similar difference in water levels for *Eleocharis* wet prairies and sawgrass stands in LOX was reported by Jordan, Jelks and Kitchens (1997). Several studies have found that sawgrass occurs in shallower water than cattails (Urban et al., 1993; David 1996). Our data did not find these differences for sawgrass vs. cattail clusters distributed across the system, but the cattail cluster sample size was small in relation to the sawgrass cluster.

4.2.7 Relation of Species Presence to Soil TP and AFDW

Logistic regressions were used to relate species presence and abundance to soil TP and soil AFDW (Figures 4.62 and 4.63). Soil AFDW is correlated with the marl vs. peat content of the soils. Low AFDW is characteristic of marl soils, while high AFDW is found in peats. Because of the relation between soil type and hydroperiod, soil AFDW is also an indirect indicator of hydroperiod. The shape of the logistic regression curves and the peak values show both the range and optimal levels of soil TP and AFDW for each species.

Sawgrass is abundant across a broad range of soil TP, while cattail is absent at low levels but becomes increasingly abundant with increasing soil TP (CLJ and TYD, Figure 4.62). *R. tracyi* occurs at the lowest soil TP levels, followed by *E. cellulosa* and *U. purpurea*. All three of these species have narrow ranges, becoming rare above 500 Fg/g TP (RHT, ELC and UTP, Figure 4.62). *N. odorata* occurs over a broader range than the preceding two species and peaks at a higher soil TP (NYO, Figure 4.62). *S. lancifolia* is present across a broad range of soil TP but increases in abundance with increasing concentrations (SAL, Figure 4.62).

As with soil TP, sawgrass is abundant across a broad range of soil AFDW (CLJ, Figure 4.63). Cattail has a similarly broad range but peaks at a somewhat higher soil AFDW (TYD, Figure 4.63). Paralleling its occurrence in low TP sites, *R. tracyi* is found at sites with the lowest soil AFDW, i.e., greatest marl content (RHT, Figure 4.62). Although *E. cellulosa* and *U. purpurea* both occur at sites with a range of AFDWs, *E. cellulosa* has peak abundance at lower AFDW, while *U. purpurea* peaks at higher AFDWs (ELC and UTP, Figure 4.63). *N. odorata* is restricted to soils with AFDW > 60% (NYO, Figure 4.63). As with soil TP levels, *S. lancifolia* is present across a broad range of soil AFDW, with peak abundance at intermediate levels (SAL, Figure 4.63).

4.3 Morphometric Indicators

4.3.1 Variation Among Morphological Parameters

Leaves of *Cladium jamaicense* were longer and slightly wider in the wet season than in the dry season (Table 4.22). Otherwise, there were no differences between seasons in the morphological parameters measured for *C. jamaicense*. Similarly, leaves of *Sagittaria lancifolia* were longer in the wet season than in the dry season, but there was no difference in lamina width between seasons (Table 4.22).

Covariances among morphological measurements of *C. jamaicense* were all strongly positive (Table 4.23). Covariances among *S. lancifolia* parameters were also positive, with the exception of a negative covariance between petiole length and lamina width in Cycle 4 and 5 and petiole length and lamina length in Cycle 5 (Table 4.23). The relationship between petiole length and lamina length in Cycle 4 was positive but weak.

The relationships between leaf length and width for *C. jamaicense* and between lamina length and width for *S. lancifolia* were the strongest for any of the measured parameters (Table 4.23). These relationships are illustrated in Figure 4.64. While the correlation of leaf length to width at different sizes in *C. jamaicense* could be described by a line, the relationship of lamina length and width in *S. lancifolia* is more complex (Figure 4.64).

The first principal component (PC1) of the *C. jamaicense* morphometric data explained 77% and 79% of the variation observed in Cycles 4 and 5, respectively. The second principal component (PC2) explained an additional 16% (Cycle 4) and 12% (Cycle 5) of the variation. Thus the first 2 principal components of sawgrass explained 93% and 91% of the morphological variation in Cycles 4 and 5, respectively.

The first principal component of the *S. lancifolia* morphometric data explained 54% and 55% of the variation observed in Cycles 4 and 5, respectively, while PC2 explained 35% (Cycle 4) and 28% (Cycle 5). Together, these 2 principal components explained 89% (Cycle 4) and 83% (Cycle 5) of the variation in the *Sagittaria* morphological data.

Since the first two principal components captured the bulk of the variation observed in each data set, we focused our analyses on these two principal components. The distribution of variation between the 2 principal components differed between these species, however, with PC1 larger in *C. jamaicense* than in *S. lancifolia*, and, conversely, PC2 larger in *S. lancifolia*.than in sawgrass.

All of the morphological measurements were positively associated with PC1 of the *C. jamaicense* morphometric data in both seasons (Table 4.24). Thus, we interpreted this component primarily to explain variation in the size of plants. Number of leaves was positively associated with PC2, whereas leaf length and leaf width were negatively associated with PC2 of the *C. jamaicense* morphometric data (Table 4.24). The second principal component of sawgrass, however, explained < 20% of the variation in the data and its interpretation was not obvious.

All of the *S. lancifolia* morphological measurements except petiole length were positively associated with PC1 of the morphometric data. Petiole length had a weak negative loading on PC1 of the Cycle 5 data (Table 4.24). As with *C. jamaicense*, we interpreted PC1 of the *S. lancifolia* data to explain variation in size among plants. *S. lancifolia* petiole length and leaf base length were positively associated with PC2, whereas laminae width was negatively associated with PC2. Thus, PC2 could be interpreted as explaining variation in shape among leaves, contrasting leaves with proportionately long petioles and narrow laminae with leaves with short petioles and broad laminae.

The most important sources of variation in scores for both the first and second principal components from the *C. jamaicense* morphometric data were between sites, as indicated by the difference between sites and plants in the magnitude of Type III Shark River Slough in the analysis of variance (Table 4.25). There was little variation among plants within sites (Table 4.25).

Comparison among Type III Shark River Slough for sites, plants, and leaves within plants for Cycle 4 *S. lancifolia* principal components indicated that the majority of variation occurred among sites, then among plants within sites (Table 4.26). A similar result was found for Cycle 5 (Table 4.26).

4.3.2 Spatial Variation in Morphology

Morphology of both sawgrass and *S. lancifolia* varied across the ecosystem. Both species showed spatial variation in the parameters associated with plant size, as seen in significant differences among subareas in many of the morphological parameters (Tables 4.27 and 4.28; Figure 4.27). *S. lancifolia* also showed marked spatial variation in lamina width (Table 4.28; Figure 4.65). This spatial variation was present in both the wet and dry season (Figure 4.66).

Some of the variation in lamina width was independent of plant size, as seen in the large contribution of lamina width to PC2 (Table 4.24).

4.3.3 Analysis of Variation Among Soil Parameters

Soil bulk density was considerably lower during Cycle 5 than during Cycle 4 sampling (Table 4.29) at sites where *C. jamaicense* and/or *S. lancifolia* were sampled. Otherwise, there were no differences between sample periods in the soil parameters. Covariances among soil parameters were similar between the two sampling periods, with the exception of alkaline phosphatase, which changed signs between periods in its covariance with both soil total phosphorus and bulk density (Table 4.30). Percent ash-free dry weight and percent mineral content were perfectly negatively correlated, as expected. Covariances among soil parameters were similar for both *C. jamaicense* and *S. lancifolia* data sets (Table 4.30).

The first two principal components of the soil data from the *C. jamaicense* sites explained 82% and 78% of the variation observed in Cycles 4 and 5, respectively (Table 4.31). The first two principal components of the soil data from the *S. lancifolia* sites explained 83% and 80% of the variation observed in Cycles 4 and 5, respectively (Table 4.31). Since the first two principal components captured the bulk of the variation observed in the soil data, we focused our analyses on these two principal components.

Eigenvectors of the first two principal components of the soil physicochemical data were similar between *C. jamaicense* and *S. lancifolia* sites, probably reflecting the broad overlap in collection sites (Table 4.31). Total phosphorus, alkaline phosphatase and ash-free dry weight were positively associated with PC1 in both sampling periods, while bulk density and mineral content were negatively associated with PC1 (Table 4.31). The second principal component was positively associated with total phosphorus and bulk density in Cycle 4, but negatively associated with total phosphorus and very weakly associated with bulk density in Cycle 5. The second principal component was also strongly positively associated with alkaline phosphatase in both sampling periods. The strongest loadings on the first principal components of the soil data were from the physical measurements, ash-free dry weight, bulk density, and mineral content. Thus, PC1 was interpreted as explaining variation in soil physical characteristics, probably distinguishing peat- vs. marl-based soils. Note, however, that during the Cycle 4 sampling, some of the physical variation (bulk density) is reflected in the second principal component

(Table 4.31). Total phosphorus and alkaline phosphatase are strongly associated with PC2 from the soil data in both sampling periods, thus PC2 appears to reflect variation in phosphorus availability among sites.

4.3.4 Correlation of Soil Data with Morphological Data

4.3.4.1 Sawgrass - *Cladium jamaicense*

The first principal component of the soil data from the *C. jamaicense* sites was positively correlated with PC1 of the plant morphometric data in both sampling periods (Table 4.32). This suggests that larger *C. jamaicense* plants occurred in soils with more peat, resulting in positive relationships between characters such as leaf length and AFDW (Figure 4.67).

The correlations between PC2-soil, which reflected soil phosphorus status, and the principal components of the morphometric data were less strong than correlations with PC1-soil (Table 4.32). Similarly, univariate correlations between site averages for the morphological characters, such as leaf length, and soil TP, were not significant (Figure 4.67, Table 4.33).

4.3.4.2 Lance Leaf - *Sagittaria lancifolia*

The first principal component of the soil data from the *S. lancifolia* sites was positively correlated with PC1 of the morphometric data during Cycle 4, but there was no significant relationship during Cycle 5 (Table 4.32). Thus during Cycle 4, *S. lancifolia* plants had larger leaves on soils with a higher peat component, but this pattern was not observed during Cycle 5. This result could be an indirect effect of the correlation between deeper water, longer hydroperiod and peat soils. Because sites with peat soils are more likely to have standing water during the dry season, effects of water parameters on plant morphology are seen at these peat sites in the dry season, but would be found at additional sites during the wet season. The first principal component of the soil data was not significantly correlated with PC2 of the plant data in either cycle, indicating that soil physical characteristics did not significantly affect leaf shape (Table 4.32).

The correlation of PC2 of the soil data and both the first and second principal component of the plant data changed signs between the two sampling periods (Table 4.32), as was observed with the *C. jamaicense* data. The correlation between PC2-soil and PC1-plant was stronger in Cycle 4 than in Cycle 5. This, again, is probably an indirect result of the strong effect of water

depth on plant size. Univariate correlations of soil TP with morphological parameters reflecting plant size, such as total leaf length, were not significant (Figure 4.68).

Soil PC2 and plant PC2 were strongly correlated in both Cycle 4 and Cycle 5. Plants with proportionately shorter petioles and leaf bases and wider laminae tended to occur in sites where soil phosphorus availability was higher (Table 4.32). These patterns were supported by pairwise correlations between plant morphological and soil physicochemical measures (Table 4.33, Figure 4.68).

4.3.5 Correlation of Plant Tissue Nutrients to Soil and Morphological Parameters

Mean (S.E.) % C, % N, % P, and N:P molar ratio in *S. lancifolia* leaves collected during Cycle 4 were 41.0 (0.08), 2.77 (0.03), 0.16 (0.01), and 41.1 (0.37), respectively. Plants with higher % N and % P tended to occur on soils with higher phosphorus availability and higher bulk density, as seen in the positive correlations between % tissue N and P to PC2-soil (Table 4.34). This is illustrated in the significant positive correlation between soil TP and plant % P (Figure 4.69; $P < 0.0001$). Plant tissue nutrients were weakly correlated or uncorrelated with soil physical properties (ash-free dry weight and mineral content), as seen in the non-significance or low negative correlations between % tissue C, N, and P and PC1-soil (Table 4.34).

Percent C, N and P of *Sagittaria* leaves were strongly negatively correlated to PC2-plant, indicating that plants with short petioles and wide laminae had higher % C, N, and P in their leaves (Table 4.23, Figures 4.68 and 4.69). The correlations between plant PC1 and tissue nutrients show that larger plants tended to have lower values of % C and higher values of % P in their leaves, but these relationships were weak (Table 4.23).

Mean (S.E.) % C, % N, % P, and N:P molar ratio in the subsample of *C. jamaicense* leaves collected during Cycle 5 and bulked by site ($n = 30$) were 46.1 (0.11), 0.64 (0.01), 0.027 (0.001), and 55.6 (2.2), respectively. Sawgrass % N and % P are much lower than *S. lancifolia* leaf nutrients (see above) or plants in general (Bedford, Walbridge, and Aldous 1999, Koerselman and Meuleman 1996, Fourqurean, Zieman and Powell 1992). The values reported here are similar to those previously reported for *C. jamaicense* (Miao et al. 1998, Newman et al. 1996, Davis 1991, Craft et al. 1995, Steward and Ornes 1975, 1983). Sites with higher mean plant % N and % P occurred in soils with higher phosphorus availability (% N vs. PC2 = -0.46,

Table 4.35, Figure 4.72). Otherwise, correlations between *C. jamaicense* plant tissue nutrients and soil principal component scores were marginally significant or not significant (Table 4.35).

Relationships between *C. jamaicense* plant tissue nutrients and plant morphometric principal component scores were weak (Table 4.35), but consistent with patterns observed in comparisons between morphometric principal component scores and soil principal component scores (see above). Larger plants had higher % P in their leaves (Table 4.35).

4.3.6 Correlations of Hydroperiod Parameters to Plant Morphology and Soil Physicochemistry

All measures of hydroperiod varied among subarea divisions (Table 4.36). The ranking among subareas was nearly identical for the different hydroperiod measures. This similarity among measures was also reflected in strong, positive correlations among the variables (Table 4.37), indicating that where water is deeper, hydroperiod is longer. Since the measures were strongly correlated, we chose mean annual water depth, the hydroperiod variable most strongly correlated with the others (Table 4.37), to examine the relationships between hydroperiod and plant morphology, as well as soil physicochemisty.

Soil total phosphorus was weakly positively correlated with water depth at Cycle 4 sites but had no significant relationship in Cycle 5, while soil alkaline phosphatase had a less consistent relationship to water depth (Table 4.38). Soil physical parameters were more strongly correlated with water depth, indicating that soils with more peat occurred where water was deeper and hydroperiod longer (Table 4.38).

Hydroperiod was positively correlated with all *C. jamaicense* morphological parameters (Table 4.38). Larger sawgrass plants were found in deeper water and longer hydroperiods (Table 4.38, Figure 4.73).

In *S. lancifolia,* leaf base length and petiole length were positively correlated with hydroperiod (Table 4.38, Figure 4.74). Lamina length and width showed no or a small negative correlation with water depth (Table 4.38, Figure 4.74).

4.3.7 Summary of Morphometric Indicators

Morphology of both *C. jamaicense* and *S. lancifolia* varied across the Everglades ecosystem. This morphological variation was correlated with soil physicochemical parameters, but the two species responded to different aspects of the environment.

The morphological characters measured in sawgrass were highly interrelated and largely reflected variation in plant size. Variation in sawgrass size has been previously noted and related to soil depth (Davis 1943, Gunderson 1994). We do not know whether this variation represents genetic variation or phenotypic plasticity in sawgrass morphology. In a parallel study of sawgrass allozyme variation, we found no evidence for genetic differentiation in sawgrass that was related to environmental variation (Ivey and Richards, submitted), and our morphological data show continuous variation in size-related characters across the ecosystem.

Cladium jamaicense morphology was relatively insensitive to soil phosphorus levels, although sawgrass leaf % P and % N increased with soil TP. These results support conclusions from the plant census study, where sawgrass had a high probability of occurrence across a broad range of soil phosphorus levels (Figure 4.63). Although high sawgrass N:P ratios suggest that this species is severely P-limited, both the sawgrass morphological and plant census data show a lack of plant response to soil phosphorus. This insensitivity may result from the extremely low % P and N needed by sawgrass to make plant tissues. This suggests that tissue N:P ratios in plants adapted to oligotrophic environments may differ substantially from those in other plants. Departure from typical plant Redfield ratios may indicate differences in physiology and not necessarily nutrient limitation.

Sawgrass size correlated strongly to soil type and hydroperiod parameters. Thus, sawgrass size is an indicator of marl vs. peat soil, deep vs. shallow water, and long vs. short hydroperiod. Smaller plants occur in shallower, shorter hydroperiod, marl sites, while larger plants are found in deeper, longer hydroperiod, peat sites. As shown by the Plant Census results, sawgrass is abundant across the entire range of soil AFDW, but it has a broad peak of abundance at intermediate levels (Figure 4.62).

There was a positive relationship between *C. jamaicense* plant size and soil phosphorus, albeit a weak one. For example, one of the populations of *C. jamaicense* analyzed for tissue nutrients occurred near a canal, where soil phosphorus was high, and that population had the largest averages for morphological characters among the 30 populations analyzed for tissue nutrients. That population also had the highest % P in leaf tissue. These observations suggest that

C. jamaicense plants that absorb more soil phosphorus can respond by growing larger. The weak size correlation overall may indicate limitations on uptake of phosphorus by *C. jamaicense* (e.g., Newman et al. 1996, Davis 1991).

Sagittaria lancifolia responded differently than sawgrass to soil phosphorus levels, soil physical parameters and hydroperiod. Variation in plant size explained approximately half of the total variation in *S. lancifolia* morphology, while another third of *S. lancifolia'*s morphological variation was summarized by PC2 in our analysis. This latter portion of the total variation was thus independent of size-related factors, was strongly correlated with variation in lamina width, and was correlated to soil phosphorus levels. Thus, *S. lancifolia* morphological characters provide not just size-related variation, but more specific responses to environmental factors.

These different aspects of *S. lancifolia'*s morphology also responded to different environmental factors. Leaf size, especially leaf base and petiole length, increased in peat soils with longer hydroperiod, while leaf shape, especially lamina width, increased in soil with higher phosphorus levels and was unaffected by hydrological parameters. Water depth has been shown to affect *Sagittaria* leaf morphology in other ecosystems (Wooten 1986, Howard and Mendelssohn 1995).

We found additional support for the role of phosphorus in influencing leaf morphological changes in *S. lancifolia* from the data on leaf tissue nutrient content. *S. lancifolia* leaves with high tissue nutrients also had broader laminae and shorter petioles, and these plants grew in high-phosphorus soils. Common garden and controlled nutrient experiments (Ivey and Richards, unpublished data) also support the importance of phosphorus to lamina morphology of *S. lancifolia*. leaves. Together, our studies suggest that *S. lancifolia* leaf shape, especially as reflected in lamina width, provides an indication of soil nutrient status, and, specifically in the Everglades ecosystem, of P availability.

In both sawgrass and *S. lancifolia* we observed increasing leaf tissue nitrogen with increasing soil phosphorus availability. This was unexpected, since the soil data set included no specific measure of nitrogen availability and the availability of N and P can vary independently in soils. This increase in tissue N may indicate that nitrogen is available in Everglades soils, but its uptake by these plants is inhibited when phosphorus availability is low (Bloom et al. 1985).

4.4 Summary and Conclusions

This study presents a quantitative evaluation of marsh community types and their distributions across the Everglades ecosystem. As such, it provides a background against which to evaluate community change during and after restoration.

There are 4 major communities that are found across the entire ecosystem: sawgrass, waterlily-*Utricularia purpurea*, *Eleocharis cellulosa*, and cattail. These communities differ in their hydroperiod/water depth, soil type, and nutrient levels. The dominant species within each community have different tolerances for soil TP.

Sawgrass is the only community that occurs across the entire system; the other communities are more localized in their distributions. The sawgrass community type is dominated by *Cladium jamaicense*, with the next most common species present less than one quarter of the time. Thus, although specialized for survival in an oligotrophic environment, sawgrass is a generalist in this environment, occurring across a broad range of hydroperiods, soil types, and soil nutrient levels.

Although sawgrass is present throughout the Everglades, sawgrass morphology and density vary across the environment, correlated most strongly with changes in soil type and water level/hydroperiod. These variations in size and density have been used to describe different sawgrass communities (Davis 1943; Loveless 1959; Olmstead and Loope 1984; Gundersen 1994), but the correlations among the morphological parameters and their associations with environmental parameters have been confused. Controls on variations in density and morphology, as well as patchiness, represent areas of future research.

Although different parts of the ecosystem and different water management districts share many plant species, these areas do not have equal representation of the major plant communities identified here (Figure 4.75). The frequency and abundance of these communities differ across the system, indicating that ecosystem processes, such as nutrient or mercury cycling, vary among the regions.

Some communities that have been noted to be prominent historically did not appear as distinct communities in our analysis. For example, the *Rhynchospora tracyi* (beakrush) community described by Loveless (1959), Goodrick (1974), and Gunderson (1994), as well as others, did not form a distinct community in our clustering. In their study of vegetation in ENP Olmstead and Loope (1984) also did not recognize a distinct beakrush community, noting that *R. tracyi* is a common associate of their spikerush community. These differences could represent

a historical change in community composition in the ecosystem or could be a result of the quantitative rather than subjective nature of our analysis.

A rare but taxonomically diverse wetland community was identified at site M707 in ENP. Olmstead and Loope (1984) describe a species-rich prairie community that shares at least some species with this site. In order to understand the effects that ecosystem restoration might have on this community, additional information is needed on its distribution and the factors that control its diversity.

Sagittaria lancifolia is found across a broad range of soil TP and organic content in the Everglades. *S. lancifolia* leaf morphology provides an indication of soil nutrient level and water depth. Plants with broader laminae and shorter petioles are found in sites with higher nutrients, while plants with longer petioles are found in deeper sites.

Table 4.1. Percent cover of major vegetation classes by region, Cycles 4 and 5 combined.

Vegetation Class	Rotenberger/ Holey Land EAA Percent Cover	LOX Percent Cover	WCA 2 Percent Cover	WCA 3 Percent Cover	ENP Percent Cover
Cattail	11.1	8.7	24.9	7.8	1.0
Sawgrass	24.7	41.7	43.3	37.0	55.1
Wet Prairie	19.4	28.8	15.6	28.0	10.9
Other	44.8	20.8	16.2	27.2	33.0

Table 4.2. Percent cover of major vegetation classes by latitudinal zone, Cycles 4 and 5 combined.

Vegetation Class	26.68E to 26.36E	26.36E to 26.16E	26.16E to 25.95E	25.95° to 25.76E	25.76° to 25.56E	25.56° to 25.24E
Cattail	11.5	16.8	7.9	5.6	1.5	0.4
Sawgrass	39.9	40.0	35.7	34.5	68.0	43.5
Wet Prairie	22.6	14.9	32.2	36.9	7.1	14.4
Other	26.0	28.3	24.2	23.0	23.4	41.7

Table 4.3. Percent cover of vegetation in monitoring sites and corresponding areas in existing databases.

Vegetation Classes	% Cover ENP-N Existing Database	% Cover ENP-N Monitoring Sites	% Diff.	% Cover WCA3-N Existing Database	% Cover WCA3-N Monitoring Sites	% Diff.
Sawgrass	85.2	92.3	-7.1	68.7	69.6	-0.9
Wet Prairie	0.7	0.2	0.5	10.2	11.5	1.3
Muhly Grass	1.8	2.1	-0.3	0	0	0
Cattail	1.1	0.7	0.4	11.3	10.9	0.4
Mixed Graminoid	2.6	0.1	2.5	0	0	0
Non-gram. Emergent	0.1	0	0.1	2.9	2.7	0.2
Bayhead	1.7	1.6	0.1	0	0	0
Pine/Hardwood	0	0	0	0	0	0
Other Vegetation	6.0	2.3	3.7	6.5	5.2	1.3
Water	0.8	0.7	0.1	0.4	0.1	0.3

Table 4.4. Species identified during phase 2 sampling.

No.	Scientific name[1]	Code	or EX?[1,2]	Family
1	*Acrostichum daneaefolium*	ACD	N	Pteridaceae
2	*Aeschynomene partensis*	AEP	EN	Fabaceae
3	*Agalinis linifolia*	AGL	N	Scrophulariaceae
4	*Alternanthera philoxeroides*	ALP	EX	Amaranthaceae
5	*Amaranthus australis*	AMA	N	Amaranthaceae
6	*Ammannia latifolia*	AML	N	Lythraceae
7	*Andropogon species*	ANsp		Poaceae
8	*Anemia adiantifolia*	ANA	N	Schizaeaceae
9	*Angadenia berteri*	ANB	N	Apocynaceae
10	*Annona glabra*	ANG	N	Annonaceae
11	*Aristida purpurascens*	ARP	N	Poaceae
12	*Aster dumosus*	ASD	N	Asteraceae
13	*Azolla caroliniana*	AZC	N	Azollaceae
14	*Baccharis glomeruliflora*	BAG	N	Asteraceae
15	*Bacopa caroliniana*	BAC	N	Scrophulariaceae
16	*Blechnum serrulatum*	BLS	N	Blechnaceae
17	*Boltonia diffusa*	BOD	N	Asteraceae
18	*Caperonia castaneifolia*	CAC	N	Euphorbiaceae
19	*Cassytha filiformis*	CAF	N	Lauraceae
20	*Centella asiatica*	CEA	N	Apiaceae
21	*Cephalanthus occidentalis*	CEO	N	Rubiaceae
22	*Chara spp.*	CHsp		Characeae
23	*Chiococca alba (= C. pinetorum)*	CHP	N	Rubiaceae
24	*Cladium jamaicense*	CLJ	N	Cyperaceae
25	*Coelorachis (= Manisuris) rugosa*	COR	N	Cyperaceae
26	*Conoclinium coelestinum*	COC	N	Asteraceae
27	*Crinum americanum*	CRA	N	Amaryllidaceae
28	*Cynanchum sp.*	CYNsp	N	Asclepiadaceae
29	*Cyperus haspan*	CYH	N	Cyperaceae
30	*Cyperus sp.*	CYPsp		Cyperaceae
31	*Dichanthelium (= Panicum) portoricense*	DIP	N	Poaceae
32	*Diodia virginiana*	DIV	N	Rubiaceae
33	*Drosera species*	DRsp	N	Droseraceae
34	*Echites umbellata*	ECU	N	Apocynaceae
35	*Eleocharis cellulosa*	ELC	N	Cyperaceae
36	*Eleocharis elongata*	ELE	N	Cyperaceae
37	*Eleocharis interstincta*	ELI	N	Cyperaceae
38	*Elytraria caroliniensis*	EYC	EN/N	Acanthaceae
39	*Eragrostis elliottii*	ERE	N	Poaceae
40	*Erigeron species*	ERsp	N	Asteraceae
41	*Eriocaulon compressum*	ERC	N	Eriocaulaceae

42	*Eupatorium capillifolium*	EUC	N	Asteraceae
43	*Eupatorium mikanioides*	EUM	EN	Asteraceae
44	*Evolvulus sericeus*	EVS	N	Convolvulaceae
45	*Flaveria linearis*	FLL	N	Asteraceae
46	*Fuirena breviseta*	FUB	N	Cyperaceae
47	*Fuirena scirpoidea*	FUS	N	Cyperaceae
48	*Galium hispidulum*	GAH	N	Rubiaceae
49	*Helenium pinnatifidum*	HEP	N	Asteraceae
50	*Hydrocotyle umbellata*	HDU	N	Apiaceae
51	*Hymenocallis latifolia*	HYL	N	Amaryllidaceae
52	*Hypericum fasciculatum*	HYF	N	Clusiaceae
53	*Hyptis alata*	HYA	N	Lamiaceae
54	*Ilex cassine*	ILC	N	Aquifoliaceae
55	*Ipomoea sagittata*	IPS	N	Convolvulaceae
56	*Iva microcephala*	IVM	N	Asteraceae
57	*Jacquemontia curtisii*	JAC	EN	Convolvulaceae
58	*Justicia angusta*	JUA	EN	Acanthaceae
59	*Kosteletzkya virginica*	KOV	N	Malvaceae
60	*Leersia hexandra*	LEH	N	Poaceae
61	*Lemna valdiviana*	LEV	N	Lemnaceae
62	*Linum species*	LIsp	N	Linaceae
63	*Lobelia glandulosa*	LOG	N	Campanulaceae
64	*Ludwigia alata*	LUA	N	Onagraceae
65	*Ludwigia curtissii*	LUC	N	Onagraceae
66	*Ludwigia microcarpa*	LUM	N	Onagraceae
67	*Ludwigia octovalvis*	LUO	N	Onagraceae
68	*Ludwigia peruviana*	LUP	EX	Onagraceae
69	*Ludwigia repens*	LUR	N	Onagraceae
70	*Lygodium japonicum*	LYJ	EX	Schizaeaceae
71	*Lythrum alatum*	LYA	N	Lythraceae
72	*Melaleuca quinquinervia*	MEQ	EX	Myrtaceae
73	*Melanthera nivea*	MEN	N	Asteraceae
74	*Mikania scandens*	MIS	N	Asteraceae
75	*Mitreola petiolata*	MIP	N	Loganiaceae
76	*Muhlenbergia capillaris*	MUC	N	Poaceae
77	*Myrica cerifera*	MYC	N	Myricaceae
78	*Nymphaea odorata*	NYO	N	Nymphaeaceae
79	*Nymphoides aquatica*	NMA	N	Menyanthaceae
80	*Osmunda regalis*	OSR	N	Osmundaceae
81	*Oxypolis filiformis*	OXF	N	Apiaceae
82	*Panicum hemitomon*	PAH	N	Poaceae
83	*Panicum repens*	PAR	EX	Poaceae
84	*Panicum rigidulum*	PARI	N	Poaceae
85	*Panicum tenerum*	PAT	N	Poaceae

86	*Panicum virgatum*	PAV	N	Poaceae
87	*Paspalidium geminatum*	PDG	N	Poaceae
88	*Paspalum monostachyum*	PAM	N	Poaceae
89	*Paspalum monostachyum*	PSM	N	Poaceae
90	*Peltandra virginica*	PEV	N	Araceae
91	*Pentodon pentandrus*	PEP	N	Rubiaceae
92	*Phyla nodiflora*	PHN	N	Verbenaceae
93	*Pinguicula species*	PIN	N	Lentibulariaceae
94	*Pinus elliottii*	PIE	N	Pinaceae
95	*Piriqueta caroliniana*	PIC	N	Turneraceae
96	*Pityopsis (= Heterotheca) graminifolia*	PIG	N	Asteraceae
97	*Pluchea rosea*	PLR	N	Asteraceae
98	*Polygonum hirsutum*	POH	N	Polygonaceae
99	*Polygonum hydropiperoides*	POHY	N	Polygonaceae
100	*Polygonum punctatum*	POP	N	Polygonaceae
101	*Polygonum setaceum*	POS	N	Polygonaceae
102	*Pontederia cordata*	PNC	N	Pontederiaceae
103	*Potamogeton illinoensis*	POI	N	Potamogetonaceae
104	*Proserpinaca palustris*	PRP	N	Haloragaceae
105	*Rhynchospora (= Dichromena) colorata*	DIC	N	Cyperaceae
106	*Rhynchospora decurrens*	RHD	N	Cyperaceae
107	*Rhynchospora divergens*	RHDI	N	Cyperaceae
108	*Rhynchospora filifolia*	RHF	N	Cyperaceae
109	*Rhynchospora inundata*	RHI	N	Cyperaceae
110	*Rhynchospora microcarpa*	RHM	N	Cyperaceae
111	*Rhynchospora tracyi*	RHT	N	Cyperaceae
112	*Rumex species (verticillatus?)*	RUsp		Polygonaceae
113	*Sabatia grandiflora*	SBG	N	Gentianaceae
114	*Saccharum (= Erianthus) giganteum*	SAGI	N	Poaceae
115	*Sagittaria graminea*	SAG	N	Alismataceae
116	*Sagittaria lancifolia*	SAL	N	Alismataceae
117	*Salvinia minima (= S. rotundifolia)*	SLM	N	Salviniaceae
118	*Samolus ebracteatus*	SAE	N	Primulaceae
119	*Sarcostemma clausum*	SAC	N	Asclepiadaceae
120	*Saururus cernuus*	SACE	N	Saururaceae
121	*Schoenus nigricans*	SCN	N	Cyperaceae
122	*Scleria reticularis*	SCR	N	Cyperaceae
123	*Setaria parviflora (= S. geniculata)*	SEP	N	Poaceae
124	*Solidago stricta*	SOS	N	Asteraceae
125	*Spermacoce terminalis*	SPT	EN	Rubiaceae
126	*Taxodium distichum*	TAD	N	Taxodiaceae
127	*Tetrazygia bicolor*	TEB	N	Melastomataceae
128	*Teucrium canadense*	TEC	N	Lamiaceae
129	*Typha domingensis*	TYD	N	Typhaceae

130	*Utricularia cornuta*	UTC	N	Lentibulariaceae
131	*Utricularia foliosa*	UTF	N	Lentibulariaceae
132	*Utricularia gibba*	UTG	N	Lentibulariaceae
133	*Utricularia purpurea*	UTP	N	Lentibulariaceae
134	*Vernonia blodgettii*	VEB	N	Asteraceae
135	*Woodwardia virginica*	WOV	N	Blechnaceae
136	*Xyris smalliana*	XYS	N	Xyridaceae

[1] Authority for plant names and status =Wunderlin, R.P. 1998. Guide to the Vascular Plants of Florida. University Press of Florida, Gainesville.

[2] EN = endemic; N = native; EX = exotic

Table 4.5. Frequency of species present among transects.

No. Transects	No. Species Found	Cumulative No. Species Found	Cumulative % Found
1	54	54	34
2	24	77	48
3	19	96	60
4	7	103	65
5	6	109	68
6 – 10	14	123	77
11- 42	23	146	91
43 – 309	15	161	100

Table 4.6. Frequency among 418 transects of the 15 most common species.

Species	Presence in Transects:	
	No. of Transects	%
Cladium jamaicense Crantz	309	74%
Utricularia purpurea Walter	182	44%
Eleocharis cellulosa Torr.	151	36%
Panicum hemitomon Schult.	132	32%
Sagittaria lancifolia L.	114	27%
Bacopa caroliniana (Walter) B.L. Rob.	99	24%
Nymphaea odorata Sol.	98	23%
Utricularia foliosa L.	98	23%
Utricularia gibba L.	95	23%
Eleocharis elongata Chapm.	81	19%
Rhynchospora tracyi Britton	78	19%
Paspalidium geminatum (Forssk.) Stapf	73	17%
Typha domingensis Pers.	55	13%
Peltandra virginica (L.) Schott & Endl.	48	11%
Hymenocallis latifolia (Mill.) M. Roem.	43	10%

Table 4.7. Distribution of species among Systematic Groups.

	No. Spp.	%
Monocotyledon	45	33%
Dicotyledon	80	59%
Gymnosperm	2	1%
Fern	8	6%
Macroalgae	1	1%

Table 4.8. Summary data on the number of species per transect. Data include known and unknown species (N_{Spring} = 418 = 178; N_{Fall} = 240).

No. Species/Transect:	Total	Spring 1999 (Cycle 4)	Fall 1999 (Cycle 5)
Maximum	30	30	24
Minimum	0	0	1
Median	5	4	5
Mode	5	5	5

Table 4.9. Distribution among sites and transects of unidentified species from plant census.

	Total	Spring 1999 (Cycle 4)	Fall 1999 (Cycle 5)
No. of species	25	15	10
No. of transects	18	7	11
No. of sites	15	7	8
Median Freq./site	1	2	1
Range of Freq./site	1 – 4	1 - 4	1 – 2

Table 4.10. Species composition and frequency within each cluster. Names associated with species codes given in Table 4.4.

	Cladium Cluster		*Typha* Cluster		*Nymphaea-Utricularia* Cluster		*Eleocharis* Cluster		*Typha-Sagittaria* Cluster		Rocky Clades Cluster		*Rhynchospora* Cluster		Wet Prairie Grass Cluster	
No. Transects	229		18		69		93		2		1		3		2	
No. Species	82		29		36		41		12		23		15		28	
	Species	%	Species	%	Species	%	Species	%	Species	%	Species	%	Species	%	Species	%
	CLJ	100	TYD	100	NYO	87	ELC	100	SAL	100	CHP	100	RHT	100	ANG	100
	UTP	26	SAL	44	UTP	78	UTP	72	TYD	100	MUC	100	SAL	67	ASD	100
	SAL	25	MIS	39	ELE	57	CLJ	60	BAC	50	CLJ	100	PIN	67	BAC	100
	PAH	19	CLJ	28	PAH	57	PAH	46	CRA	50	MEN	100	PAH	67	CAC	100
	BAC	17	SAC	28	UTF	55	BAC	39	HYL	50	RHF	100	NYO	67	CAF	100
	RHT	17	POP	22	UTG	52	PDG	38	JUA	50	ANB	100	CLJ	67	CEA	100
	UTG	16	AZC	17	ELC	33	SAL	31	PAH	50	GAH	100	RHM	33	COR	100
	ELC	15	KOV	17	BAC	29	UTF	28	PDG	50	SPT	100	RHF	33	DIC	100
	PLR	15	PNC	17	NMA	28	UTG	25	PNC	50	PIC	100	PNC	33	ERE	100
	PEV	14	ALP	11	PDG	25	RHT	24	POHY	50	ANA	100	PDG	33	FUB	100
	UTF	14	LIsp	11	CLJ	22	NYO	19	POI	50	ANsp	100	OXF	33	HYA	100
	ELE	13	LUR	11	SAL	22	CHsp	17	UTF	50	DIP	100	HYA	33	IPS	100
	CEO	10	PAH	11	RHT	20	HYL	15			TEC	100	FUS	33	LUM	100
	HYL	10	RHF	11	PEV	10	ELE	13			EVS	100	ELE	33	MUC	100
	PNC	10	RUsp	11	HYL	9	CRA	12			LIsp	100	BAC	33	PAT	100
	TYD	10	SACE	11	PSM	9	JUA	9			PAH	100			RHF	100
	CRA	9	AMA	6	RHI	9	NMA	8			SAE	100			RHM	100
	MIS	9	DIC	6	TYD	9	ERC	6			CAF	100			UTP	100
	JUA	8	ERC	6	CHsp	6	PEV	6			COC	100			BOD	50
	PDG	8	HDU	6	CRA	6	PNC	5			PIG	100			CLJ	50
	CAF	7	LEH	6	ERC	4	TYD	5			PLR	100			CRA	50
	NYO	7	LYA	6	JUA	4	LUC	4			SOS	100			JUA	50
	PAT	7	NYO	6	OXF	4	PAT	4			TEB	100			LEH	50
	RHF	7	PARI	6	ELI	3	PAV	4							MIP	50
	IPS	6	PEV	6	PNC	3	POI	4							PAV	50
	LUC	6	SAGI	6	RHF	3	RHM	4							PEV	50
	LUR	6	SOS	6	RHM	3	PSM	3							PLR	50
	POHY	6	TEC	6	XYS	3	XYS	3							SOS	50
	XYS	6	XYS	6	BLS	1	AEP	2								
	ERC	5			CEO	1	AGL	2								
	PHN	5			FUS	1	LEH	2								
	RHM	5			HYF	1	PAM	2								
	MUC	4			LUC	1	TAD	2								
	MYC	4			SAG	1	CAF	1								
	OSR	4			UTC	1	EUM	1								
	PRP	4			WOV	1	FUS	1								
	BLS	3					IPS	1								
	CEA	3					OXF	1								
	EUC	3					PLR	1								
	HYA	3					POH	1								
	LOG	3					SEP	1								
	PAV	3														
	SAC	3														
	SOS	3														
	AZC	2														
	CAC	2														
	COR	2														
	ERsp	2														
	FLL	2														
	AEP	1														
	AGL	1														
	AML	1														
	ANG	1														
	ARP	1														
	ASD	1														
	CHsp	1														
	CYH	1														
	DIP	1														
	DRsp	1														
	ERE	1														
	EUM	1														
	EYC	1														
	HYF	1														
	ILC	1														
	LEH	1														
	LEV	1														
	LUO	1														
	LUP	1														
	LYJ	1														
	NMA	1														
	PAR	1														
	POS	1														
	PSM	1														
	RHD	1														
	RHI	1														
	SAGI	1														
	SBG	1														
	SCR	1														
	SEP	1														
	SPT	1														
	TEC	1														
	WOV	1														

NOTE: % = Percent of cluster transects where species occurred.

Table 4.11. Classification of sites in complete data set by cluster and subarea within cluster.

Cluster Classes[1]: 1 = NYO cluster; 2 = ELC+ cluster; 3 = CLJ cluster; 4 = TYD cluster; 5 = site 604; 6 = RHY 3 transects; 7 = 707 transects; 8 = SAL + TYD 3 transects

Subarea Classes: 0 = Rotenberger-Holeylan; 1 = WCA1A; 2 = WCA2A; 3 = WCA3A north of Alligator Alley; 4 = WCA3A south of Alligator Alley, western region; 5 = WCA3A south of Alligator Alley, eastern region and WCA3B; 6 = Everglades National Park, Shark River Slough drainage; 7 = Everglades National Park, Taylor Slough drainage and southern boundary.

[1] Site M605 in Subarea 6 had no living plants and was excluded.

Cluster 1, *Nymphaea odorata-Utricularia purpurea* cluster

Station	Station No.	Cluster Class	Subarea Class
M501	501	1	1
M502	502	1	1
M503	503	1	1
M504	504	1	1
M506	506	1	1
M508	508	1	1
M509	509	1	1
M511	511	1	1
M623	623	1	1
M623	623	1	1
M624	624	1	1
M625	625	1	1
M625	625	1	1
M626	626	1	1
M627	627	1	1
M627	627	1	1
M628	628	1	1
M630	630	1	1
M631	631	1	1
M635	635	1	1
M636	636	1	1
M636	636	1	1
M521	521	1	2
M530	530	1	2
M644	644	1	2
M649	649	1	2
M658	658	1	2
M535	535	1	3
M552	552	1	4
M565	565	1	4
M567	567	1	4

	M569	569	1	4		
	M570	570	1	4		
	M571	571	1	4		
	M573	573	1	4		
	M575	575	1	4		
	M676	676	1	4		
	M678	678	1	4		
	M678	678	1	4		
	M683	683	1	4		
	M685	685	1	4		
	M688	688	1	4		
	M690	690	1	4		
	M690	690	1	4		
	M691	691	1	4		
	M692	692	1	4		
	M694	694	1	4		
	M698	698	1	4		
	M548	548	1	5		
	M553	553	1	5		
	M555	555	1	5		
	M561	561	1	5		
	M568	568	1	5		
	M671	671	1	5		
	M674	674	1	5		
	M674	674	1	5		
	M675	675	1	5		
	M675	675	1	5		
	M677	677	1	5		
	M677	677	1	5		
	M686	686	1	5		
	M686	686	1	5		
	M581	581	1	6		
	M592	592	1	6		
	M594	594	1	6		
	M596	596	1	6		
	M599	599	1	6		
	M602	602	1	6		
	M705	705	1	6		
Cluster 2, *Eleocharis cellulosa* cluster						
		Station	**Cluster**	**Subarea**		
	Station	**No.**	**Class**	**Class**		
	M661	661	2	2		
	M662	662	2	2		
	M665	665	2	2		
	M534	534	2	3		
	M534	534	2	3		
	M542	542	2	4		
	M544	544	2	4		

	M549	549	2	4		
	M550	550	2	4		
	M551	551	2	4		
	M552	552	2	4		
	M554	554	2	4		
	M557	557	2	4		
	M558	558	2	4		
	M559	559	2	4		
	M563	563	2	4		
	M563	563	2	4		
	M564	564	2	4		
	M574	574	2	4		
	M673	673	2	4		
	M673	673	2	4		
	M676	676	2	4		
	M679	679	2	4		
	M682	682	2	4		
	M693	693	2	4		
	M699	699	2	4		
	M700	700	2	4		
	M543	543	2	5		
	M547	547	2	5		
	M556	556	2	5		
	M670	670	2	5		
	M670	670	2	5		
	M684	684	2	5		
	M689	689	2	5		
	M695	695	2	5		
	M697	697	2	5		
	M697	697	2	5		
	M701	701	2	5		
	M576	576	2	6		
	M577	577	2	6		
	M577	577	2	6		
	M578	578	2	6		
	M578	578	2	6		
	M585	585	2	6		
	M587	587	2	6		
	M600	600	2	6		
	M600	600	2	6		
	M601	601	2	6		
	M601	601	2	6		
	M606	606	2	6		
	M606	606	2	6		
	M607	607	2	6		
	M607	607	2	6		
	M702	702	2	6		
	M702	702	2	6		
	M703	703	2	6		
	M703	703	2	6		

	M709	709	2	6		
	M709	709	2	6		
	M710	710	2	6		
	M710	710	2	6		
	M711	711	2	6		
	M711	711	2	6		
	M712	712	2	6		
	M715	715	2	6		
	M715	715	2	6		
	M716	716	2	6		
	M717	717	2	6		
	M717	717	2	6		
	M718	718	2	6		
	M718	718	2	6		
	M719	719	2	6		
	M719	719	2	6		
	M720	720	2	6		
	M720	720	2	6		
	M722	722	2	6		
	M722	722	2	6		
	M723	723	2	6		
	M724	724	2	6		
	M726	726	2	6		
	M728	728	2	6		
	M728	728	2	6		
	M731	731	2	6		
	M731	731	2	6		
	M732	732	2	6		
	M732	732	2	6		
	M614	614	2	7		
	M617	617	2	7		
	M618	618	2	7		
	M620	620	2	7		
	M621	621	2	7		
	M621	621	2	7		
	M744	744	2	7		
Cluster 3, *Cladium jamaicense* cluster						
		Station	**Cluster**	**Subarea**		
	Station	**No.**	**Class**	**Class**		
	M510	510	3	0		
	M512	512	3	0		
	M514	514	3	0		
	M515	515	3	0		
	M516	516	3	0		
	M632	632	3	0		
	M633	633	3	0		
	M633	633	3	0		
	M639	639	3	0		
	M641	641	3	0		

	M641	641	3	0		
	M643	643	3	0		
	M643	643	3	0		
	M496	496	3	1		
	M498	498	3	1		
	M499	499	3	1		
	M500	500	3	1		
	M502	502	3	1		
	M504	504	3	1		
	M511	511	3	1		
	M622	622	3	1		
	M622	622	3	1		
	M624	624	3	1		
	M626	626	3	1		
	M628	628	3	1		
	M630	630	3	1		
	M631	631	3	1		
	M635	635	3	1		
	M640	640	3	1		
	M640	640	3	1		
	M507	507	3	2		
	M513	513	3	2		
	M517	517	3	2		
	M520	520	3	2		
	M521	521	3	2		
	M524	524	3	2		
	M528	528	3	2		
	M530	530	3	2		
	M533	533	3	2		
	M538	538	3	2		
	M539	539	3	2		
	M634	634	3	2		
	M634	634	3	2		
	M638	638	3	2		
	M638	638	3	2		
	M644	644	3	2		
	M647	647	3	2		
	M647	647	3	2		
	M648	648	3	2		
	M648	648	3	2		
	M649	649	3	2		
	M650	650	3	2		
	M650	650	3	2		
	M658	658	3	2		
	M661	661	3	2		
	M662	662	3	2		
	M665	665	3	2		
	M522	522	3	3		
	M523	523	3	3		
	M525	525	3	3		

	M526	526	3	3		
	M529	529	3	3		
	M531	531	3	3		
	M535	535	3	3		
	M536	536	3	3		
	M537	537	3	3		
	M540	540	3	3		
	M646	646	3	3		
	M646	646	3	3		
	M651	651	3	3		
	M651	651	3	3		
	M652	652	3	3		
	M652	652	3	3		
	M654	654	3	3		
	M654	654	3	3		
	M656	656	3	3		
	M656	656	3	3		
	M657	657	3	3		
	M657	657	3	3		
	M659	659	3	3		
	M660	660	3	3		
	M660	660	3	3		
	M663	663	3	3		
	M663	663	3	3		
	M664	664	3	3		
	M664	664	3	3		
	M666	666	3	3		
	M666	666	3	3		
	M542	542	3	4		
	M546	546	3	4		
	M550	550	3	4		
	M551	551	3	4		
	M557	557	3	4		
	M558	558	3	4		
	M559	559	3	4		
	M564	564	3	4		
	M565	565	3	4		
	M567	567	3	4		
	M569	569	3	4		
	M570	570	3	4		
	M573	573	3	4		
	M574	574	3	4		
	M575	575	3	4		
	M667	667	3	4		
	M667	667	3	4		
	M668	668	3	4		
	M668	668	3	4		
	M672	672	3	4		
	M672	672	3	4		
	M679	679	3	4		

	M680	680	3	4		
	M680	680	3	4		
	M682	682	3	4		
	M683	683	3	4		
	M685	685	3	4		
	M688	688	3	4		
	M691	691	3	4		
	M692	692	3	4		
	M693	693	3	4		
	M694	694	3	4		
	M698	698	3	4		
	M699	699	3	4		
	M700	700	3	4		
	M541	541	3	5		
	M545	545	3	5		
	M547	547	3	5		
	M548	548	3	5		
	M560	560	3	5		
	M562	562	3	5		
	M566	566	3	5		
	M568	568	3	5		
	M572	572	3	5		
	M572	572	3	5		
	M669	669	3	5		
	M669	669	3	5		
	M671	671	3	5		
	M681	681	3	5		
	M681	681	3	5		
	M684	684	3	5		
	M687	687	3	5		
	M687	687	3	5		
	M689	689	3	5		
	M695	695	3	5		
	M696	696	3	5		
	M696	696	3	5		
	M701	701	3	5		
	M580	580	3	6		
	M581	581	3	6		
	M582	582	3	6		
	M583	583	3	6		
	M584	584	3	6		
	M585	585	3	6		
	M586	586	3	6		
	M589	589	3	6		
	M590	590	3	6		
	M591	591	3	6		
	M591	591	3	6		
	M592	592	3	6		
	M594	594	3	6		
	M595	595	3	6		

	M595	595	3	6		
	M597	597	3	6		
	M599	599	3	6		
	M602	602	3	6		
	M704	704	3	6		
	M704	704	3	6		
	M705	705	3	6		
	M706	706	3	6		
	M706	706	3	6		
	M708	708	3	6		
	M708	708	3	6		
	M712	712	3	6		
	M714	714	3	6		
	M714	714	3	6		
	M716	716	3	6		
	M723	723	3	6		
	M724	724	3	6		
	M725	725	3	6		
	M725	725	3	6		
	M726	726	3	6		
	M727	727	3	6		
	M727	727	3	6		
	M730	730	3	6		
	M730	730	3	6		
	M734	734	3	6		
	M734	734	3	6		
	M598	598	3	7		
	M598	598	3	7		
	M603	603	3	7		
	M608	608	3	7		
	M608	608	3	7		
	M610	610	3	7		
	M610	610	3	7		
	M612	612	3	7		
	M612	612	3	7		
	M613	613	3	7		
	M614	614	3	7		
	M615	615	3	7		
	M615	615	3	7		
	M616	616	3	7		
	M616	616	3	7		
	M617	617	3	7		
	M618	618	3	7		
	M619	619	3	7		
	M619	619	3	7		
	M620	620	3	7		
	M729	729	3	7		
	M729	729	3	7		
	M733	733	3	7		
	M733	733	3	7		

	M735	735	3	7		
	M735	735	3	7		
	M738	738	3	7		
	M738	738	3	7		
	M740	740	3	7		
	M740	740	3	7		
	M741	741	3	7		
	M741	741	3	7		
	M742	742	3	7		
	M742	742	3	7		
	M743	743	3	7		
	M743	743	3	7		
	M744	744	3	7		
	M745	745	3	7		
	M745	745	3	7		
	M746	746	3	7		
	M746	746	3	7		
	M747	747	3	7		
	M747	747	3	7		
Cluster 4, *Typha domingensis* cluster						
		Station	**Cluster**	**Subarea**		
	Station	**No.**	**Class**	**Class**		
	M632	632	4	0		
	M637	637	4	0		
	M637	637	4	0		
	M639	639	4	0		
	M519	519	4	2		
	M642	642	4	2		
	M642	642	4	2		
	M645	645	4	2		
	M645	645	4	2		
	M518	518	4	3		
	M523	523	4	3		
	M527	527	4	3		
	M532	532	4	3		
	M653	653	4	3		
	M653	653	4	3		
	M655	655	4	3		
	M655	655	4	3		
	M561	561	4	5		
Cluster 5, Rocky glades cluster						
		Station	**Cluster**	**Subarea**		
	Station	**No.**	**Class**	**Class**		
	M604	604	5	7		

Cluster 6, *Rhynchospora tracyi* cluster

Station	Station No.	Cluster Class	Subarea Class
M496	496	6	1
M497	497	6	1
M587	587	6	6

Cluster 7, wet prairie grass cluster

Station	Station No.	Cluster Class	Subarea Class
M707	707	7	6
M707	707	7	6

Cluster 8, *Typha domingensis* + *Sagittaria lancifolia* cluster

Station	Station No.	Cluster Class	Subarea Class
M538	538	8	2
M659	659	8	3

Table 4.12. Five most common species in each of the 4 large clusters in the total dataset.

Cluster	No. Transects	Species	% Transects /Cluster
Sawgrass			
	229	*Cladium jamaicense*	100
		Utricularia purpurea	26
		Sagittaria lancifolia	25
		Panicum hemitomon	19
		Bacopa caroliniana	17
Cattail			
	18	*Typha domingensis*	100
		Sagittaria lancifolia	44
		Mikania scandens	39
		Cladium jamaicense	28
		Sarcostemma clausum	28
Water lily-bladderwort			
	69	*Nymphaea odorata*	87
		Utricularia purpurea	78
		Eleocharis elongata	57
		Panicum hemitomon	57
		Utricularia foliosa	55
Spikerush			
	93	*Eleocharis cellulosa*	100
		Utricularia purpurea	72
		Cladium jamaicense	60
		Panicum hemitomon	46
		Bacopa caroliniana	39
Small cattail			
	2	*Typha domingensis*	100

Cluster	No. Transects	Species	% Transects /Cluster
		Sagittaria lancifolia	100
		Bacopa caroliniana	50
		Crinum americanum	50
		Hymenocallis latifolia	50
Rocky glades			
	1	*Chiococca alba*	100
		Muhlenbergia capillaris	100
		Cladium jamaicense	100
		Melanthera nivea	100
		Rhynchospora filifolia	100
Beakrush			
	3	*Rhynchospora tracyi*	100
		Cladium jamaicense	67
		Nymphaea odorata	67
		Panicum hemitomon	67
		Pinguicula species	67
Wet prairie grasses			
	2	*Eragrostis elliottii*	100
		Panicum tenerum	100
		Caperonia castaneifolia	100
		Cassytha filiformis	100
		Utricularia purpurea	100

Table 4.13. Number of transects, species, and species per transect for each subarea, excluding the Rotenberger-Holeyland (Rot-Hol) tract.

Subarea	No. Transects	No. Species	No. Species per transect				
			Mean	Median	Mode	Max.	Min.
LOX	41	48	7	8	8	16	2
WCA2	41	23	3	3	3	9	1
WCA3-N	43	49	6	5	5	12	1
WCA3-SE	49	18	5	4	5	11	1
WCA3-SW	76	36	6	6	9	14	1
SRS	96	66	6	5	5	24	0
TS	51	81	8	7	4	30	1

Table 4.14. Subarea 1 Clusters. Names associated with species codes given in Table 4.4.

3 Clusters

No. Transects		17			2			22
No. Species		41			11			28
	Species	%	Species	%		Species	%	
	CLJ	100	RHT	100		NYO	100	
	PEV	65	PIN	100		ELE	77	
	MYC	41	PAH	100		UTP	73	
	CEO	35	NYO	100		PAH	68	
	OSR	35	SAL	100		UTF	59	
	BLS	29	BAC	50		UTG	45	
	ELE	29	CLJ	50		RHT	36	
	PNC	29	FUS	50		BAC	27	
	RHF	18	HYA	50		PEV	27	
	SCR	18	PDG	50		RHI	27	
	TYD	18	PNC	50		NMA	23	
	UTF	18				PDG	23	
	UTG	18				ELC	18	
	UTP	18				SAL	18	
	AZC	12				CLJ	14	
	DRsp	12				ERC	14	
	ERC	12				CHsp	9	
	ERsp	12				ELI	9	
	NYO	12				RHF	9	
	PDG	12				TYD	9	
	RHD	12				BLS	5	
	RHI	12				FUS	5	
	SAL	12				HYF	5	
	WOV	12				PNC	5	
	ACD	6				RHM	5	
	BAC	6				UTC	5	
	CYPsp	6				WOV	5	
	HYA	6				XYS	5	
	HYF	6						
	ILC	6						
	LUP	6						
	LYJ	6						
	MIS	6						
	NMA	6						
	PAH	6						
	PEP	6						
	PIN	6						
	POHY	6						
	RHT	6						
	SAGI	6						
	SLM	6						

NOTE: % = Percent of cluster transects where species occurred.

Table 4.15. Subarea 2 clusters. Names associated with species codes given in Table 4

No. Transects		30		6			5	
No. Species		15		7			12	
	Species	**%**	**Species**	**%**		**Species**	**%**	
	CLJ	100	TYD	100		NYO	100	
	NYO	30	LIsp	33		UTP	80	
	SAL	27	SAL	33		ELC	80	
	TYD	20	AZC	17		UTG	60	
	UTF	17	CLJ	17		CHsp	60	
	ELC	13	LUR	17		SAL	40	
	CEO	10	POP	17		PDG	40	
	RHF	7				PAH	40	
	XYS	7				FUS	20	
	BAC	3				ELE	20	
	ELE	3				CLJ	20	
	MEQ	3				BAC	20	
	PAH	3						
	PDG	3						
	POHY	3						

NOTE: % = Percent of cluster transects where species occurred.

Table 4.16. Subarea 3 clusters. Names associated with species codes given in Table 4.4.

No. Transects			7			33			3		
No. Species			49			37			13		
	Species	%		Species	%		Species	%			
1	TYD		100	CLJ		97	PDG		100		
2	MIS		71	SAL		76	BAC		67		
3	SAC		71	PNC		36	CLJ		67		
4	KOV		43	PAH		30	ELC		67		
5	PNC		43	POHY		30	ERC		67		
6	SAL		43	HYL		21	LUC		67		
7	ALP		29	TYD		21	RHT		67		
8	AZC		29	UTF		21	SAL		67		
9	POP		29	BAC		15	ELE		33		
10	RHF		29	CRA		15	HYL		33		
11	RUsp		29	MIS		15	NMA		33		
12	SACE		29	PDG		15	OXF		33		
13	CLJ		14	LUR		12	PAH		33		
14	ERC		14	PLR		12					
15	LEH		14	RHF		12					
16	LYA		14	CEO		9					
17	PAH		14	IPS		9					
18	AEP		0	LUC		9					
19	AML		0	PEV		9					
20	BAC		0	UTG		9					
21	CEO		0	AML		6					
22	CHsp		0	AZC		6					
23	CRA		0	NYO		6					
24	CYH		0	POS		6					
25	ELC		0	PRP		6					
26	ELE		0	UTP		6					
27	HYA		0	AEP		3					
28	HYL		0	CHsp		3					
29	IPS		0	CYH		3					
30	JUA		0	ELE		3					
31	LUA		0	ERC		3					
32	LUC		0	HYA		3					
33	LUR		0	JUA		3					
34	NMA		0	LUA		3					
35	NYO		0	NMA		3					
36	OXF		0	POI		3					
37	PDG		0	RHM		3					
38	PEV		0								
39	PLR		0								
40	POHY		0								
41	POI		0								
42	POS		0								
43	PRP		0								
44	RHM		0								
45	RHT		0								
46	UTF		0								
47	UTG		0								
48	UTP		0								
49	XYS		0								

NOTE: % = Percent of cluster transects where species occurred.

Table 4.17. Subarea 4 clusters. Names associated with species codes given in Table 4.4.

4 Clusters

No. Transects	38		34		4		1	
No. Species	30		26		12		7	
Species	%	Species	%	Species	%	Species	%	
CLJ	100	NYO	91	ELC	100	ELE	100	
CEO	32	UTP	91	BAC	75	UTG	100	
ELC	29	PAH	88	PAH	75	SAL	100	
BAC	24	ELC	85	SAL	75	CHsp	100	
UTF	24	BAC	59	UTF	50	ELC	100	
PEV	21	UTG	59	UTP	50	SAG	100	
CRA	18	PDG	56	ERC	25	PAH	100	
HYL	18	NMA	53	PDG	25			
UTP	18	UTF	50	PEV	25			
XYS	18	ELE	41	PNC	25			
JUA	16	SAL	38	TAD	25			
PAH	16	CRA	26	XYS	25			
LUC	11	CLJ	24					
IPS	8	HYL	21					
PNC	8	PSM	12					
BLS	5	CHsp	9					
ELE	5	JUA	9					
LEV	5	OXF	9					
LOG	5	XYS	9					
NYO	5	PNC	6					
PDG	5	CEO	3					
SAL	5	ERC	3					
UTG	5	LUC	3					
AEP	3	PEV	3					
AZC	3	POH	3					
ERC	3	POI	3					
LUR	3							
NMA	3							
POHY	3							
SAC	3							

6 Clusters

No. Transects	38		15		4		18		1		1	
No. Species	30		24		12		22		7		8	
Species	%	Species	%	Species	%	Species	%	Species	%	Species	%	
CLJ	100	ELC	100	ELC	100	NYO	100	ELE	100	BAC	100	
CEO	32	PAH	100	BAC	75	UTP	100	UTG	100	ELE	100	
ELC	29	UTP	87	PAH	75	ELC	78	SAL	100	PDG	100	
BAC	24	NYO	80	SAL	75	PAH	78	CHsp	100	PAH	100	
UTF	24	PDG	80	UTF	50	NMA	72	ELC	100	NYO	100	
PEV	21	BAC	67	UTP	50	UTG	61	SAG	100	OXF	100	
CRA	18	SAL	60	ERC	25	ELE	56	PAH	100	SAL	100	
HYL	18	UTG	60	PDG	25	BAC	50			CLJ	100	
UTP	18	UTF	53	PEV	25	UTF	50					
XYS	18	CRA	33	PNC	25	PDG	33					
JUA	16	HYL	33	TAD	25	CLJ	22					
PAH	16	NMA	33	XYS	25	CRA	22					
LUC	11	CLJ	20			PSM	17					
IPS	8	ELE	20			SAL	17					
PNC	8	CHsp	13			HYL	11					
BLS	5	XYS	13			JUA	11					
ELE	5	ERC	7			CEO	6					
LEV	5	JUA	7			CHsp	6					
LOG	5	LUC	7			OXF	6					
NYO	5	OXF	7			PEV	6					
PDG	5	PNC	7			PNC	6					
SAL	5	POH	7			XYS	6					
UTG	5	POI	7									
AEP	3	PSM	7									
AZC	3											
ERC	3											
LUR	3											
NMA	3											
POHY	3											
SAC	3											

NOTE: % = Percent of cluster transects where species occurred.

Table 4.18. Subarea 5 clusters. Names associated with species codes given in Table 4.4.

3 Clusters

No. Transects	28		7		14	
No. Species	15		16		11	
	Species	%	Species	%	Species	%
	CLJ	100	ELC	100	NYO	100
	UTP	77	UTP	100	UTP	100
	UTG	32	PAH	54	UTF	71
	TYD	23	PDG	54	UTG	64
	UTF	23	CLJ	46	CLJ	21
	BAC	14	UTG	46	TYD	21
	CRA	14	CHsp	38	ELC	14
	ELC	14	TYD	38	PAH	14
	NYO	14	JUA	31	PDG	14
	PNC	9	NYO	31	HYL	7
	RHF	9	UTF	31	SAL	7
	SAL	9	BAC	23		
	JUA	5	HYL	23		
	PAH	5	SAL	23		
	PEV	5	CRA	15		
			PEV	8		

4 Clusters

No. Transects	9		7		19		14	
No. Species	8		12		18		11	
	Species	%	Species	%	Species	%	Species	%
	CLJ	100	ELC	100	CLJ	100	NYO	100
	UTP	44	UTP	100	UTP	100	UTP	100
	TYD	22	PAH	71	UTG	58	UTF	71
	ELC	11	PDG	71	ELC	42	UTG	64
	PAH	11	TYD	71	NYO	37	CLJ	21
	PNC	11	CHsp	57	UTF	32	TYD	21
	UTF	11	JUA	29	BAC	26	ELC	14
	UTG	11	SAL	29	CRA	26	PAH	14
			UTF	29	JUA	16	PDG	14
			BAC	14	SAL	16	HYL	7
			HYL	14	TYD	16	SAL	7
			UTG	14	HYL	11		
					PAH	11		
					PDG	11		
					PEV	11		
					RHF	11		
					CHsp	5		
					PNC	5		

NOTE: % = Percent of cluster transects where species occurred.

Table 4.19. Subarea 6 clusters. Names associated with species codes given in Table 4.4.

5 Clusters

No. Transects	54		33		7		2		1	
No. Species	38		49		15		28		6	
Species	%	Species	%	Species	%	Species	%	Species	%	
ELC	94	CLJ	100	ELE	100	ANG	100	RHT	100	
CLJ	83	BAC	55	PAH	71	ASD	100	OXF	100	
UTP	81	ELE	52	RHT	71	BAC	100	RHM	100	
BAC	30	RHT	48	UTP	71	CAC	100	CLJ	100	
RHT	30	PLR	39	SAL	57	CAF	100	ELE	100	
UTF	28	PAH	36	UTF	57	CEA	100	RHF	100	
PAH	24	UTP	30	UTG	57	COR	100			
ELE	15	JUA	24	BAC	43	DIC	100			
PDG	15	ELC	21	PSM	43	ERE	100			
UTG	15	PAT	15	HYL	29	FUB	100			
CHsp	13	UTG	15	PDG	29	HYA	100			
SAL	13	CAF	12	CLJ	14	IPS	100			
HYL	11	ANG	9	ELC	14	LUM	100			
PEV	11	CAC	9	JUA	14	MUC	100			
CRA	7	CEA	9	RHM	14	PAT	100			
JUA	7	CRA	9			RHF	100			
PAT	7	HYL	9			RHM	100			
PAV	7	PHN	9			UTP	100			
LEH	6	PEV	9			BOD	50			
POI	6	RHM	9			CLJ	50			
PSM	6	SAL	9			CRA	50			
RHM	6	AEP	6			JUA	50			
AGL	4	AGL	6			LEH	50			
ERC	4	ASD	6			MIP	50			
NMA	4	FLL	6			PAV	50			
PAM	4	IPS	6			PEV	50			
AEP	2	LUO	6			PLR	50			
CAF	2	LUR	6			SOS	50			
ELI	2	MIS	6							
ERsp	2	PAV	6							
EUM	2	PRP	6							
IPS	2	RHF	6							
LUC	2	TYD	6							
LUR	2	UTF	6							
MIS	2	ECU	3							
PNC	2	EUM	3							
SEP	2	FUB	3							
TAD	2	HEP	3							
		LEH	3							
		LUC	3							
		MIP	3							
		NYO	3							
		PAR	3							
		PDG	3							
		PNC	3							
		PSM	3							
		RHI	3							
		SAGI	3							
		SEP	3							

NOTE: % = Percent of cluster transects where species occurred.

7 Clusters

No. Transects	53		21		7		12		2		1		1	
No. Species	37		29		15		38		28		6		7	
Species	%	Species	%	Species	%	Species	%	Species	%	Species	%	Species	%	
ELC	94	CLJ	100	ELE	100	CLJ	100	ANG	100	RHT	100	ELC	100	
CLJ	85	ELE	71	PAH	71	RHT	100	ASD	100	OXF	100	CLJ	100	
UTP	81	BAC	38	RHT	71	BAC	83	BAC	100	RHM	100	CHsp	100	
RHT	30	UTP	29	UTP	71	PLR	83	CAC	100	CLJ	100	ELE	100	
BAC	28	ELC	19	SAL	57	PAH	67	CAF	100	ELE	100	BAC	100	
UTF	28	PAH	19	UTF	57	JUA	58	CEA	100	RHF	100	PAH	100	
PAH	23	RHT	19	UTG	57	PAT	42	COR	100			CRA	100	
ELE	15	UTG	19	BAC	43	CAF	33	DIC	100					
PDG	15	CRA	14	PSM	43	UTP	33	ERE	100					
UTG	15	PLR	14	HYL	29	CAC	25	FUB	100					
CHsp	13	FLL	10	PDG	29	CEA	25	HYA	100					
HYL	11	HYL	10	CLJ	14	ELC	25	IPS	100					
PEV	11	IPS	10	ELC	14	AGL	17	LUM	100					
SAL	11	PEV	10	JUA	14	ANG	17	MUC	100					
CRA	8	RHF	10	RHM	14	ASD	17	PAT	100					
JUA	8	SAL	10	AEP		ELE	17	RHF	100					
PAT	8	TYD	10	AGL		LUO	17	RHM	100					
PAV	8	UTF	10	ANG		LUR	17	UTP	100					
LEH	6	AEP	5	ASD		MIS	17	BOD	50					
POI	6	ANG	5	BOD		PHN	17	CLJ	50					
RHM	6	JUA	5	CAC		PAV	17	CRA	50					
AGL	4	PHN	5	CAF		RHM	17	JUA	50					
ERC	4	NYO	5	CEA		AEP	8	LEH	50					
NMA	4	PAR	5	CHsp		ECU	8	MIP	50					
PAM	4	PNC	5	COR		EUM	8	PAV	50					
PSM	4	PRP	5	CRA		FUB	8	PEV	50					
AEP	2	PSM	5	DIC		HEP	8	PLR	50					
CAF	2	RHM	5	ECU		HYL	8	SOS	50					
ELI	2	SEP	5	ELI		LEH	8							
ERsp	2			ERC		LUC	8							
EUM	2			ERE		MIP	8							
IPS	2			ERsp		PDG	8							
LUC	2			EUM		PEV	8							
LUR	2			FLL		PRP	8							
MIS	2			FUB		RHI	8							
SEP	2			HEP		SAGI	8							
TAD	2			HYA		SAL	8							
				IPS		UTG	8							
				LEH										
				LUC										
				LUO										
				LUR										
				LUM										
				MIS										
				MIP										
				MUC										
				NMA										
				PHN										
				NYO										
				PAM										
				PAR										
				PAT										
				PAV										
				PEV										
				PLR										
				PNC										
				POI										
				PRP										
				RHF										
				RHI										
				SAGI										
				SEP										
				SOS										
				TAD										
				TYD										

Table 4.20. Subarea 7 clusters. Names associated with species codes given in Table 4.4.

3 Clusters + Rocky glades sites						Rocky glades site (M604)	
No. Transects	35		8		7	1	
No. Species	62		32		12	23	
Species	%	Species	%	Species	%	Species	%
CLJ	100	CLJ	100	ELC	100	CHP	100
RHT	43	PAH	88	RHT	86	MUC	100
UTG	37	CAF	75	CLJ	57	CLJ	100
PLR	31	RHT	75	SAL	43	MEN	100
MUC	26	PDG	50	BAC	29	RHF	100
PAT	26	RHM	38	PAH	29	ANB	100
UTP	23	SAL	38	PDG	29	GAH	100
MIS	20	COR	25	CAF	14	SPT	100
PAH	20	ERC	25	CRA	14	PIC	100
CAF	17	EYC	25	RHM	14	ANA	100
ELC	17	HYA	25	UTG	14	ANsp	100
PDG	17	HYL	25	UTP	14	DIP	100
SAL	17	LOG	25			TEC	100
EUC	14	MIS	25			EVS	100
LUC	14	PHN	25			LIsp	100
PHN	14	OSR	25			PAH	100
RHM	14	PAT	25			SAE	100
CEA	11	PRP	25			CAF	100
ELE	11	SOS	25			COC	100
ERC	11	SPT	25			PIG	100
HYL	11	IPS	13			PLR	100
IPS	11	IVM	13			SOS	100
LUR	11	JAC	13			TEB	100
PAV	11	LYJ	13				
PEV	11	MEN	13				
ARP	9	MUC	13				
DIP	9	PIE	13				
PRP	9	PLR	13				
RHF	9	PSM	13				
TEC	9	RHF	13				
COR	6	SAE	13				
CRA	6	SCN	13				
ERE	6						
FLL	6						
HYA	6						
JUA	6						
LOG	6						
OSR	6						
SBG	6						
SOS	6						
ANsp	3						
BAC	3						
BAG	3						
CAC	3						
CHP	3						
DIC	3						
DRsp	3						
EUM	3						
ILC	3						
LUO	3						
LYJ	3						
MYC	3						
PAM	3						
PAR	3						
RHDI	3						
SAC	3						
SEP	3						
TAD	3						
TYD	3						
UTC	3						
UTF	3						
VEB	3						

NOTE: % = Percent of cluster transects where species occurred.

Table 4.21. Abiotic factors associated with the 4 major clusters. Data from sites with only 1 type of cluster.

	NYMPHAEA & UTRICULARIA CLUSTER	ELEOCHARIS CELLULOSA + CLUSTER	CLADIUM JAMAICENSE CLUSTER	TYPHA DOMINGENSIS CLUSTER	SIGNIF.
Surface Water Nutrients (Mean (Standard error, N)):					
TOC (mg/L)	25	19	20	39	0.0054
	(2, 17)	(3, 23)	(1, 55)	(6, 5)	
TP (µg/g)	21	12	11	130	0.0004
	(6, 17)	(4, 23)	(1, 55)	(77, 5)	
TN (mg/L)	1.26	1.16	0.89	3.44	0.0081
	(0.18, 17)	(0.17, 23)	(0.07, 55)	(1.74, 5)	
NO3 (mg/L)	0.016	0.019	0.016	0.171	0.0148
	(0.002, 17)	(0.004, 24)	(0.004, 56)	(0.096, 5)	
NH4 (mg/L)	0.143	0.222	0.062	0.816	0.5574
	(0.115, 17)	(0.103, 24)	(0.025, 57)	(0.797, 5)	
AP (µM/L*hr)	1.46	1.97	1.11	0.18	<0.0001
	(0.22, 17)	(0.22, 24)	(0.10, 57)	(0.07, 5)	
Soil Characteristics (Mean (Standard error, N)):					
Soil Thickness (M)	1.87	0.45	0.75	0.81	<0.0001
	(0.26, 19)	(0.06, 31)	(0.07, 96)	(0.18, 8)	
AFDW (%)	90	47	64	79	<0.0001
	(3, 19)	(4, 31)	(3, 96)	(8, 8)	
Bulk Density (g/cc)	0.18	0.52	0.79	0.53	<0.0001
	(0.03, 19)	(0.11, 31)	(0.10, 94)	(0.08, 8)	
TP (µg/g)	268	155	279	607	<0.0001
	(26, 18)	(16, 31)	(17, 96)	(93, 8)	
AP (µM/g)	12.42	3.56	7.99	10.25	0.0130
	(2.79, 19)	(1.59, 31)	(1.53, 91)	(5.18, 8)	
Hydroperiod Characteristics (Median (N)) and Water Depth (Mean (Standard error, N))					
Annual Average Hydroperiod Class[1]	7	6	5	6.5	<0.0001
	(19)	(29)	(95)	(8)	
Annual Average Ponding Depth Class [2]	4	3	3	3	<0.0001
	(19)	(29)	(95)	(8)	
Water Depth (ft), wet season data only	3.1	2.1	1.6	1.7	0.0002
	(10, 0.3)	(17, 0.2)	(47, 0.1)	(4, 0.1)	

[1] 7 = 330-365 d; 6 = 300-330 d; 5 = 240-300 d; classes 1 – 7 possible.
[2] 4 = 1.0 to 2.0 ft; 3 = 0.5 to 1.0 ft; classes 1 – 6 possible.

Table 4.22. Means (S. E.) of measurements used to study morphological variation in *Cladium jamaicense* and *Sagittaria lancifolia* collected from the Florida Everglades.

	Cycle 4	**Cycle 5**
C. jamaicense		
Number of leaves	5.4 (0.08)	5.6 (0.09)
Leaf length (cm)	164.3 (2.6)	180.6 (2.6)
Leaf width (mm)	9.3 (0.2)	10.2 (0.1)
Rhizome diameter (mm)	15.1 (0.3)	16.4 (0.3)
S. lancifolia		
Leaf base length (cm)	23.4 (0.3)	42.4 (0.8)
Petiole length (cm)	28.0 (0.4)	55.6 (0.7)
Lamina length (cm)	14.3 (0.2)	16.1 (0.3)
Lamina width (mm)	23.0 (0.8)	22.6 (1.2)

Table 4.23. Covariance of parameters used to study morphological variation in *Cladium jamaicense* and *Sagittaria lancifolia* collected from the Florida Everglades.

	Cycle 4			**Cycle 5**		
C. jamaicense	Leaf l.	Leaf w.	Rhiz. d.	Leaf l.	Leaf w.	Rhiz. d.
No. leaves	0.39	0.53	0.64	0.58	0.62	0.82
Leaf length		0.83	0.81		0.78	0.73
Leaf width			0.88			0.81
S. lancifolia	Petiole l.	Lamina l.	Lamina w.	Petiole l.	Lamina l.	Lamina w.
Lf. Base length	0.58	0.58	0.35	0.16	0.50	0.41
Petiole length		0.07	-0.20		-0.14	-0.24
Lamina length			0.73			0.80

Table 4.24. Eigenvectors of the first (PC1) and second (PC2) principal components from an analysis of morphometric variation in *Cladium jamaicense* and *Sagittaria lancifolia* collected from the Florida Everglades.

	Cycle 4		**Cycle 5**	
C. jamaicense	**PC1**	**PC2**	**PC1**	**PC2**
No. leaves	0.40	0.86	0.47	0.72
Leaf length	0.50	-0.45	0.49	-0.55
Leaf width	0.53	-0.21	0.51	-0.37
Rhiz. Diameter	0.55	-0.01	0.53	0.21
S. lancifolia	PC1	PC2	PC1	PC2
Leaf base length	0.56	0.37	0.46	0.48
Petiole length	0.23	0.76	-0.14	0.86
Lamina length	0.61	-0.22	0.63	-0.01
Lamina width	0.51	-0.49	0.61	-0.16

Table 4.25. Results from a nested analysis of variance of first (PC1) and second (PC2) principal components of morphological data from *Cladium jamaicense* collected in the Florida Everglades.

	Source	**Df**	**Type III SS**	**MS**	***F***	***P***
PC1						
Cycle 4	Site	84	1390	16.5	24.13	0.0001
	Plant (Site)	340	233	0.7	0.53	1.0
	Error	181	234	1.3		
Cycle 5	Site	93	1194	12.8	13.38	0.0001
	Plant (Site)	373	358	0.9	0.53	0.9
	Error	55	100	1.8		
PC2						
Cycle 4	Site	84	205	2.4	7.33	0.0001
	Plant (Site)	340	113	0.3	0.75	0.9
	Error	181	81	0.4		
Cycle 5	Site	93	155	1.7	7.62	0.0001
	Plant (Site)	373	82	0.2	0.69	0.9
	Error	55	18	0.3		

Table 4.26. Results from a nested analysis of variance of first (PC1) and second (PC2) principal components of morphological data from *Sagittaria lancifolia* collected in the Florida Everglades.

	Source	df	Type III SS	MS	*F*	*P*
PC1						
Cycle 4	Site	58	1213	20.9	15.18	0.0001
	Plant (Site)	246	339	1.4	2.99	0.0001
	Leaf (Plant)	38	18	0.5	2.17	0.0001
	Error	475	101	0.2		
Cycle 5	Site	58	524	9.0	18.22	0.0001
	Plant (Site)	216	107	0.5	0.74	0.9
	Error	22	15	0.7		
PC2						
Cycle 4	Site	58	882	15.2	35.11	0.0001
	Plant (Site)	246	107	0.4	2.69	0.0002
	Leaf (Plant)	38	6	0.2	1.27	0.1
	Error	475	1161	0.1		
Cycle 5	Site	58	251	4.3	12.69	0.0001
	Plant (Site)	216	74	0.3	0.95	0.6
	Error	22	336	0.4		

Table 4.27. Differences among subareas in *Cladium jamaicense* morphological parameters. Mean (S.E.) of site averages for each subarea; N = no. of sites/subarea.

	Subarea							
Parameter	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**P***
No. of Leaves	5 (0.4)	7 (0.3)	7 (0.4)	6 (0.3)	5 (0.3)	5 (0.2)	5 (0.2)	<0.0001
Total Leaf Length (cm)	177 (17)	205 (9)	190 (16)	215 (7)	212 (12)	157 (7)	121 (6)	<0.0001
Leaf Width (mm)	10 (0.7)	12 (0.4)	11 (0.7)	12 (0.5)	12 (0.8)	8 (0.4)	7 (0.3)	<0.0001
Culm Diameter (mm)	16 (1.2)	19 (1.0)	19 (1.3)	21 (1.1)	21 (1.5)	13 (0.7)	10 (0.5)	<0.0001
N:	15	22	20	30	16	48	26	

*Probability of a greater Chi square value in Kruskal-Wallis test.

Table 4.28. Differences among subareas in *Sagittaria lancifolia* morphological parameters. Mean (S.E.) of site averages for each subarea; N = no. of sites/subarea.

	Subarea							
Parameter	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**P***
Total Leaf Length (cm)	81 (10)	104 (7)	91 (7)	92 (7)	113 (10)	81 (6)	76 (8)	0.0985
Leaf Base Length (cm)	29 (5)	38 (3)	36 (3)	34 (3)	42 (5)	28 (2)	26 (3)	0.0692
Petiole Length (cm)	38 (5)	48 (4)	36 (4)	47 (4)	57 (6)	43 (3)	35 (5)	0.0254
Petiole Width (mm)	5 (0.8)	5 (0.4)	6 (0.3)	5 (0.3)	4 (0.3)	4 (0.2)	5 (0.4)	<0.0001
Lamina Length (cm)	14 (2)	18 (1)	19 (1)	12 (1)	15 (2)	11 (1)	15 (1)	<0.0001
Lamina Width (cm)	2.3 (1.0)	3.5 (0.4)	4.1 (0.4)	1.1 (0.2)	1.3 (0.2)	0.6 (0.1)	1.4 (0.3)	<0.0001
Culm Diameter (cm)	2.7 (0.3)	3.1 (0.2)	3.1 (0.2)	2.6 (0.1)	2.6 (0.2)	2.5 (0.1)	2.8 (0.2)	0.1044
Culm FW/L (g/cm)	12 (2)	14 (2)	15 (2)	11 (1)	14 (3)	11 (1)	11 (2)	0.2674
N:	8	12	25	20	7	26	11	

*Probability of a greater Chi square value in Kruskal-Wallis test.

Table 4.29. Means (S.E.) of soil physical and chemical characteristics at sites in the Florida Everglades from which *Cladium jamaicense* and *Sagittaria lancifolia* were collected.

	Cycle 4	Cycle 5
C. jamaicense		
Total phosphorus (Fg/g)	251.1 (16.5)	260.8 (14.9)
Alkaline phosphatase (Fmole/g)	7.8 (1.6)	12.4 (2.4)
% Ash-free dry weight	63.9 (3.1)	70.7 (2.4)
Bulk density (g/cm^3)	0.91 (0.11)	0.30 (0.02)
% Mineral content	36.2 (3.1)	29.3 (2.4)
S. lancifolia		
Total phosphorus (Fg/g)	286.8 (27.1)	283.6 (20.9)
Alkaline phosphatase (Fmole/g)	7.6 (1.4)	8.5 (2.3)
% Ash-free dry weight	63.5 (3.6)	67.2 (3.0)
Bulk density (g/cm^3)	0.81 (0.01)	0.33 (0.03)
% Mineral content	36.6 (3.6)	32.8 (3.0)

Table 4.30. Covariance of soil physical and chemical characteristics at sites in the Florida Everglades from which *Cladium jamaicense* and *Sagittaria lancifolia* were collected.

	Cycle 4				Cycle 5			
	AP	**AFDW**	**BD**	**MC**	**AP**	**AFDW**	**BD**	**MC**
C. jamaicense								
TP	0.43	0.43	-0.16	-0.43	-0.10	0.35	-0.28	-0.35
AP		0.35	0.04	-0.35		0.31	-0.18	-0.31
AFDW			-0.65	-1.00			-0.64	-1.00
BD				0.65				0.64
S. lancifolia								
TP	0.54	0.34	0.00	-0.34	-0.14	0.42	-0.29	-0.42
AP		0.36	0.17	-0.36		0.33	-0.20	-0.33
AFDW			-0.52	-1.00			-0.61	-1.00
BD				0.52				0.61

TP = total phosphorus
AP = alkaline phosphatase
AFDW = % ash-free dry weight
BD = bulk density
MC = % mineral content

Table 4.31. Eigenvectors of the first (PC1) and second (PC2) principal components from analysis of soil physical and chemical characteristics at sites from which *Cladium jamaicense* and *Sagittaria lancifolia* were collected in the Florida Everglades.

	Cycle 4		Cycle 5	
	PC1	**PC2**	**PC1**	**PC2**
C. jamaicense				
TP	0.35	0.47	0.28	-0.65
AP	0.27	0.68	0.23	0.75
AFDW	0.56	-0.12	0.57	0.03
BD	-0.41	0.52	-0.47	0.06
MC	-0.56	0.12	-0.57	-0.04
S. lancifolia				
TP	0.34	0.50	0.31	-0.63
AP	0.32	0.60	0.23	0.77
AFDW	0.58	-0.15	0.57	0.03
BD	-0.32	0.59	-0.45	0.02
MC	-0.58	0.15	-0.57	-0.03

TP = total phosphorus
AP = alkaline phosphatase
AFDW = % ash-free dry weight
BD = bulk density
MC = % mineral content.

Table 4.32. Spearman's rank-sum correlation coefficients for the relationships between the first (PC1) and second (PC2) principal component scores for plant morphometric data and soil physicochemical data at sites in the Florida Everglades from which *Cladium jamaicense* and *Sagittaria lancifolia* were collected. [a]$P < 0.01$, [b]$P < 0.0001$.

	Cycle 4		Cycle 5	
	PC1-soil	**PC2-soil**	**PC1-soil**	**PC2-soil**
C. jamaicense				
PC1-plant	0.58[b]	-0.35[b]	0.43[b]	0.12[a]
PC2-plant	-0.12[a]	0.30[b]	-0.29[b]	-0.23[b]
S. lancifolia				
PC1-plant	0.33[b]	0.11[a]	N.S.	-0.41[b]
PC2-plant	N.S.	-0.49[b]	N.S.	0.45[b]

Table 4.33. Pairwise Spearman's rank-sum correlation coefficients for plant morphological and soil physicochemical parameters used in principal component analysis based on *Cladium jamaicense* and *Sagittaria lancifolia* plants collected in the Florida Everglades. [a]$P < 0.05$; [b]$P < 0.01$; [c]$P < 0.001$; [d]$P < 0.0001$.

	Cycle 4					**Cycle 5**				
C. jamaicense	**TP**	**AP**	**AFDW**	**BD**	**MC**	**TP**	**AP**	**AFDW**	**BD**	**MC**
No. leaves	0.25[d]	0.25[d]	0.31[d]	-0.18[d]	-0.31[d]	0.26[d]	N.S.	0.29[d]	-0.09[a]	-0.29[d]
Leaf length	0.30[d]	0.11[a]	0.51[d]	-0.65[d]	-0.51[d]	0.16[c]	0.35[d]	0.53[d]	-0.40[d]	-0.53[d]
Leaf width	0.27[d]	0.17[d]	0.49[d]	-0.57[d]	-0.49[d]	0.23[d]	0.33[d]	0.55[d]	-0.41[d]	-0.55[d]
Rhiz. diameter	0.25[d]	0.15[c]	0.49[d]	-0.58[d]	-0.49[d]	0.24[d]	0.30[d]	0.46[d]	-0.28[d]	-0.46[d]
S. lancifolia	**TP**	**AP**	**AFDW**	**BD**	**MC**	**TP**	**AP**	**AFDW**	**BD**	**MC**
Leaf base length	0.23[d]	N.S.	0.13[c]	-0.28[d]	-0.13[c]	N.S.	N.S.	N.S.	0.16[b]	N.S.
Petiole length	0.08[a]	-0.21[d]	0.12[c]	-0.51[d]	-0.12[c]	-0.33[d]	0.29[d]	N.S.	N.S.	N.S.
Lamina length	0.23[d]	0.30[d]	0.12[c]	0.08[d]	-0.12[c]	0.32[d]	-0.27[d]	N.S.	0.18[b]	N.S.
Lamina width	0.32[d]	0.49[d]	0.23[d]	0.17[d]	-0.23[d]	0.44[d]	-0.20[c]	0.26[d]	N.S.	-0.26[d]

Table 4.34. Spearman's rank-sum correlation coefficients for the relationships between *S. lancifolia* leaf % nutrient content and the first (PC1) and second (PC2) principal component scores for plant morphometric data and soil physicochemical data at sites from which plants were collected in the Florida Everglades. Data are from Cycle 4 sampling period only. [a]$P < 0.05$, [b]$P < 0.005$, [c]$P < 0.0001$.

	% C	% N	% P	N:P
PC1-soil	N.S.	-0.15[c]	-0.11[b]	N.S.
PC2-soil	N.S.	0.37[c]	0.49[c]	-0.37[c]
PC1-plant	-0.13[b]	-0.09[a]	0.19[c]	-0.37[c]
PC2-plant	-0.42[c]	-0.67[c]	-0.59[c]	0.33[c]

Table 4.35. Spearman's rank-sum correlation coefficients for the relationships between *C. jamaicense* leaf nutrient content and the first (PC1) and second (PC2) principal component scores for plant morphometric data and soil physicochemical data at sites from which plants were collected in the Florida Everglades. Nutrient data are a subset of Cycle 5 sampling period and represent bulk samples by site. [a]$P = 0.055$, [b]$P < 0.05$, [c]$P < 0.01$, [d]$P < 0.0001$.

	% C	% N	% P	N:P
PC1-soil	-0.37[b]	N.S.	N.S.	N.S.
PC2-soil	N.S.	-0.46[b]	-0.36[a]	N.S.
PC1-plant	N.S.	N.S.	0.21[c]	-0.29[d]
PC2-plant	0.17[b]	0.35[d]	0.19[b]	N.S.

Table 4.36. Mean (S. E.) values for five measures of hydroperiod among seven subareas of the Florida Everglades. Means are based on midpoints of categorized model output (see Methods). Hydroperiod codes: 1) mean annual number of days of inundation, 2) number of days of inundation in 1989, 3) mean annual water depth, 4) mean water depth during the month of May, and 5) mean water depth during the month of October.

Subarea	*n*	1	2	3	4	5
LOX	25	335.7 (8.0)	291.6 (15.5)	0.44 (0.04)	0.19 (0.03)	0.69 (0.04)
WCA2	25	323.8 (5.7)	191.5 (17.1)	0.32 (0.03)	0.18 (0.02)	0.47 (0.04)
WCA3-N	27	292.2 (8.2)	203.3 (11.2)	0.24 (0.03)	0.09 (0.01)	0.40 (0.03)
WCA3-SE	29	332.6 (9.2)	275.6 (17.8)	0.57 (0.04)	0.33 (0.03)	0.71 (0.05)
WCA3-SW	41	342.3 (3.8)	320.2 (6.0)	0.45 (0.03)	0.23 (0.02)	0.60 (0.03)
Shark	50	285.3 (8.5)	158.4 (9.8)	0.18 (0.01)	0.09 (0.01)	0.34 (0.02)
Taylor	25	164.4 (15.6)	85.2 (12.0)	0.06 (0.01)	0.02 (0.01)	0.14 (0.02)
	$^{1}c^{2}$=	122.98	129.21	127.93	105.32	117.45

1 Kruskal-Wallis approximation of Chi-Square test for differences among divisions, df = 6, $P < 0.0001$ for all hydroperiod categories.

LOX = Loxahatchee National Wildlife Refuge or Water Conservation Area 1
WCA2 = Water Conservation Area 2
WCA3-N = Water Conservation Area 3, north
WCA3-SE = Water Conservation Area 3, southeast
WCA3-SW = Water Conservation Area 3, southwest
Shark = Shark River Slough, Everglades National Park
Taylor = Taylor Slough, Everglades National Park.

Table 4.37. Spearman's rank-sum correlation coefficients for relationships among five variables measuring hydroperiod in the Florida Everglades. Analysis is based on midpoints of categorized model output (see Methods). $P < 0.0001$ for all correlations.

	2	3	4	5
1	0.87	0.83	0.76	0.79
2		0.82	0.70	0.79
3			0.83	0.88
4				0.79

Hydroperiod codes: 1) mean annual number of days of inundation, 2) number of days of inundation in 1989, 3) mean annual water depth, 4) mean water depth during the month of May, and 5) mean water depth during the month of October.

Table 4.38. Spearman's rank-sum correlation coefficients for relationships between mean annual water depth and morphological characteristics of *Cladium jamaicense* and *Sagittaria lancifolia*, as well as soil physicochemical characteristics, at sites from which plants were collected in the Florida Everglades. [a]$P < 0.05$; [b]$P < 0.0001$.

		C. jamaicense			***S. lancifolia***	
		Cycle 4	**Cycle 5**		**Cycle 4**	**Cycle 5**
Soil Physicochemical Parameters						
	TP	0.20[b]	0.19[b]	TP	0.19[b]	N.S.
	AP	N.S.	0.53[b]	AP	-0.18[b]	0.28[b]
	AFDW	0.56[b]	0.59[b]	AFDW	0.32[b]	0.47[b]
	BD	-0.81[b]	-0.49[b]	BD	-0.71[b]	-0.46[b]
	MC	-0.56[b]	-0.59[b]	MC	-0.32[b]	-0.47[b]
Morphological Parameters						
	No. leaves	0.20[b]	0.27[b]	Leaf base length	0.28[b]	0.13[a]
	Leaf length	0.70[b]	0.55[b]	Petiole length	0.46[b]	0.23[b]
	Leaf width	0.63[b]	0.56[b]	Lamina length	N.S.	N.S.
	Rhiz. Diameter	0.64[b]	0.45[b]	Lamina width	-0.08[a]	N.S.

TP = total phosphorus
AP = alkaline phosphatase
AFDW = % ash-free dry weight
BD = bulk density
MC = % mineral content.

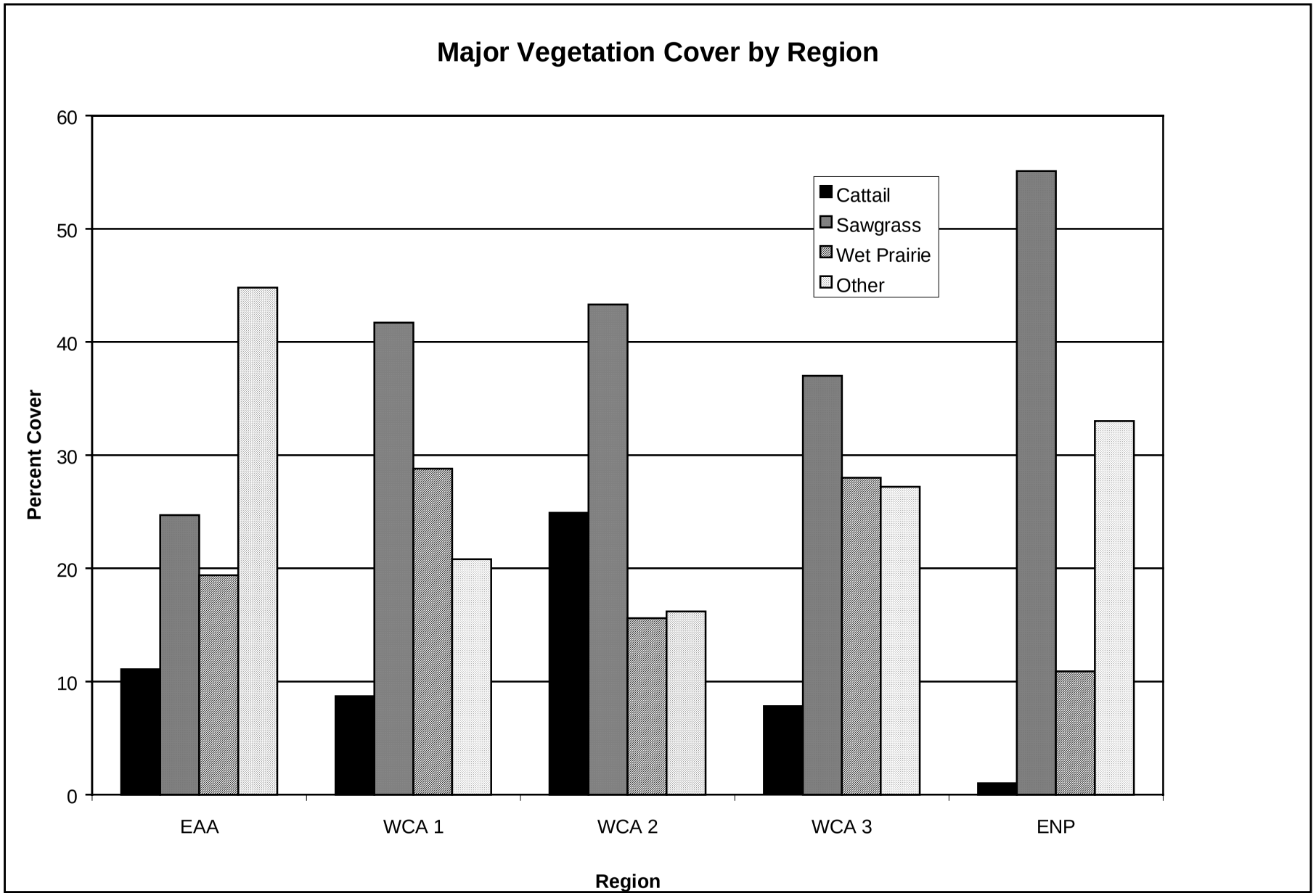


Figure 4.1. Major vegetation cover by region – Cycles 4 and 5 combined.

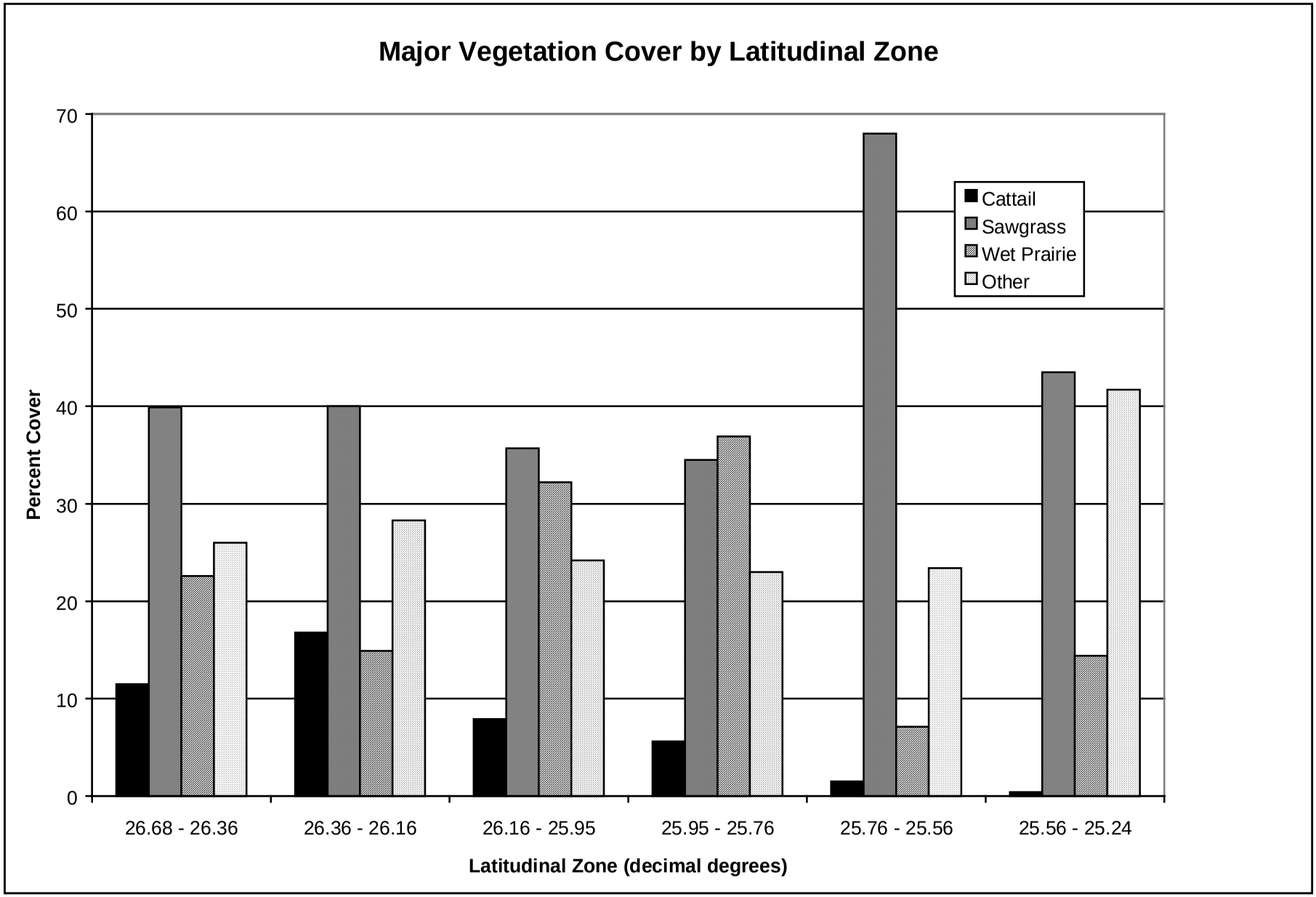


Figure 4.2. Major vegetation cover by latitudinal zone – Cycles 4 and 5 combined.

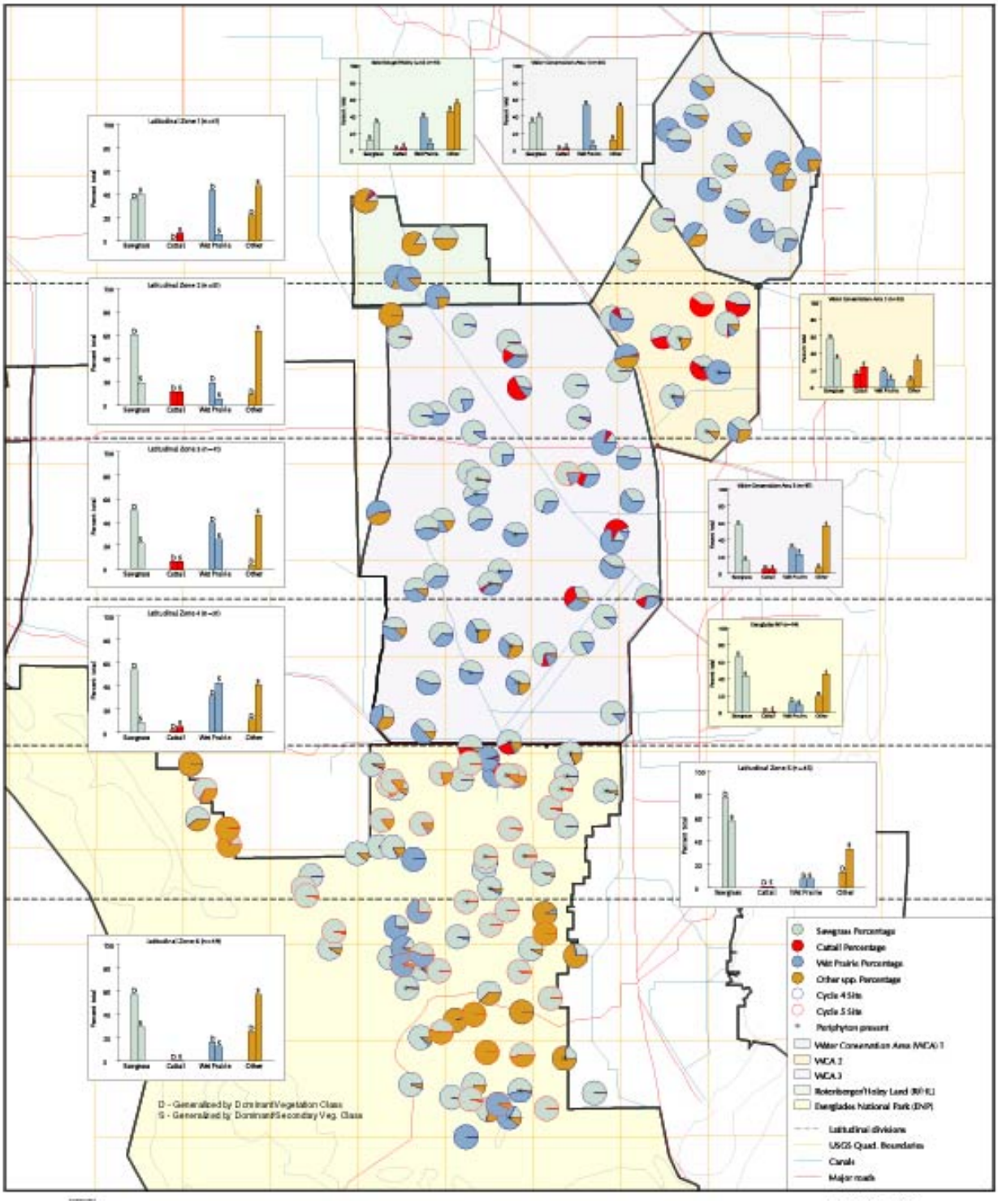


Figure 4.3. Map depicting spatial trends in major vegetation classes and summary statistics.

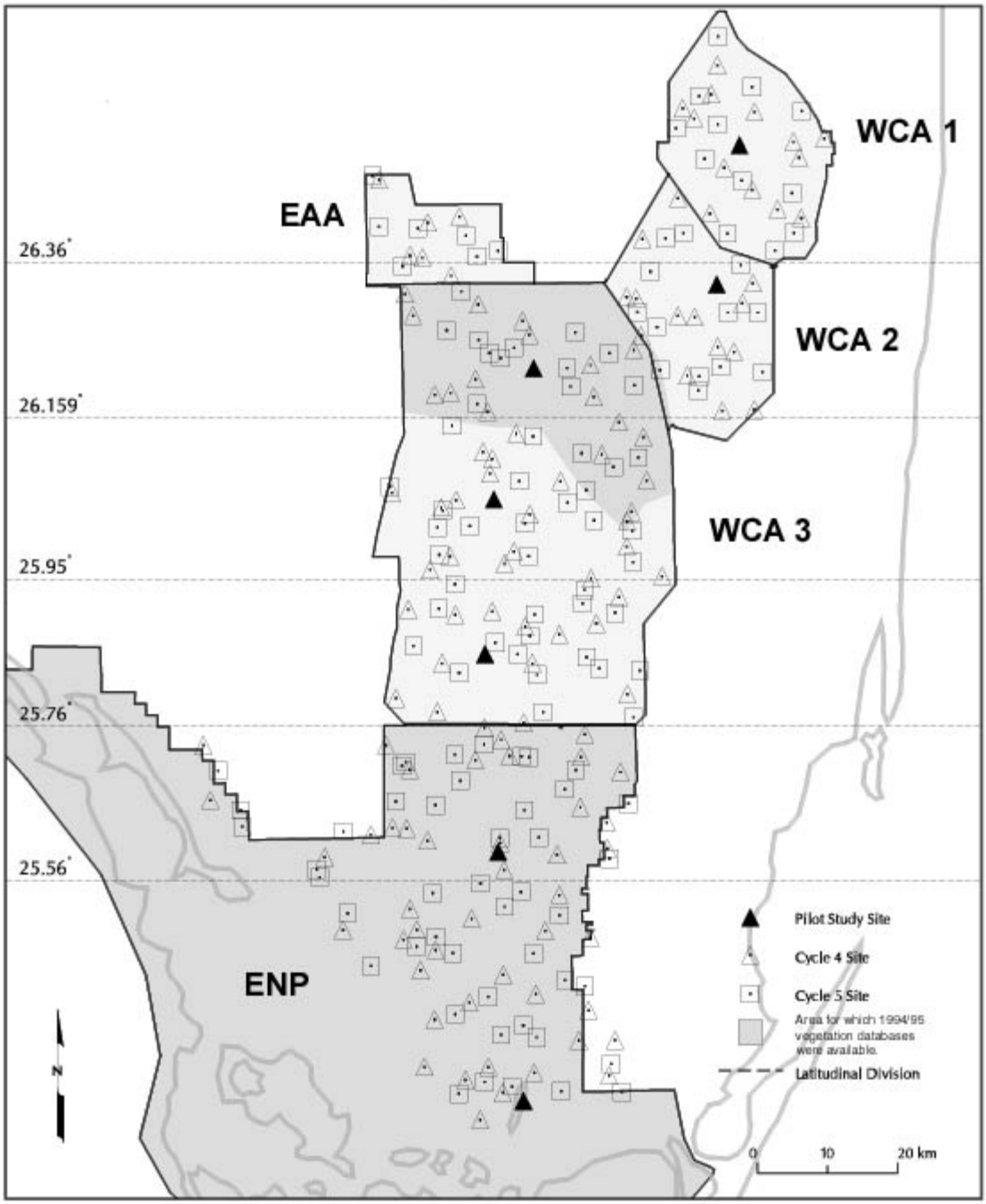


Figure 4.4. EPA South Florida Ecosystem Assessment Project study area and locations of pilot study. Cycle 4 and Cycle 5 monitoring sites.

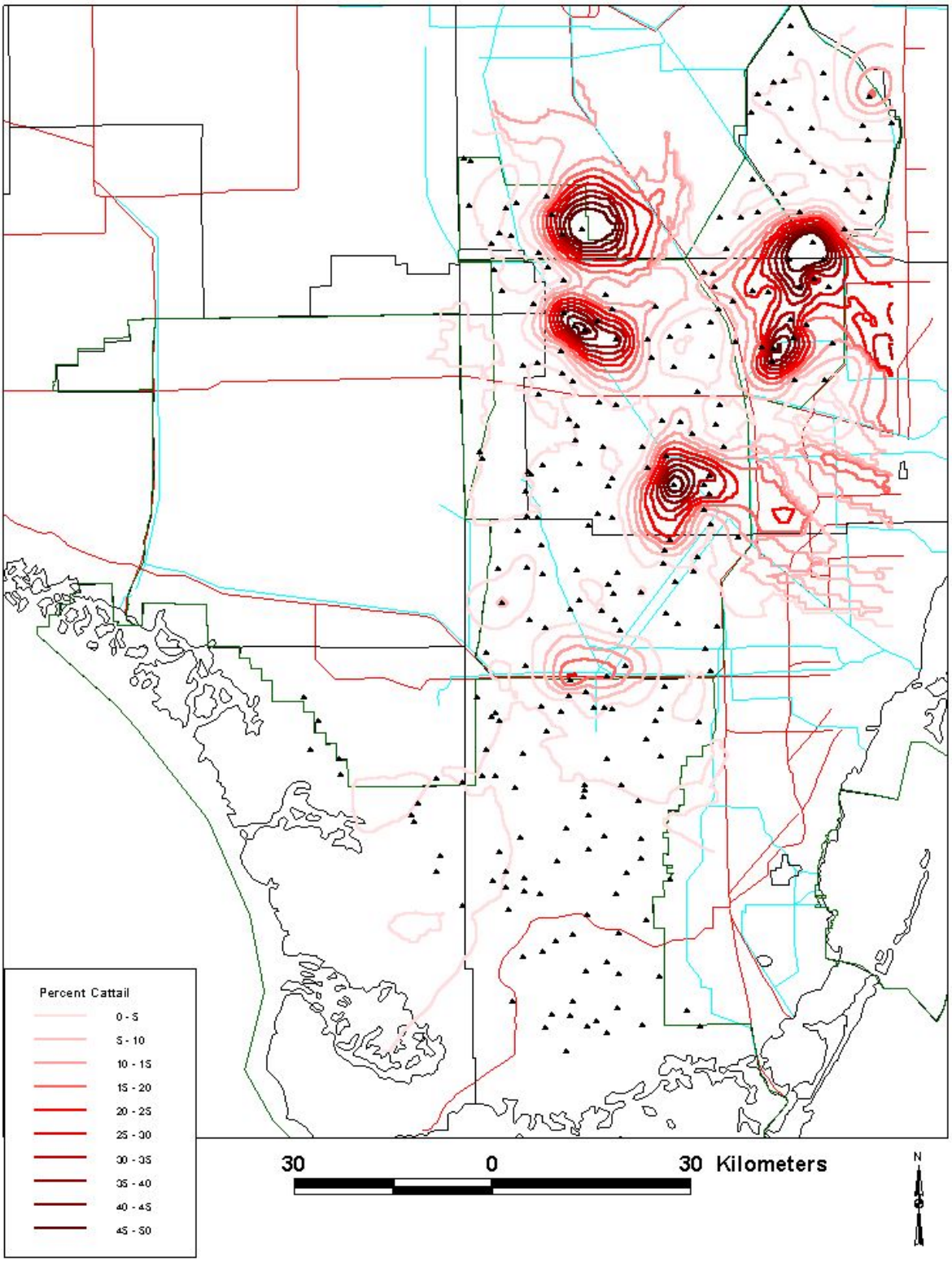


Figure 4.5. Interpolation of cattail percent cover – Cycles 4 and 5.

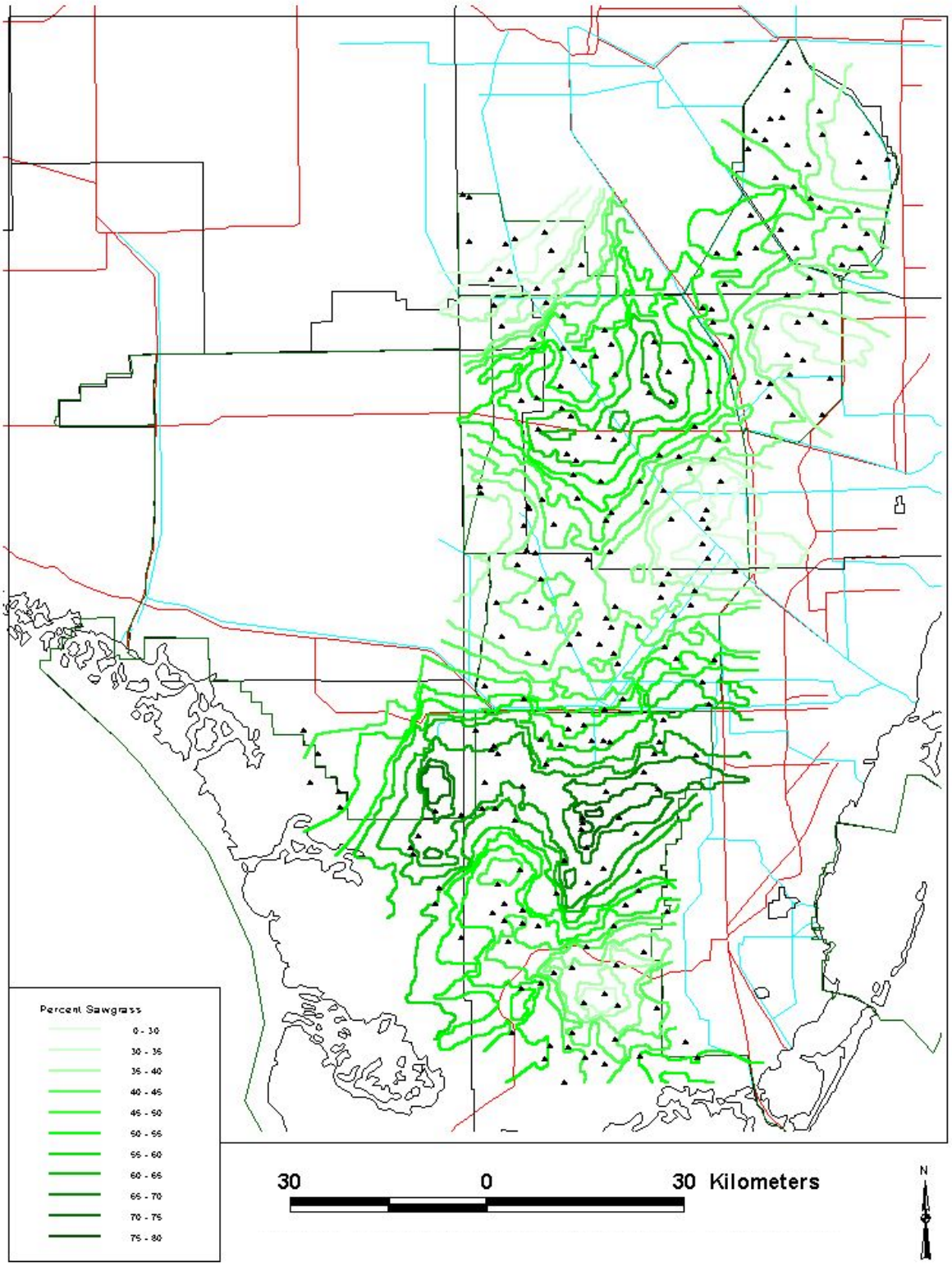


Figure 4.6. Interpolation of sawgrass percent cover – Cycles 4 and 5.

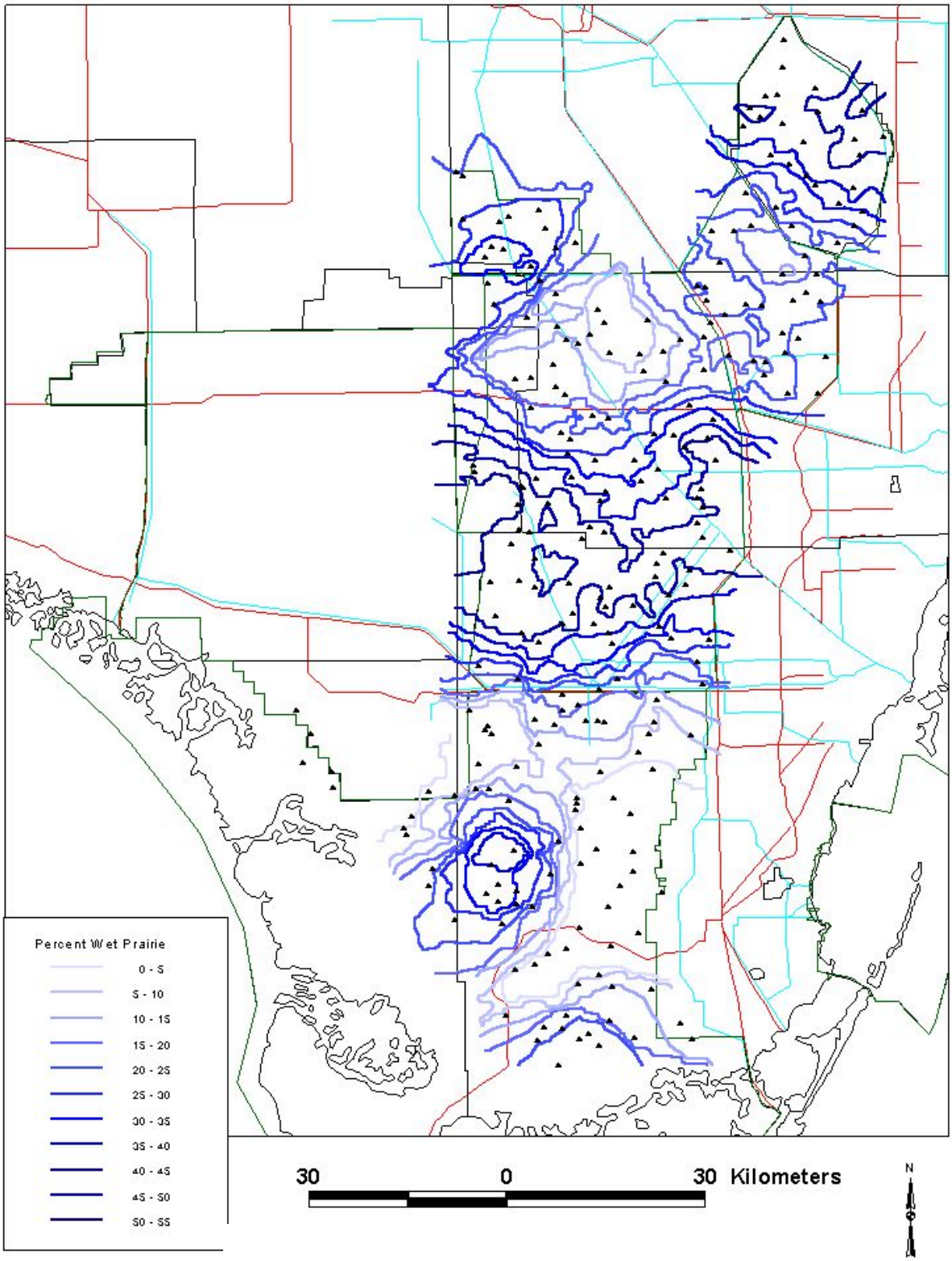


Figure 4.7. Interpolation of wet prairie percent cover – Cycles 4 and 5.

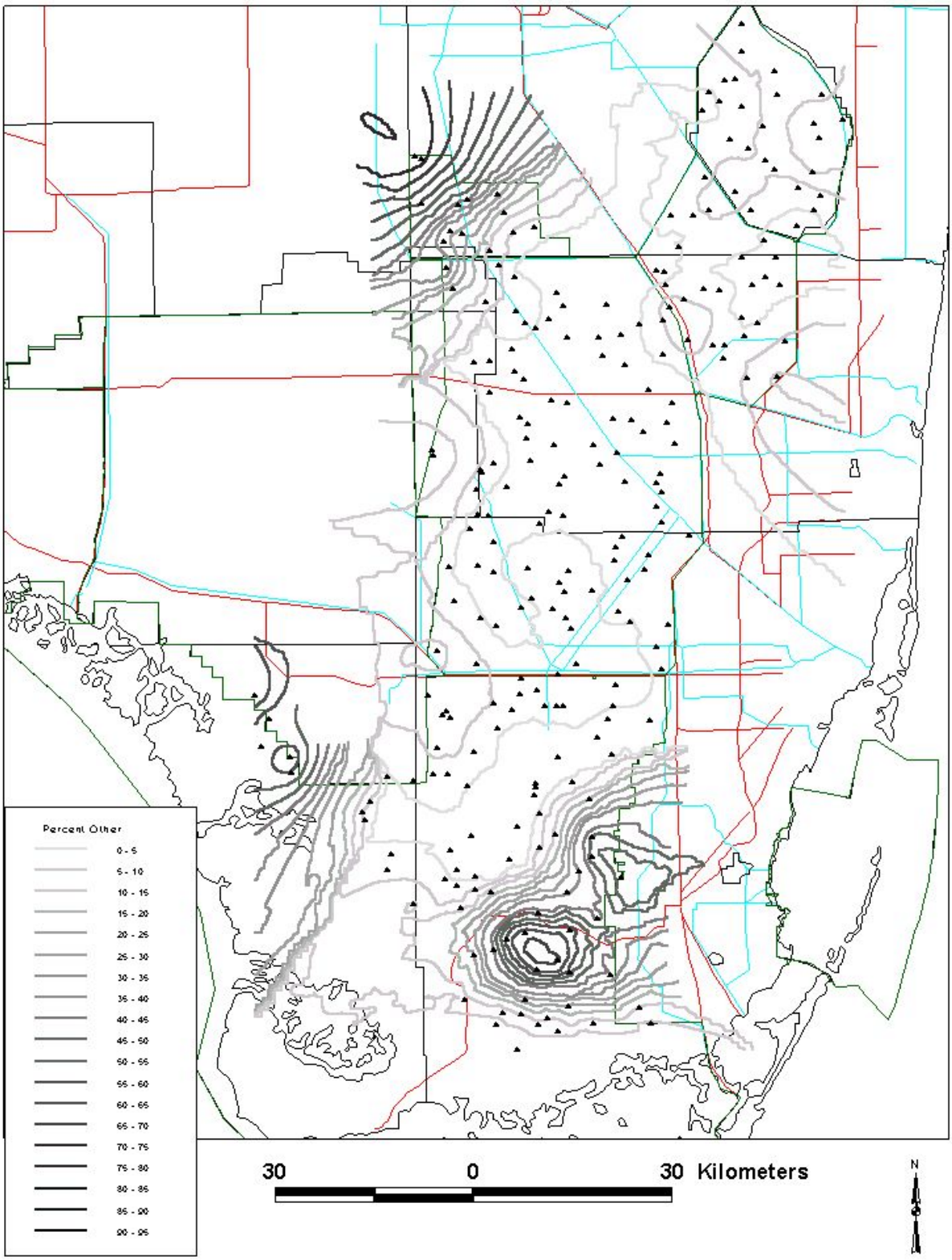


Figure 4.8. Interpolation of “other” vegetation percent cover – Cycles 4 and 5.

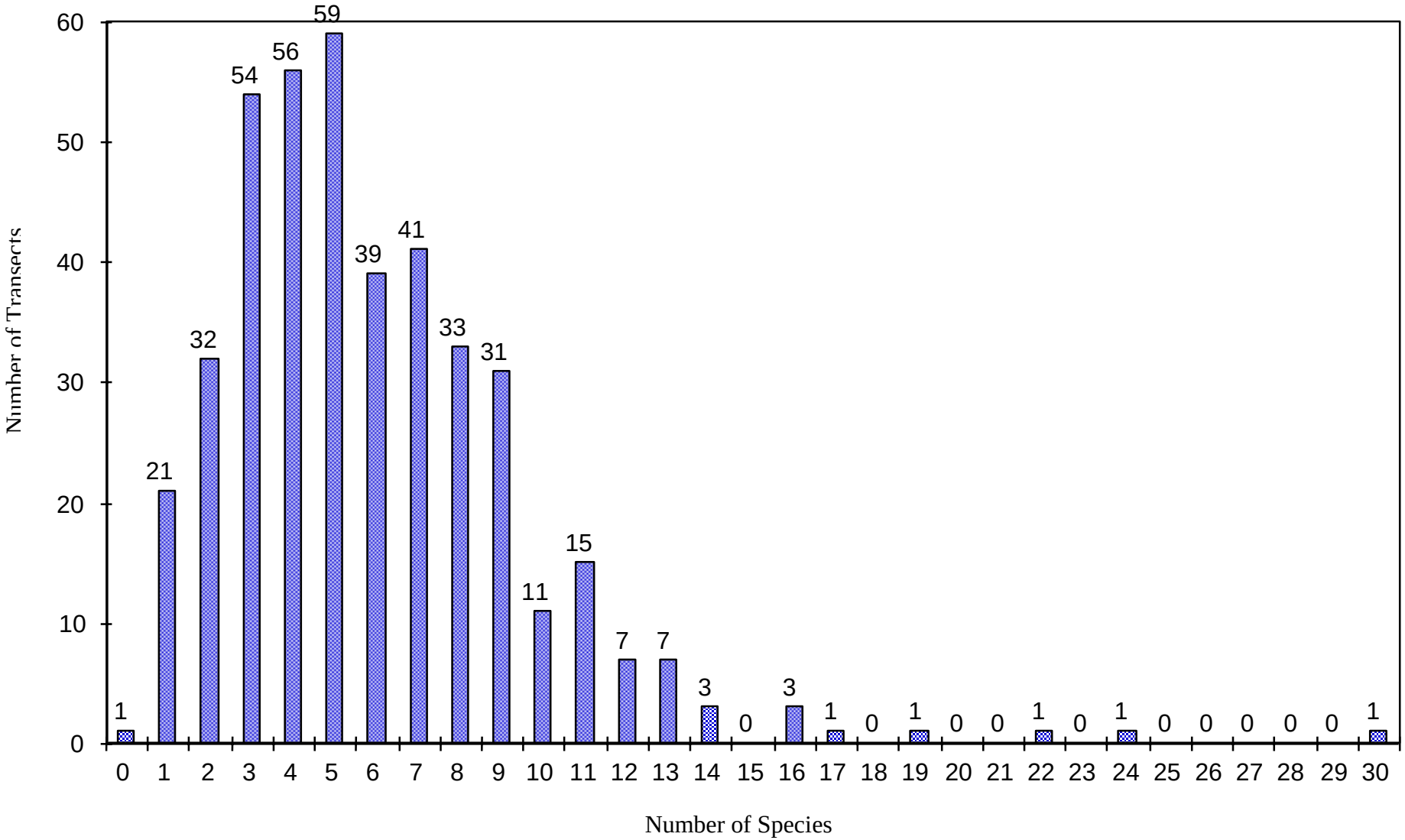


Figure 4.9. Number of species per transect.

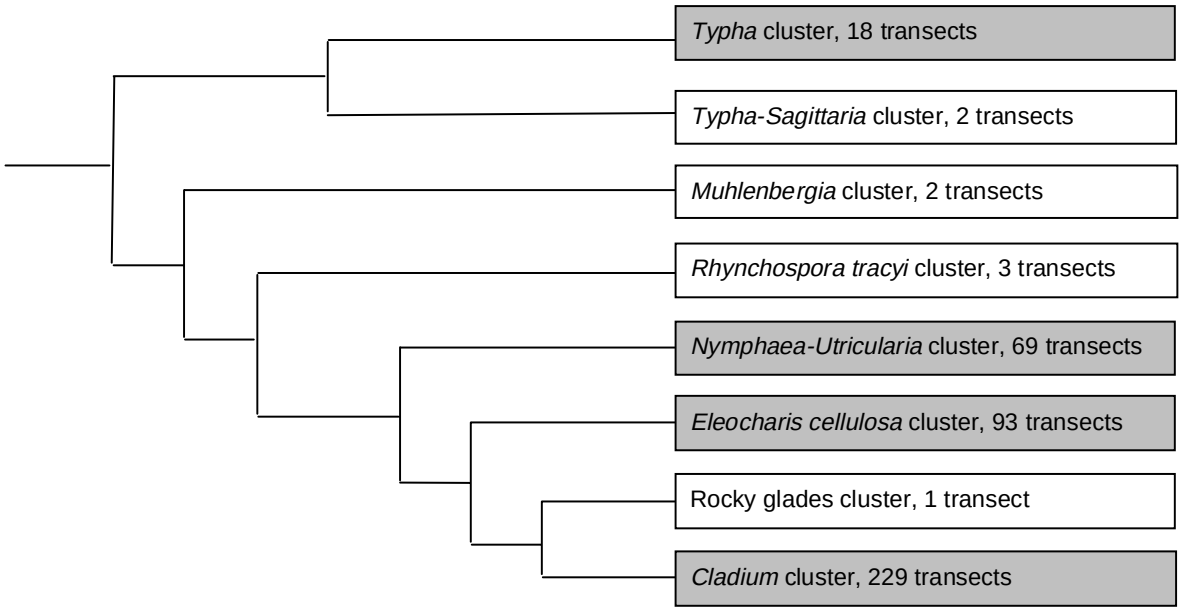


Figure 4.10. Site clusters based on species frequency and abundance.

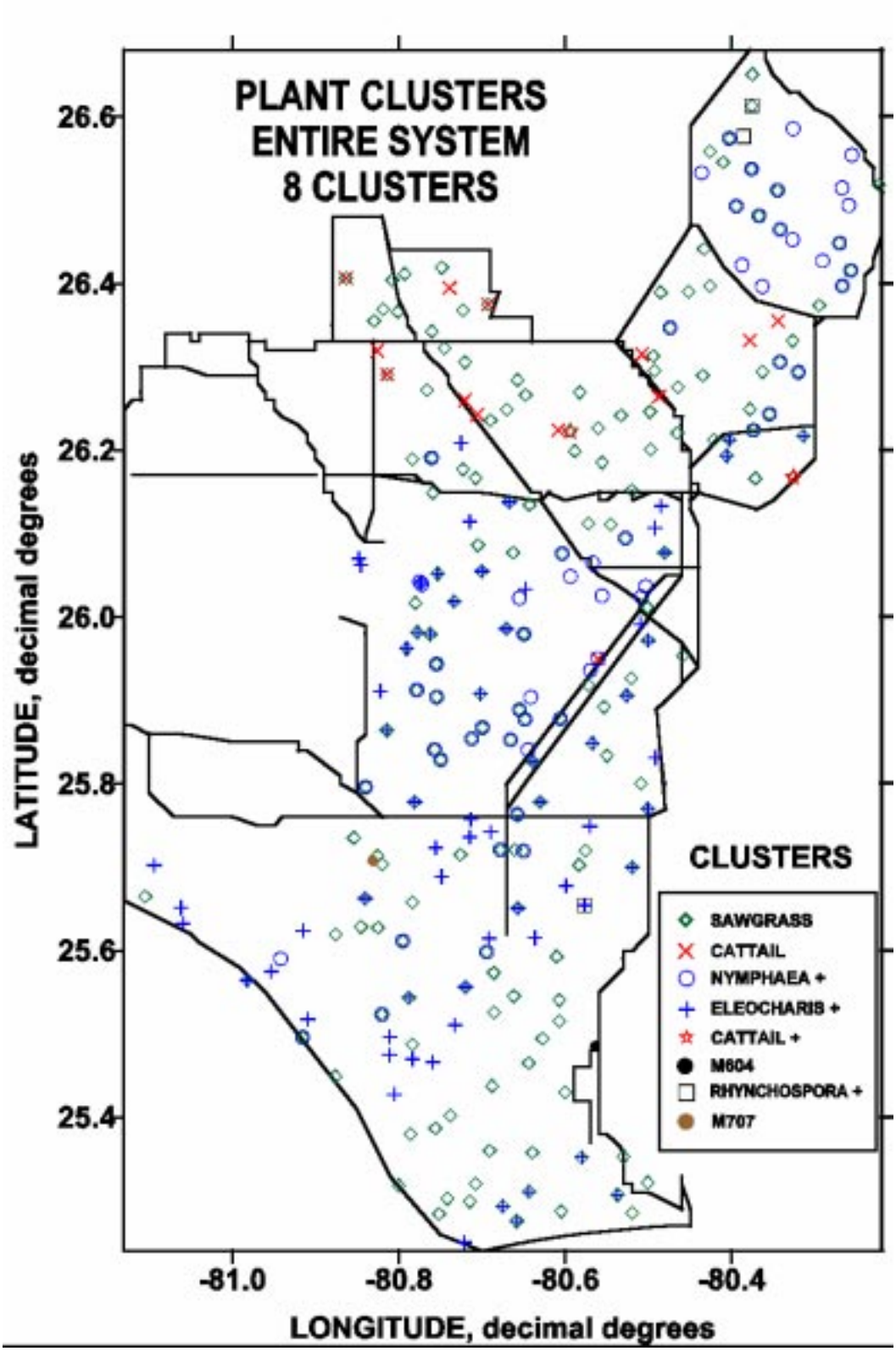


Figure 4.11. Distribution of plant clusters in study area - Cycles 4 and 5.

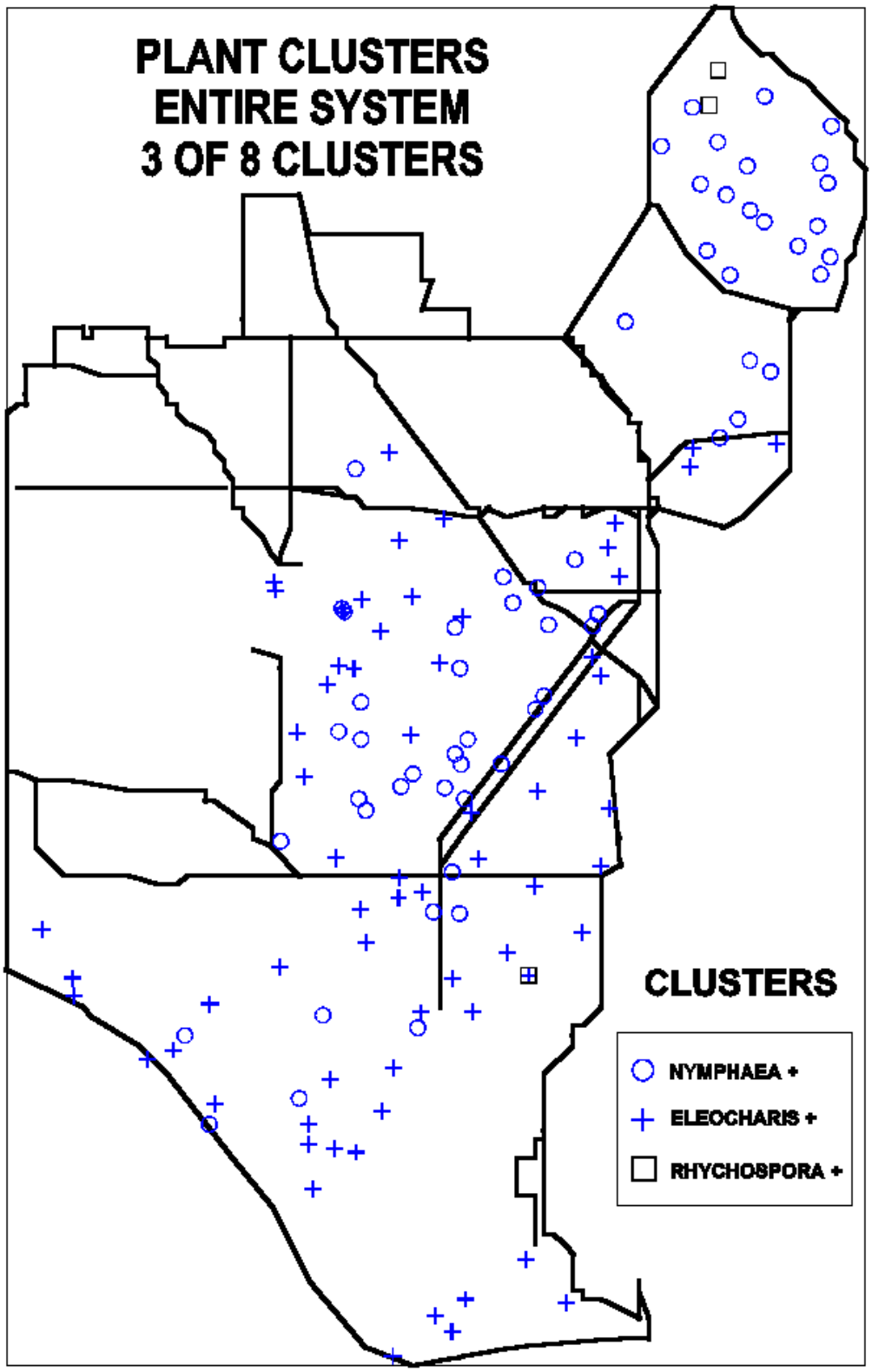


Figure 4.12. Distribution of 3 plant clusters in study area - Cycles 4 and 5.

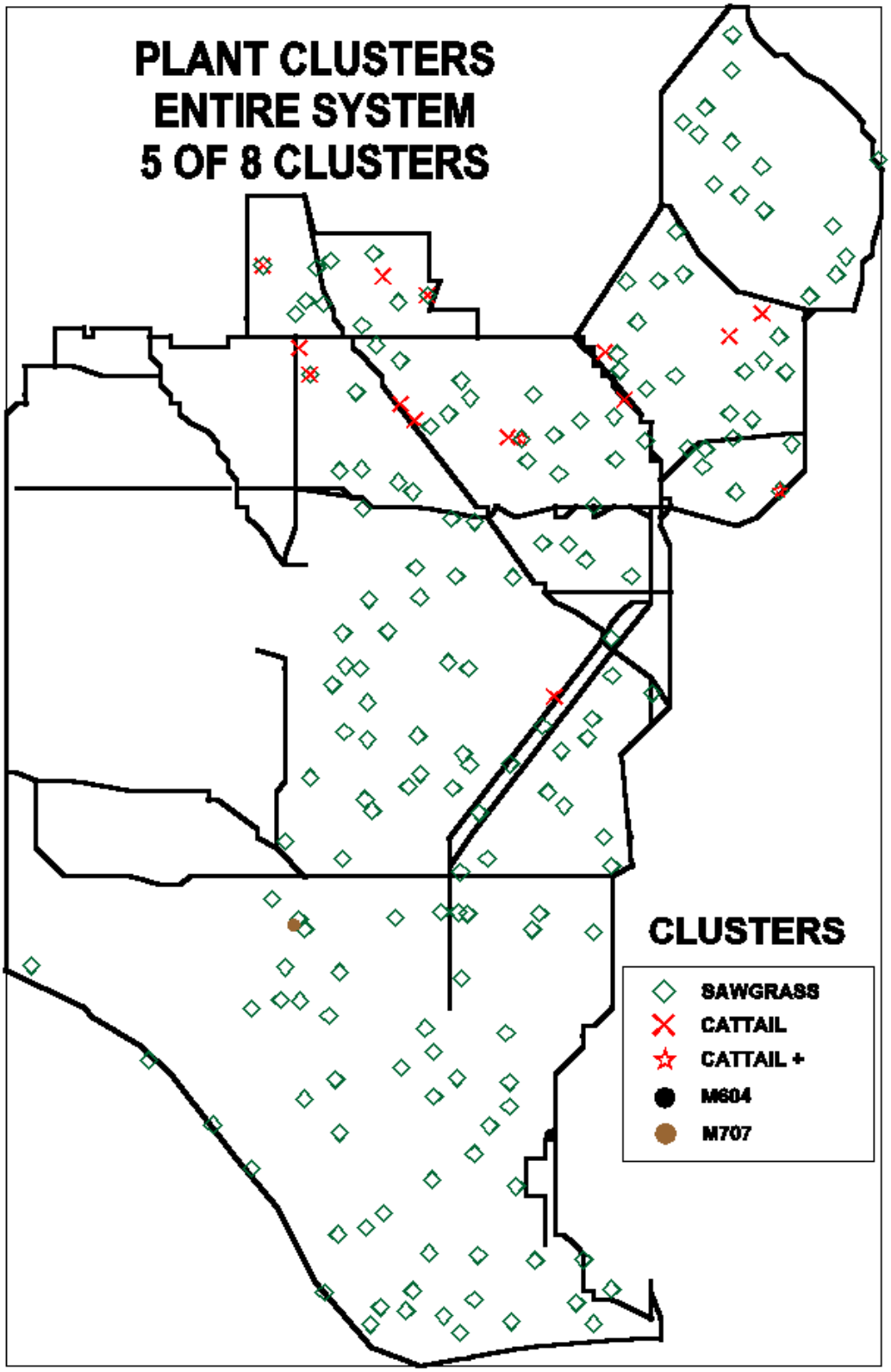


Figure 4.13. Distribution of 5 plant clusters in study area - Cycles 4 and 5.

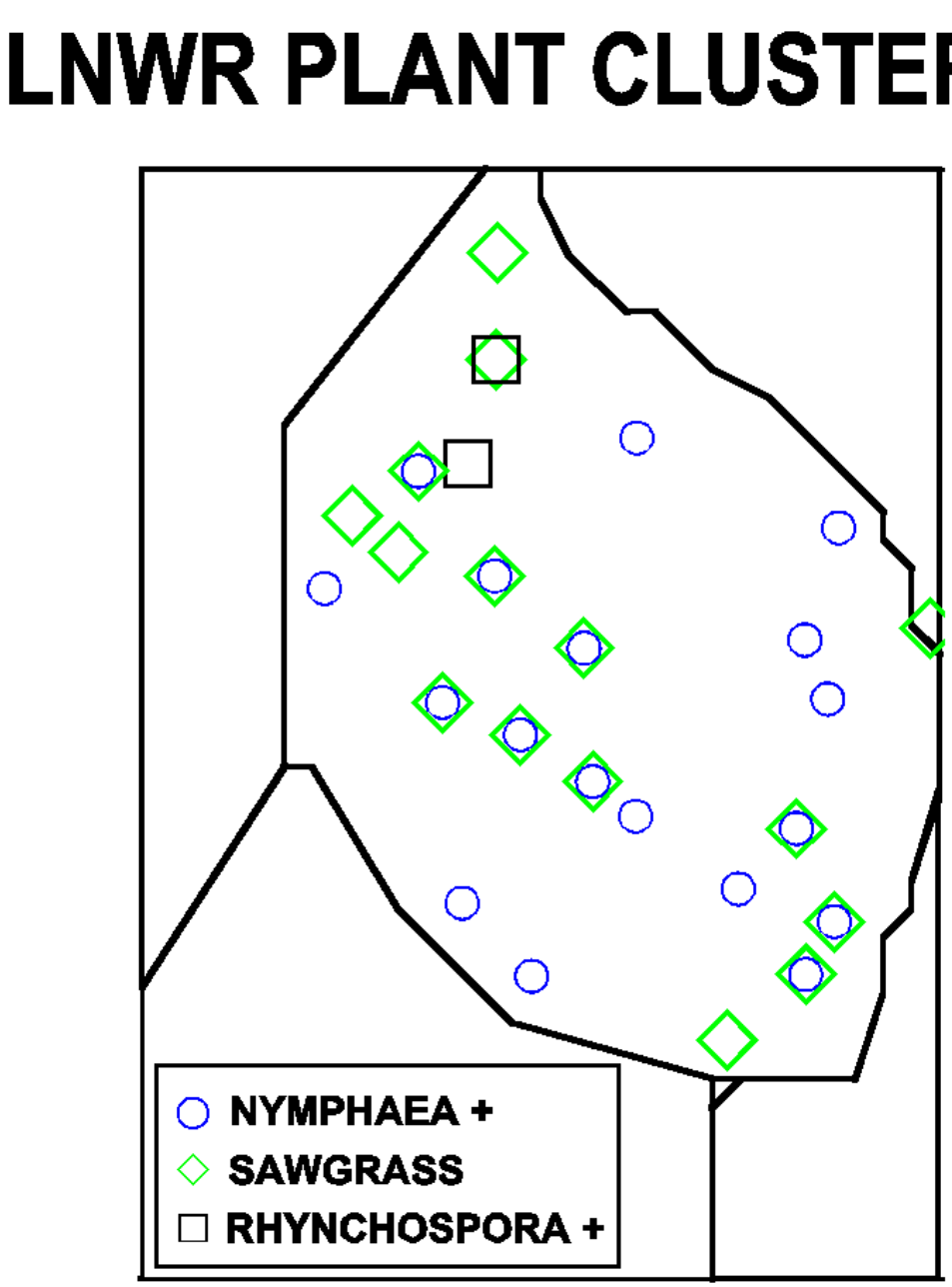


Figure 4.14. Clusters for LNWR.

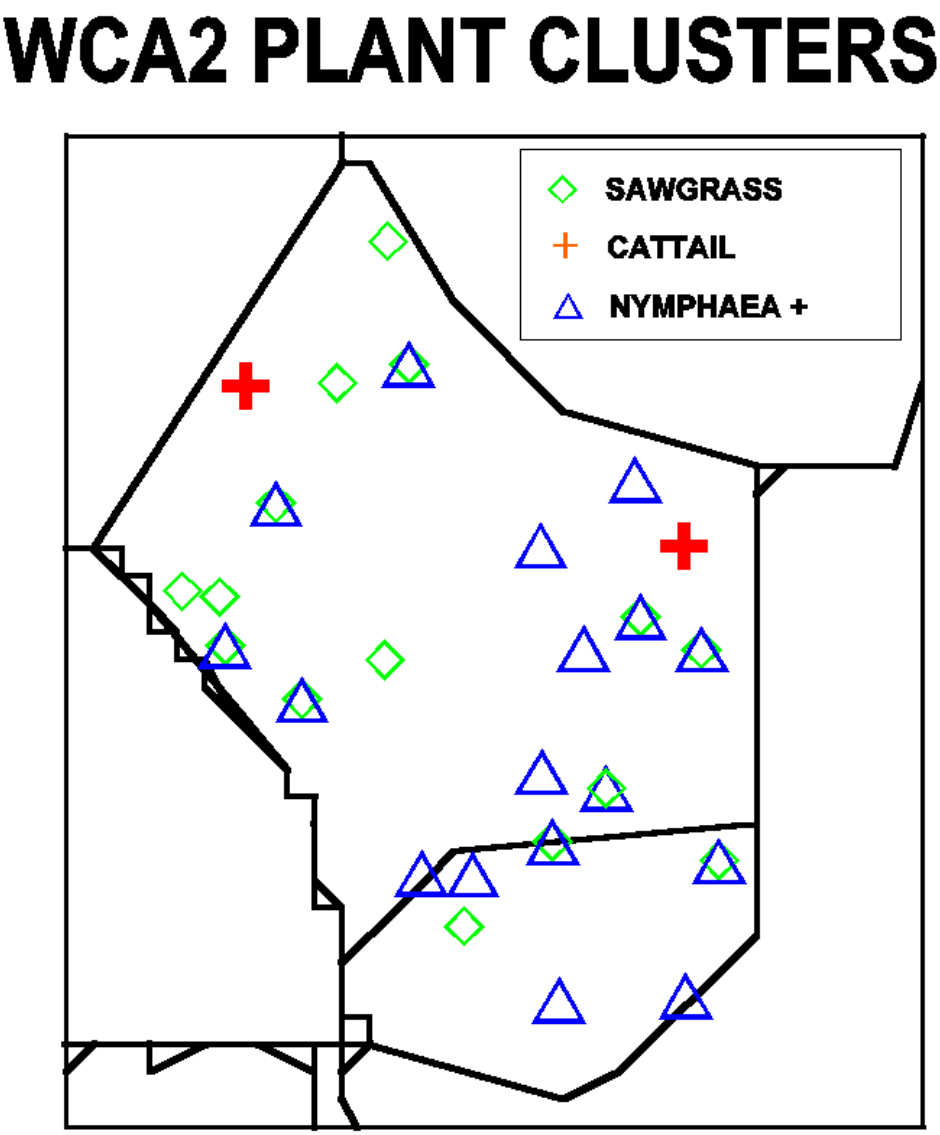


Figure 4.15. Clusters for WCA2.

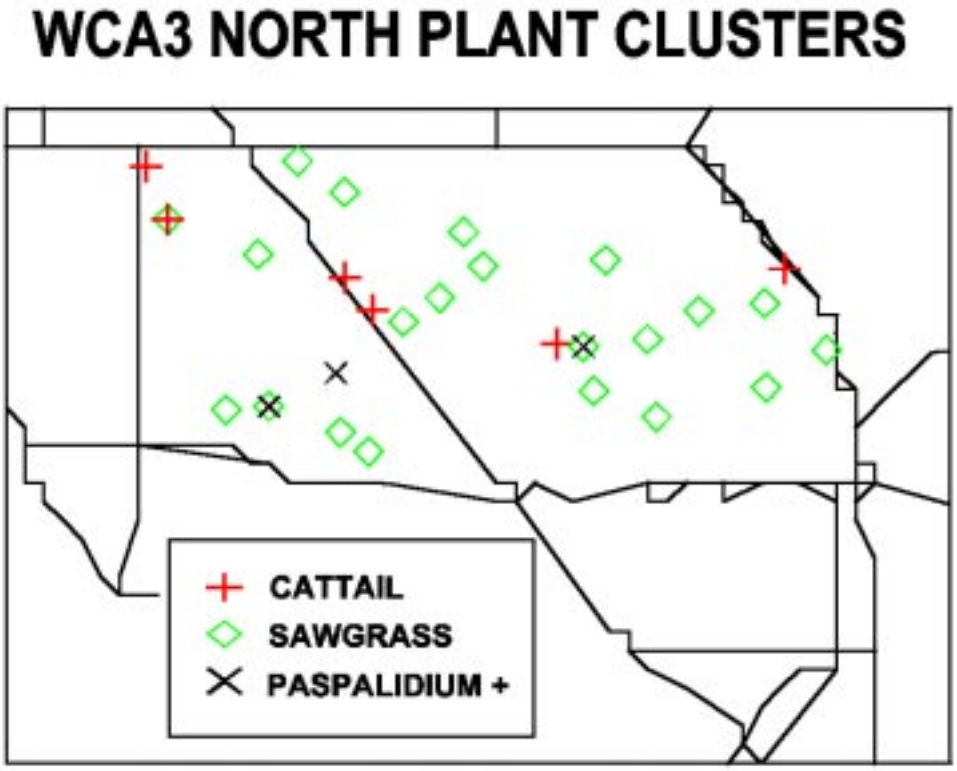


Figure 4.16. Clusters for WCA3 North.

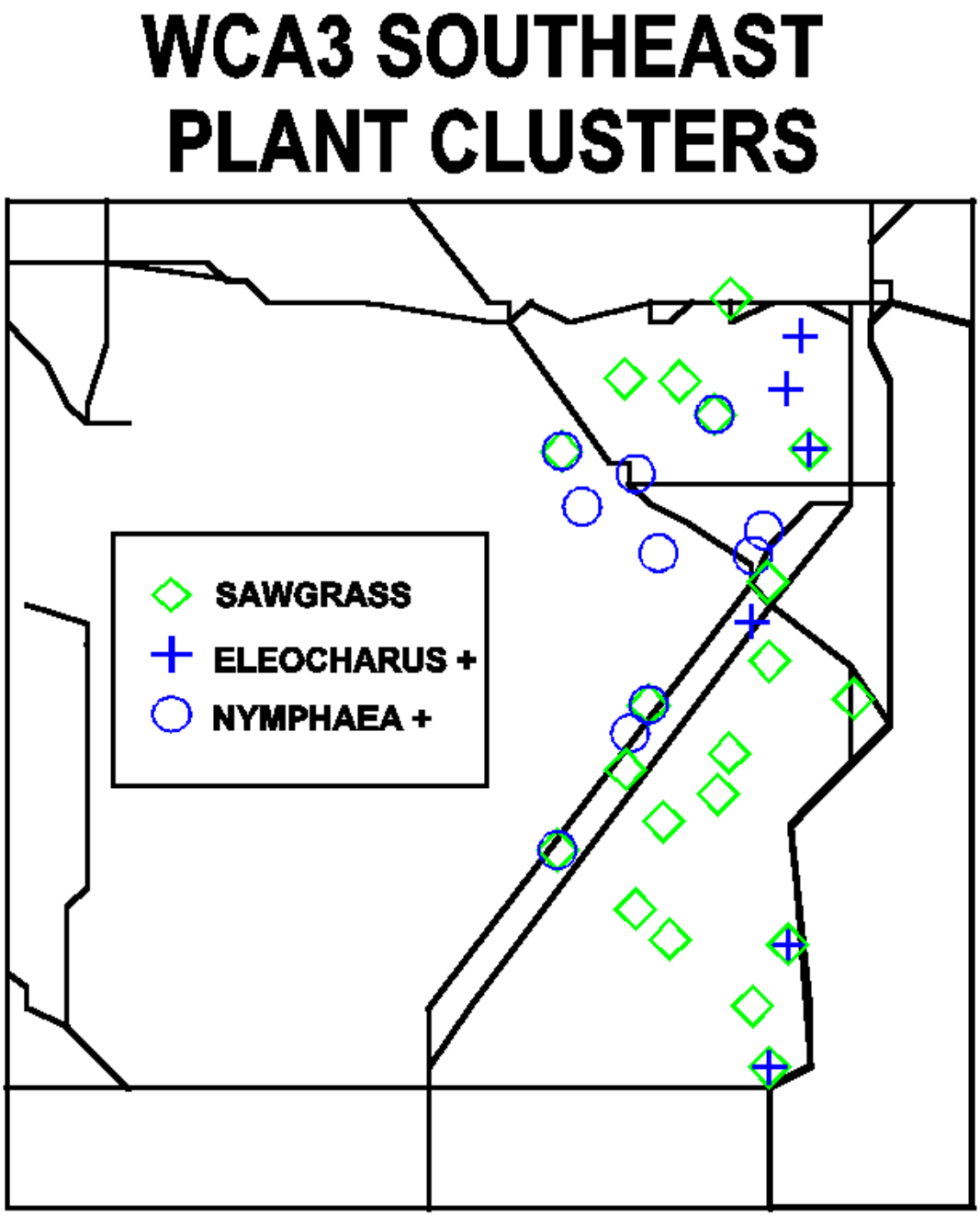


Figure 4.17. Clusters for WCA3 Southeast.

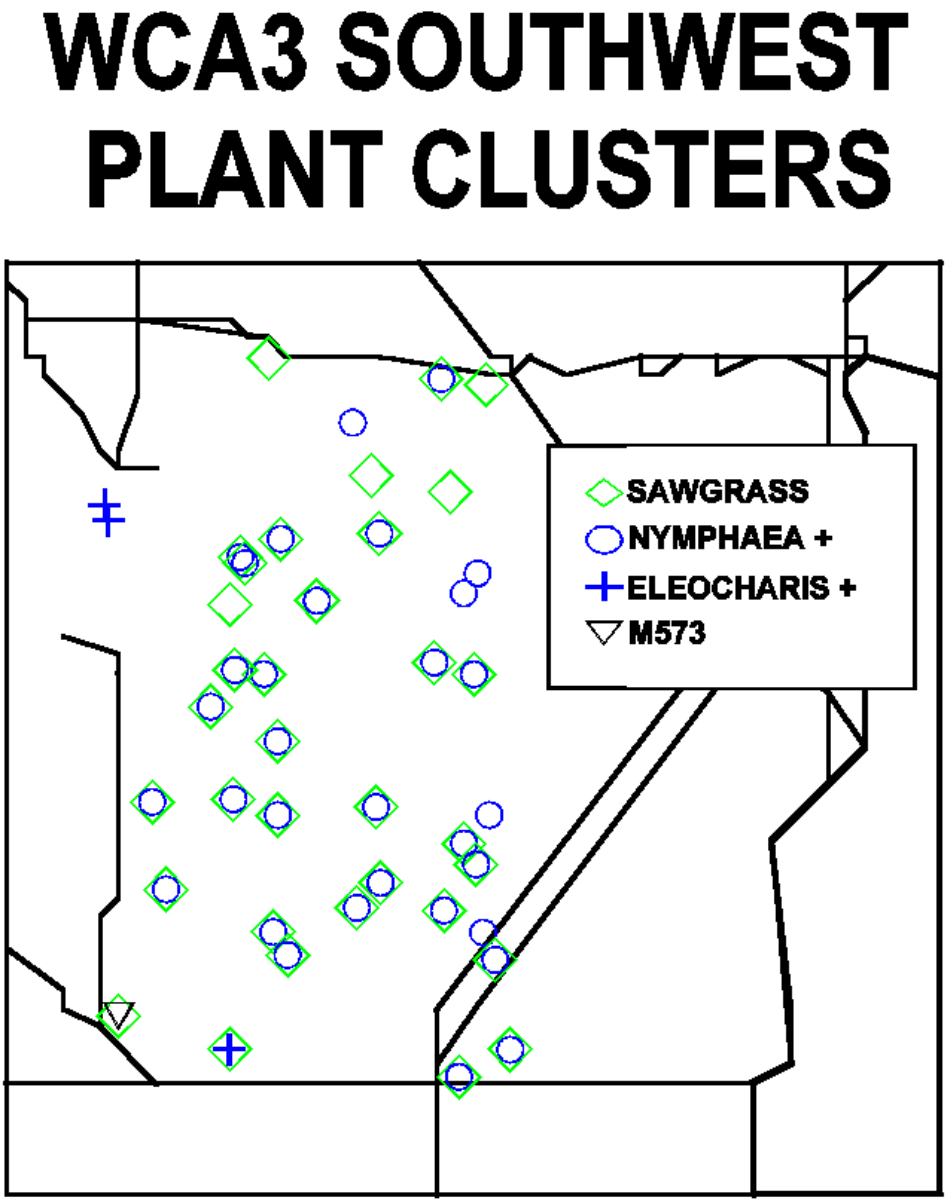


Figure 4.18. Four plant clusters for WCA3-SW.

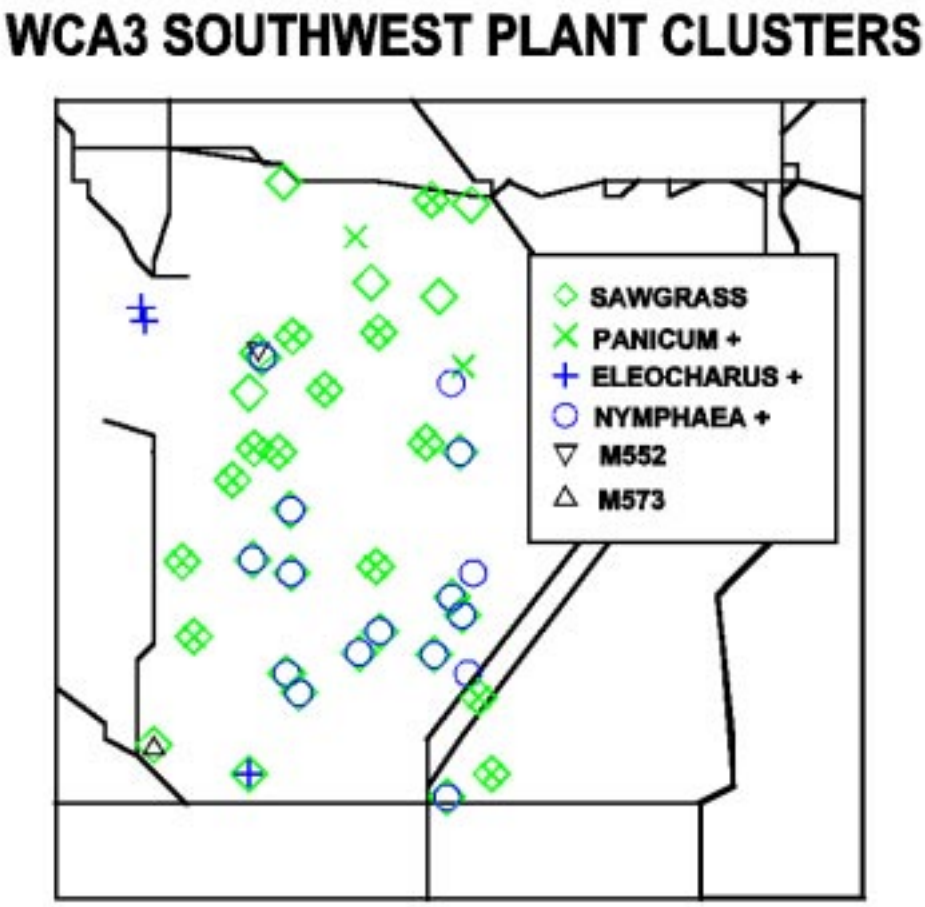


Figure 4.19. Six clusters for WCA3 Southwest.

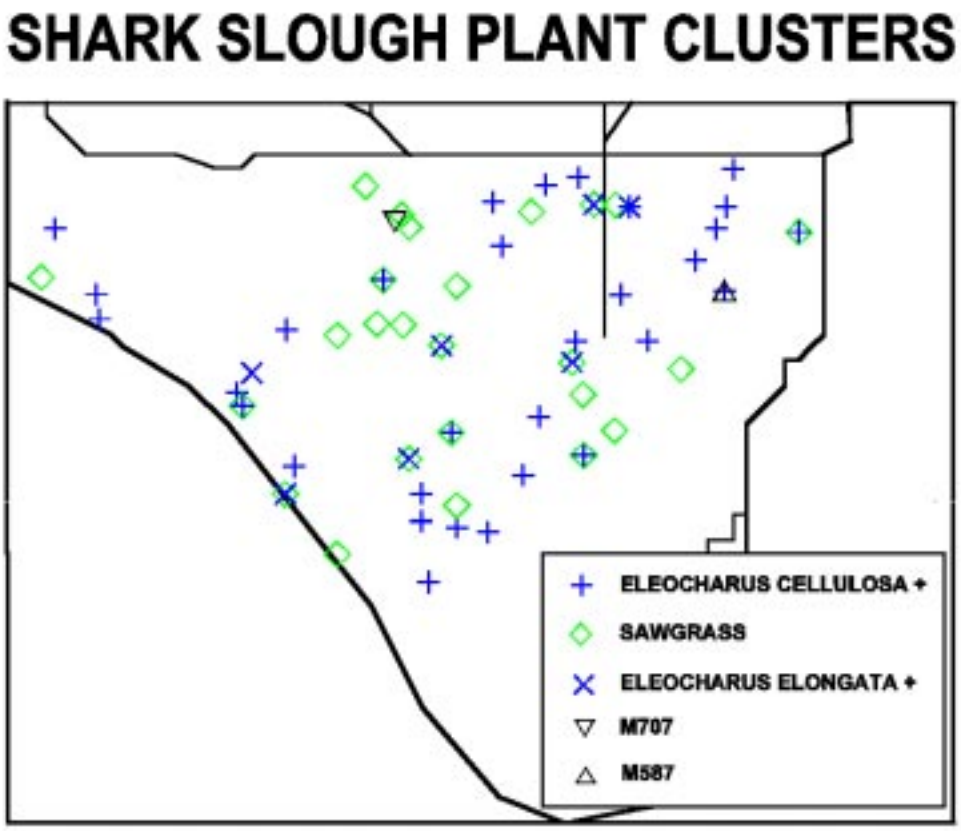


Figure 4.20. Clusters for Shark River Slough.

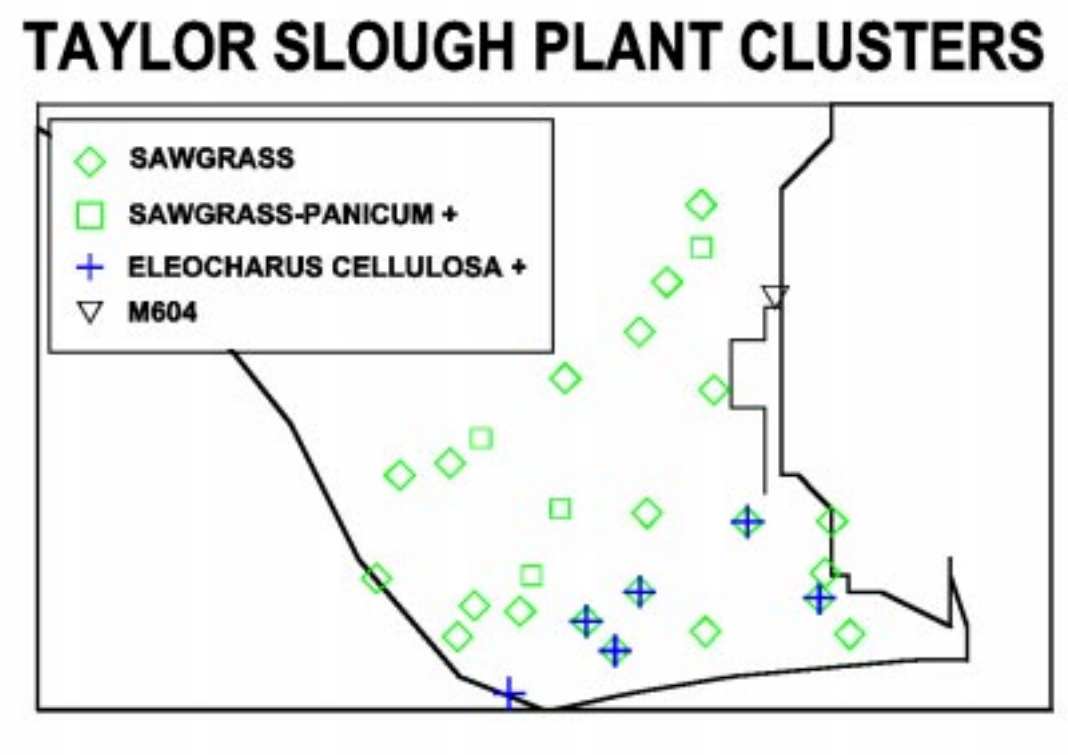


Figure 4.21. Clusters for Taylor Slough.

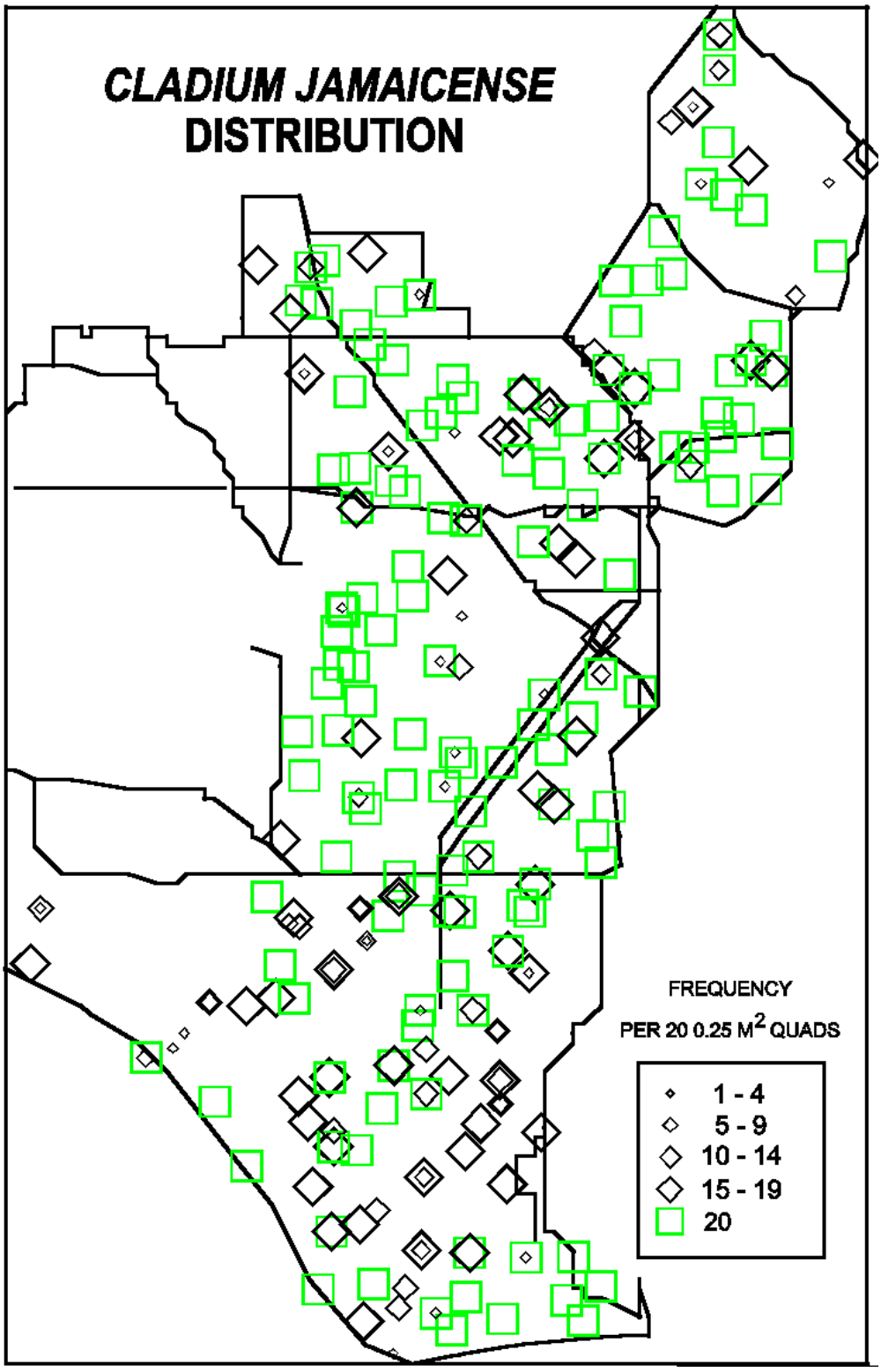


Figure 4.22. Sawgrass distribution in the study area.

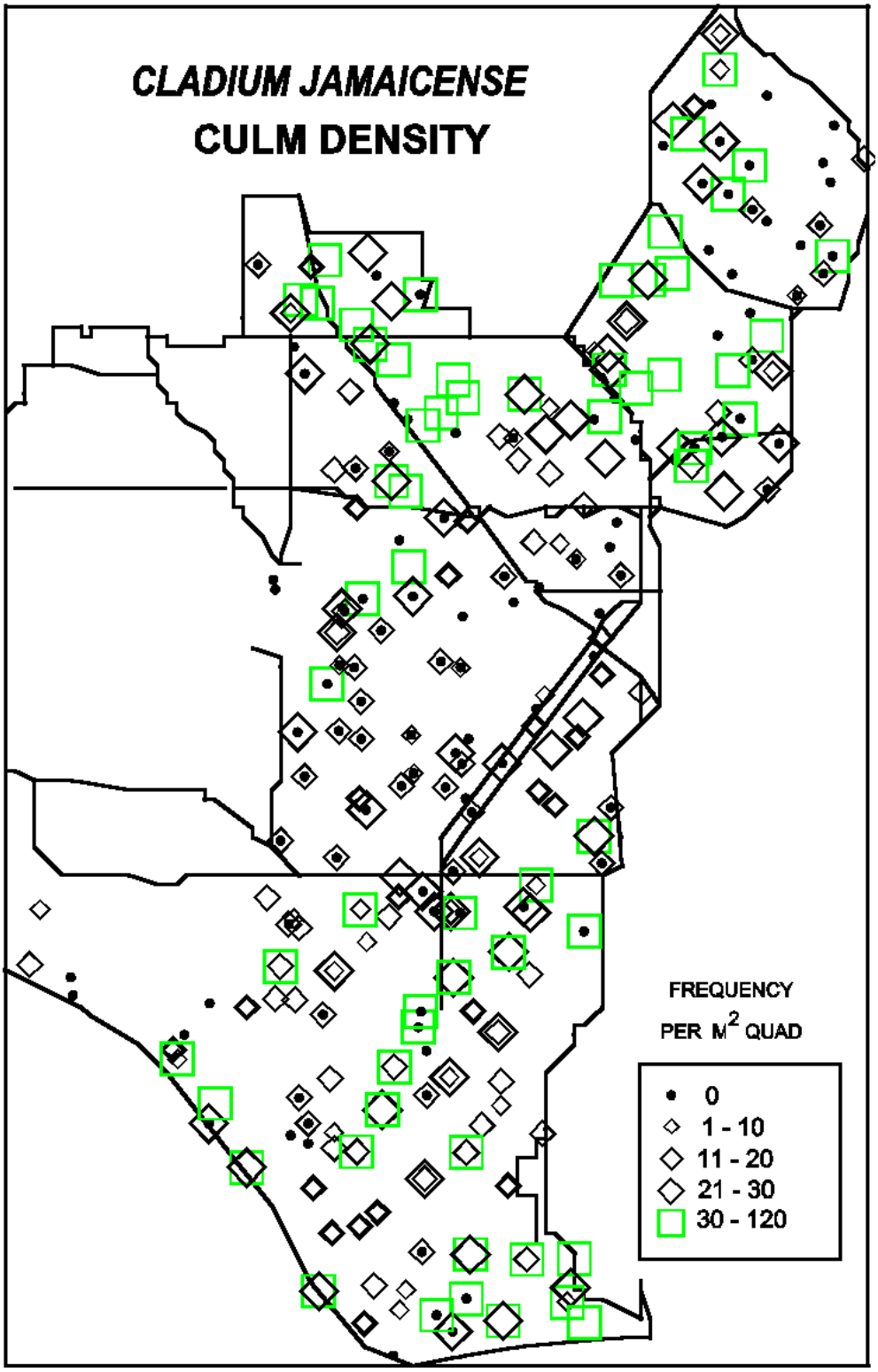


Figure 4.23. Sawgrass culm density in the study area.

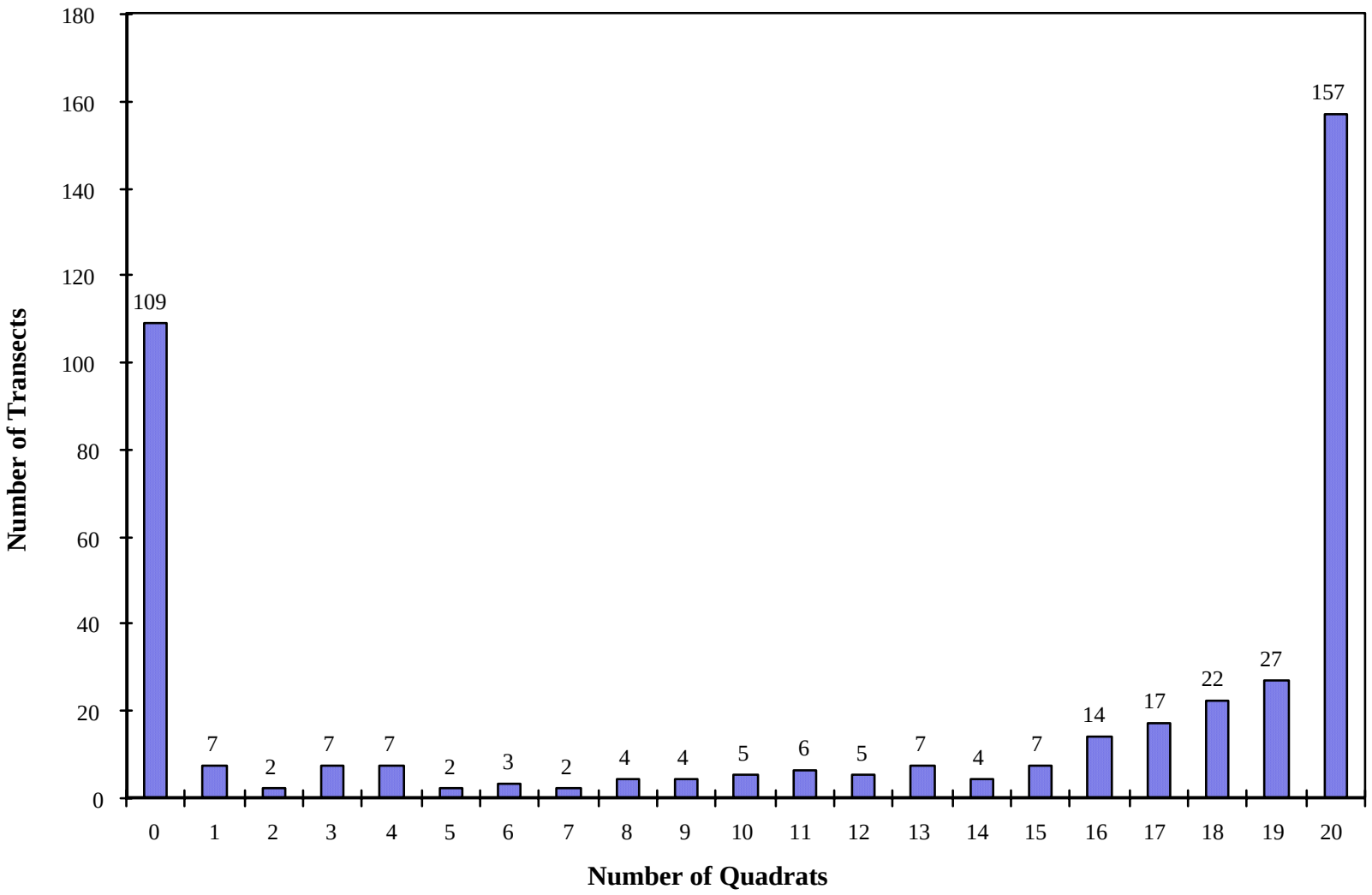


Figure 4.24. Frequency of occurrence of sawgrass per transect.

Cladium jamaicense

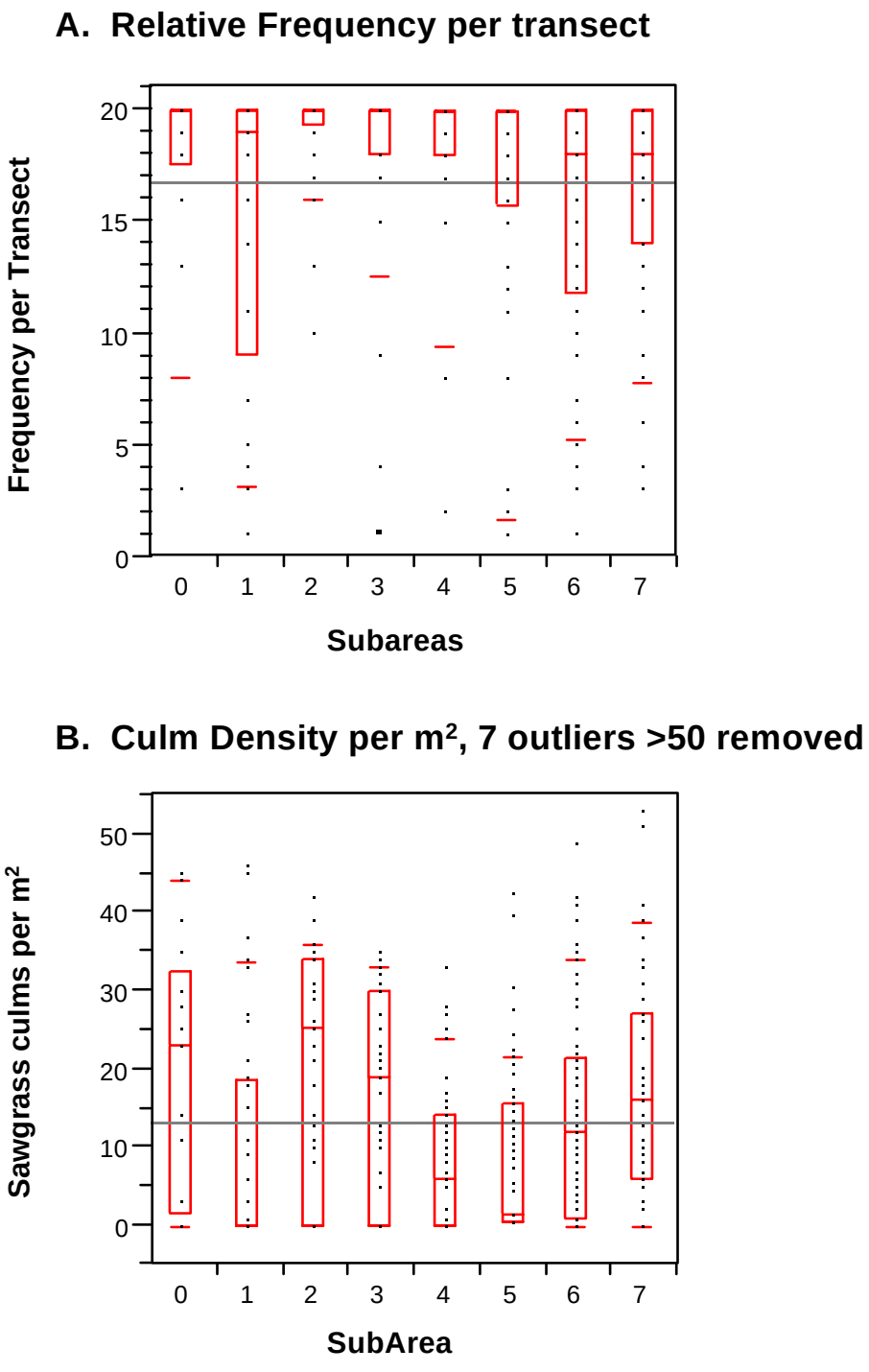


Figure 4.25. Distribution of sawgrass by subareas.

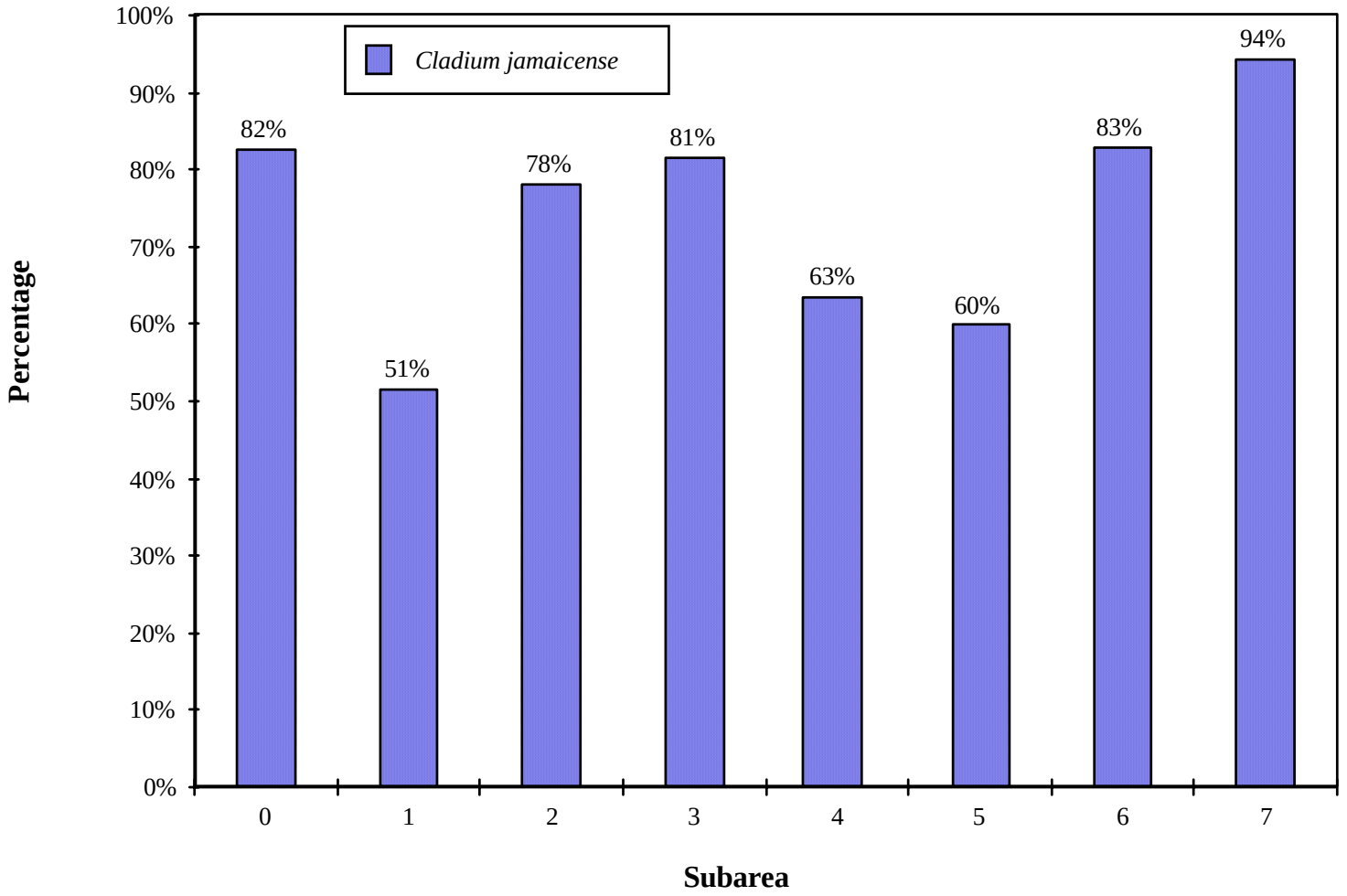


Figure 4.26. Percent of transects in which sawgrass occurs by subarea.

Cladium jamaicense (sawgrass)

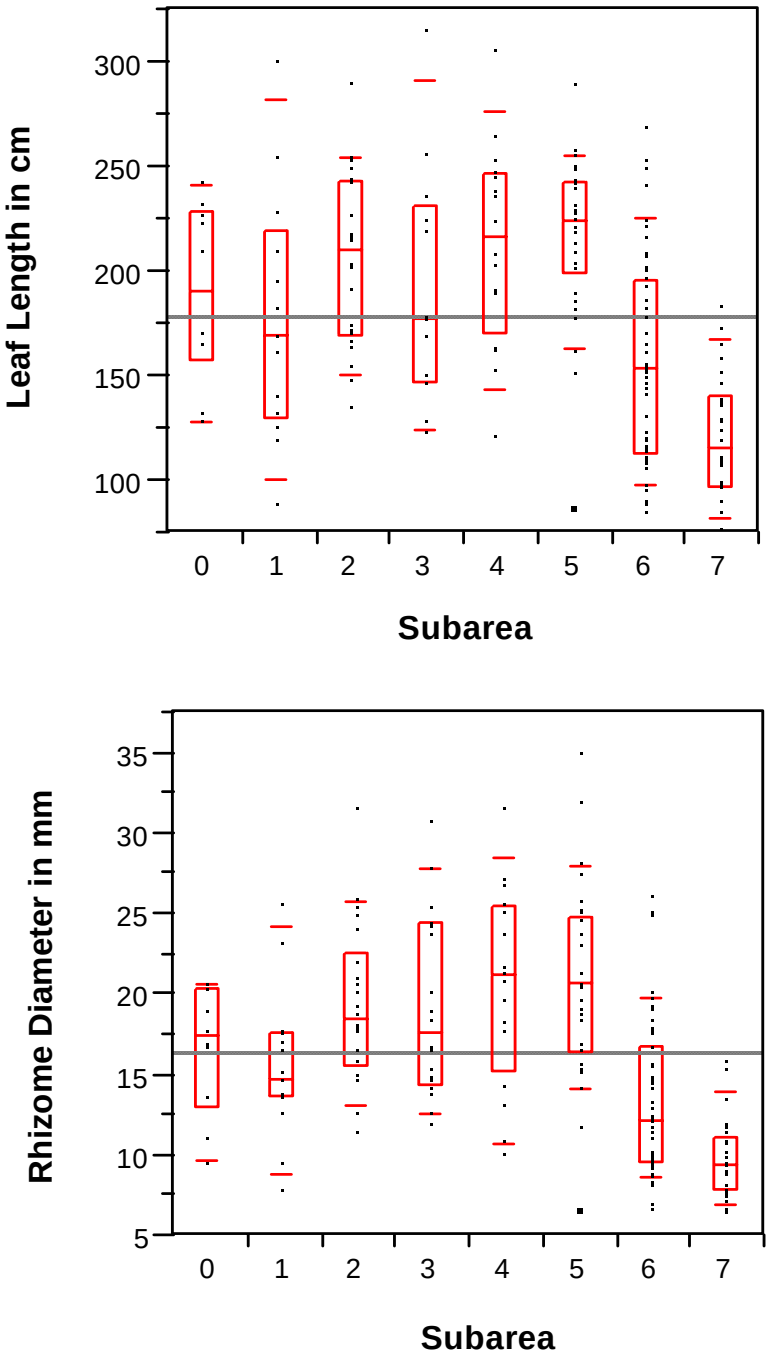


Figure 4.27. Sawgrass morphometrics by subarea.

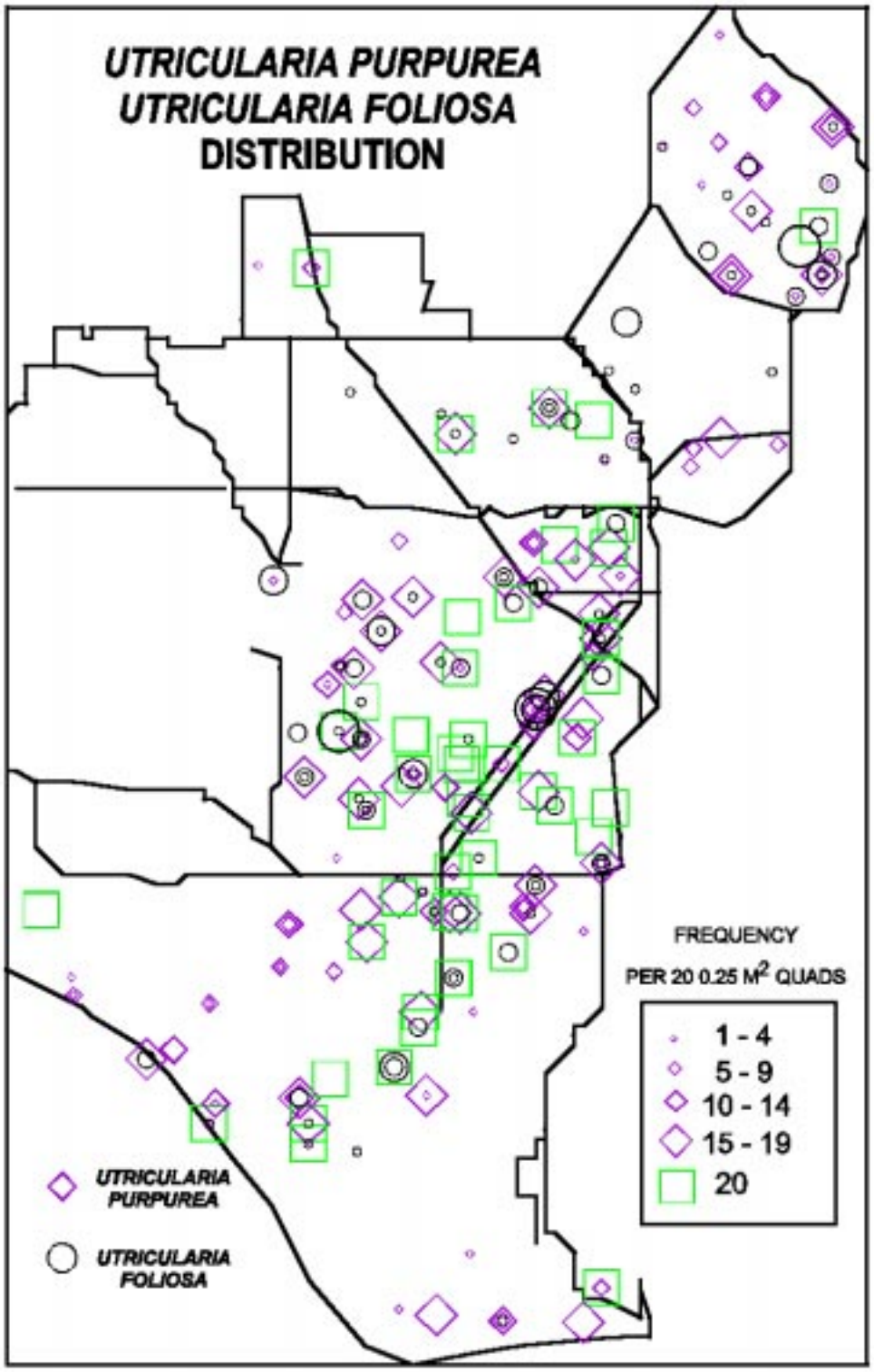


Figure 4.28. Distribution of *U. purpurea* and *U. foliosa* in the study area.

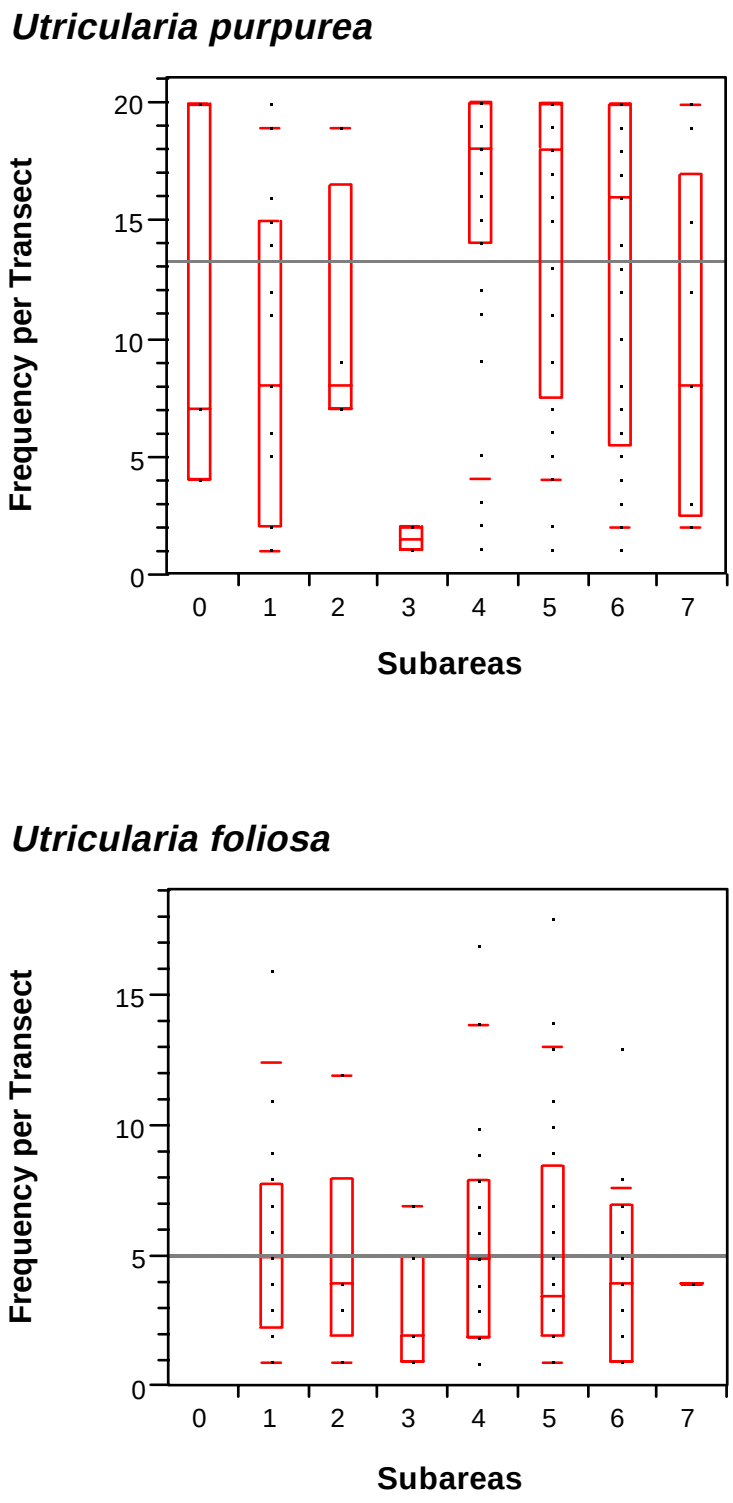


Figure 29. Distribution of *U. purpurea* and *U. foliosa* by subarea.

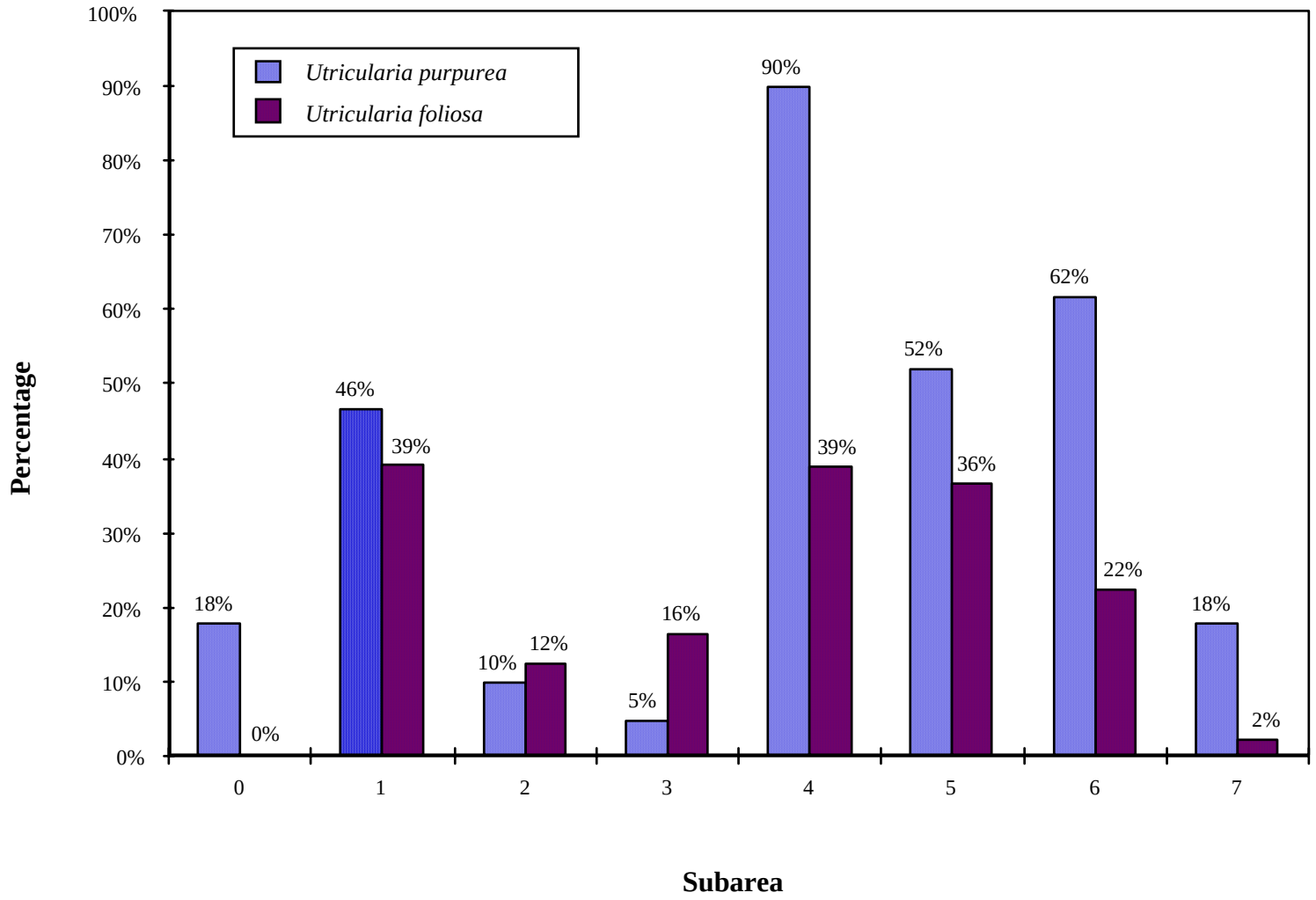


Figure 4.30. Percent of transects with *U. purpurea* and *U. foliosa* by subarea.

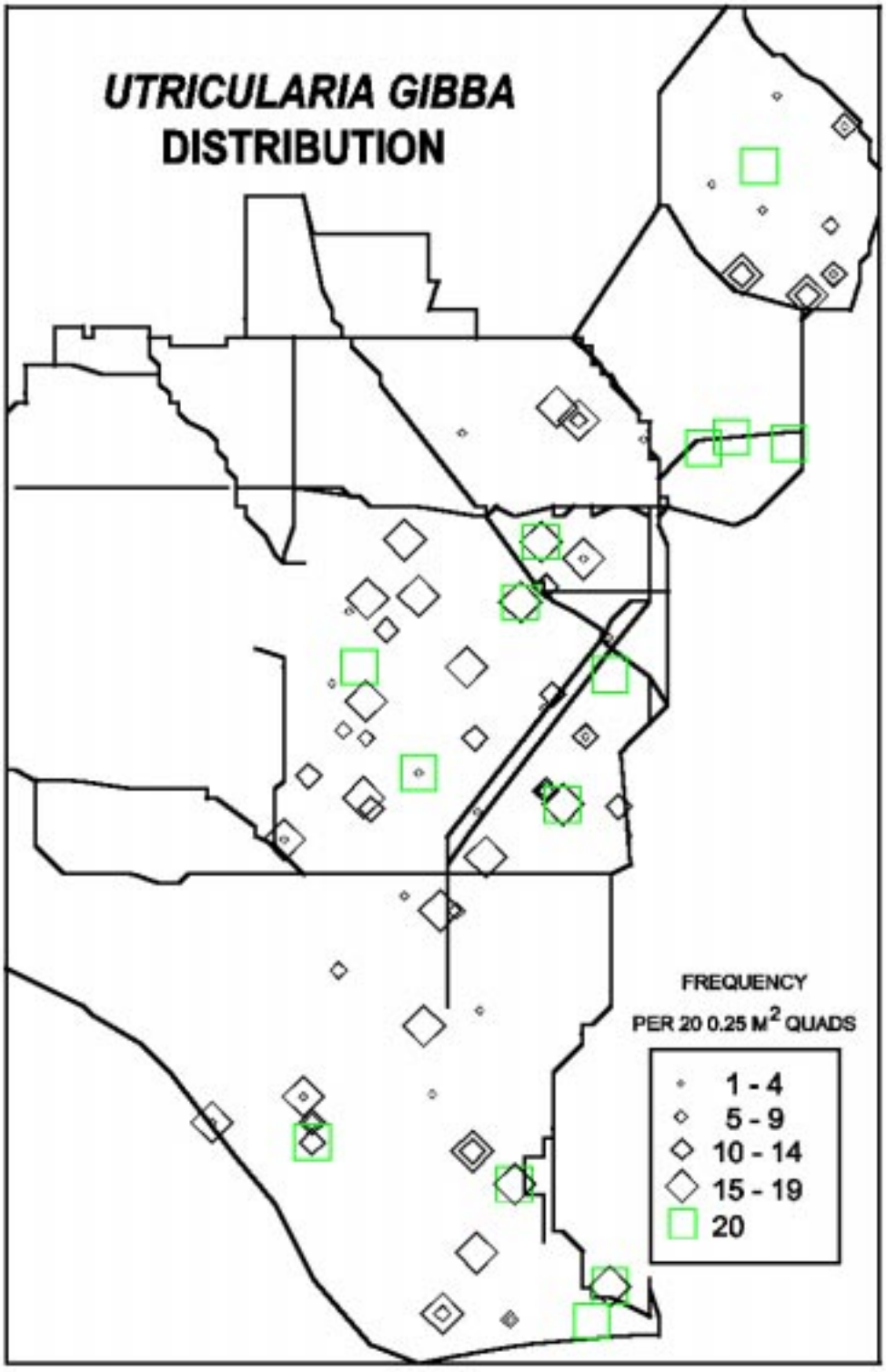


Figure 4.31. *U. gibba* distribution in the study area.

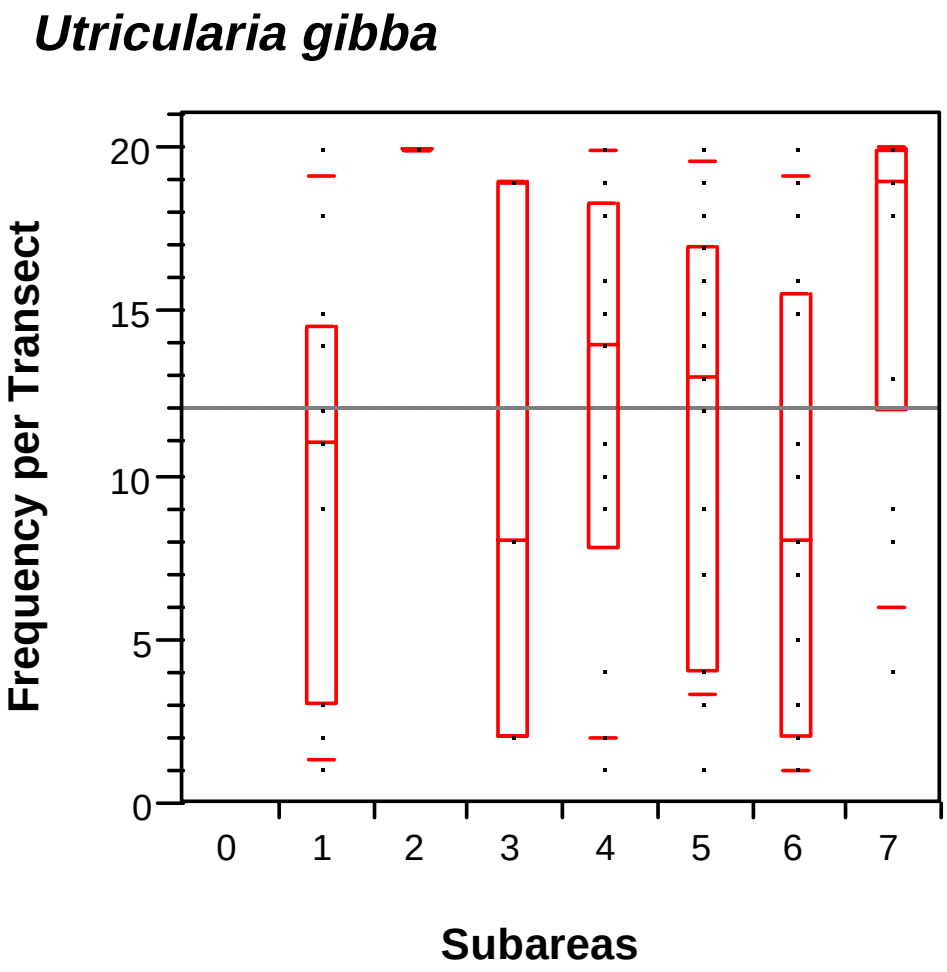


Figure 4.32. Distribution of *U. gibba* by subarea.

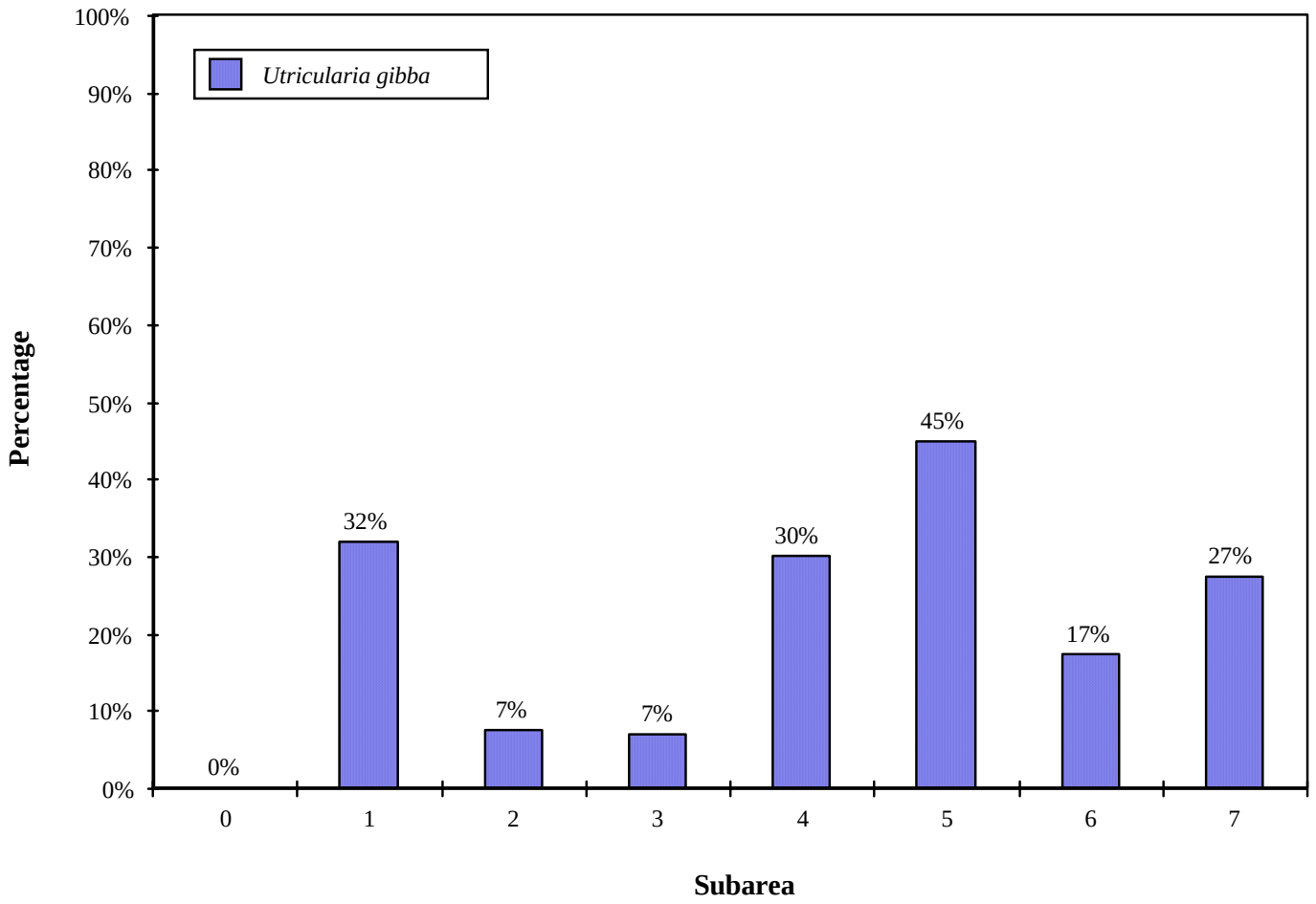


Figure 4.33. Percent of transects with *U. gibba* by subarea.

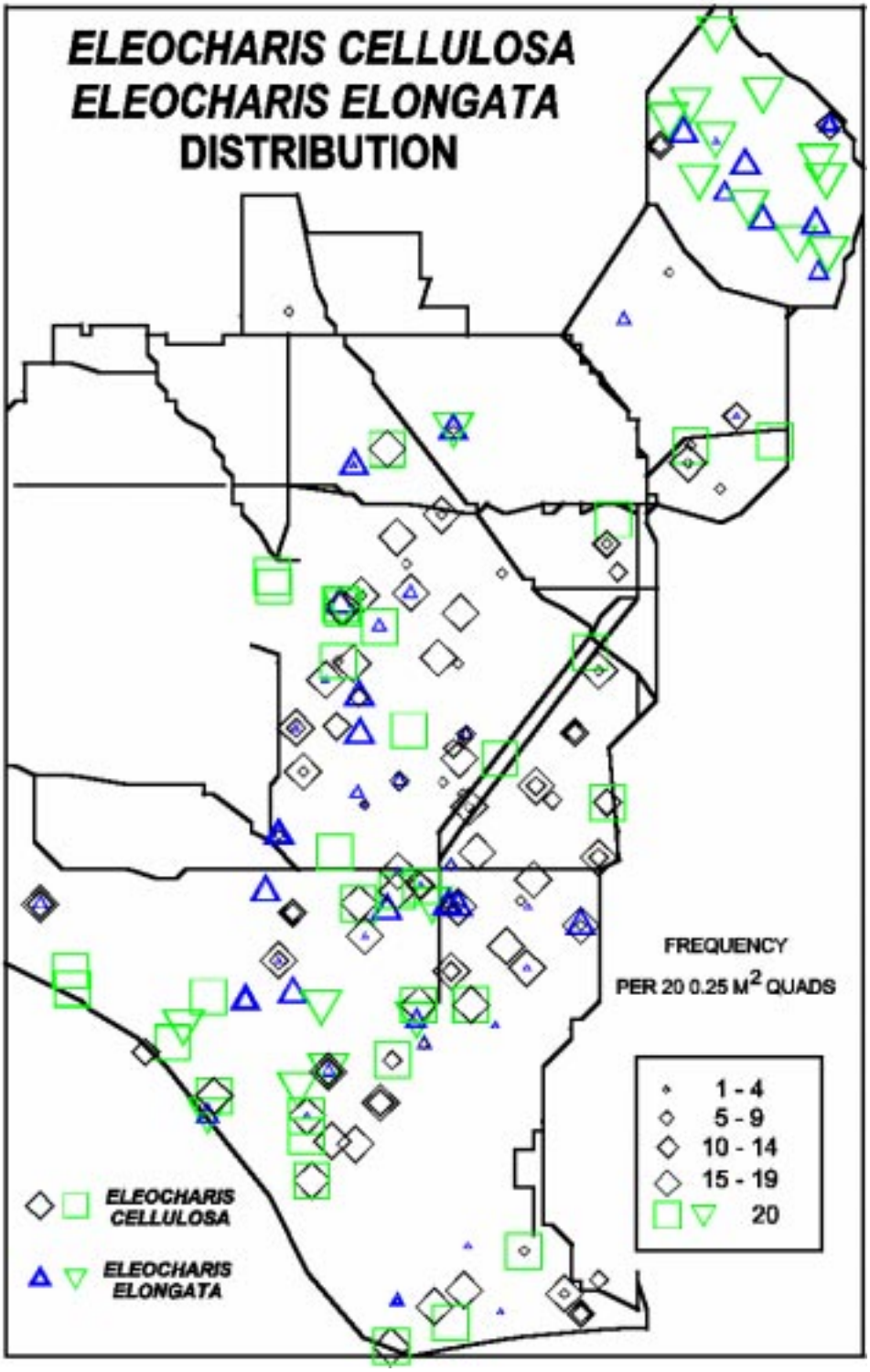
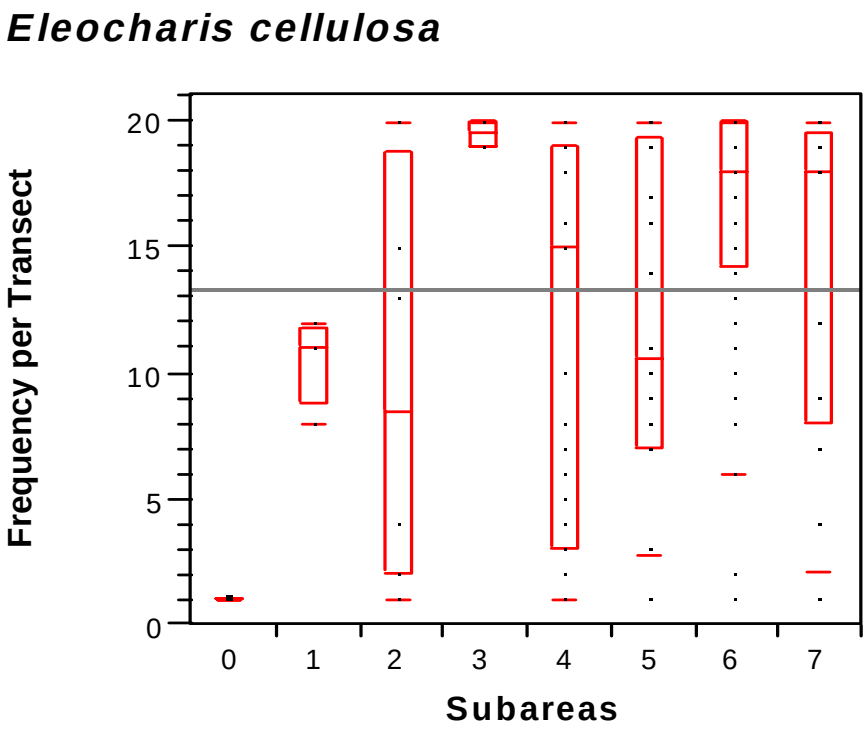


Figure 4.34. Distribution of *E. cellulosa* and *E. elongata* in the study area.



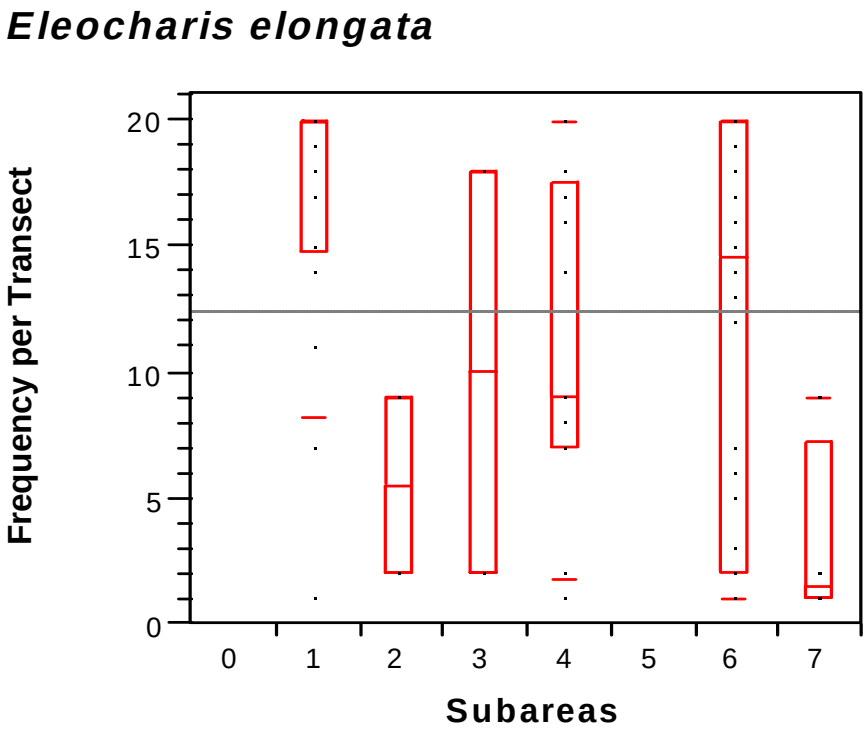


Figure 4.35. Distribution of *E. cellulosa* and *E. elongata* by subarea.

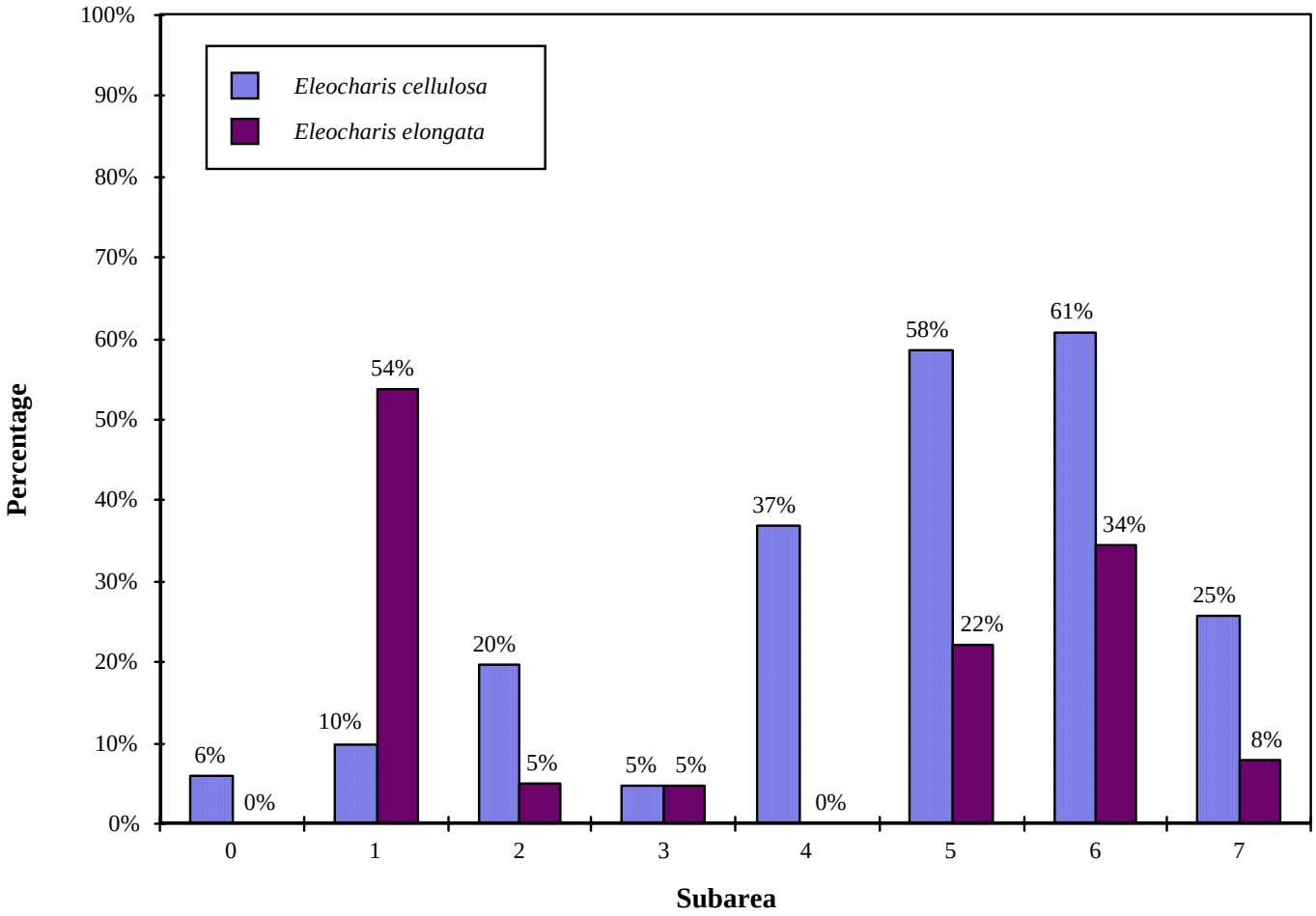


Figure 4.36. Percent of transects with *E. cellulosa* and *E. elongata* by subarea.

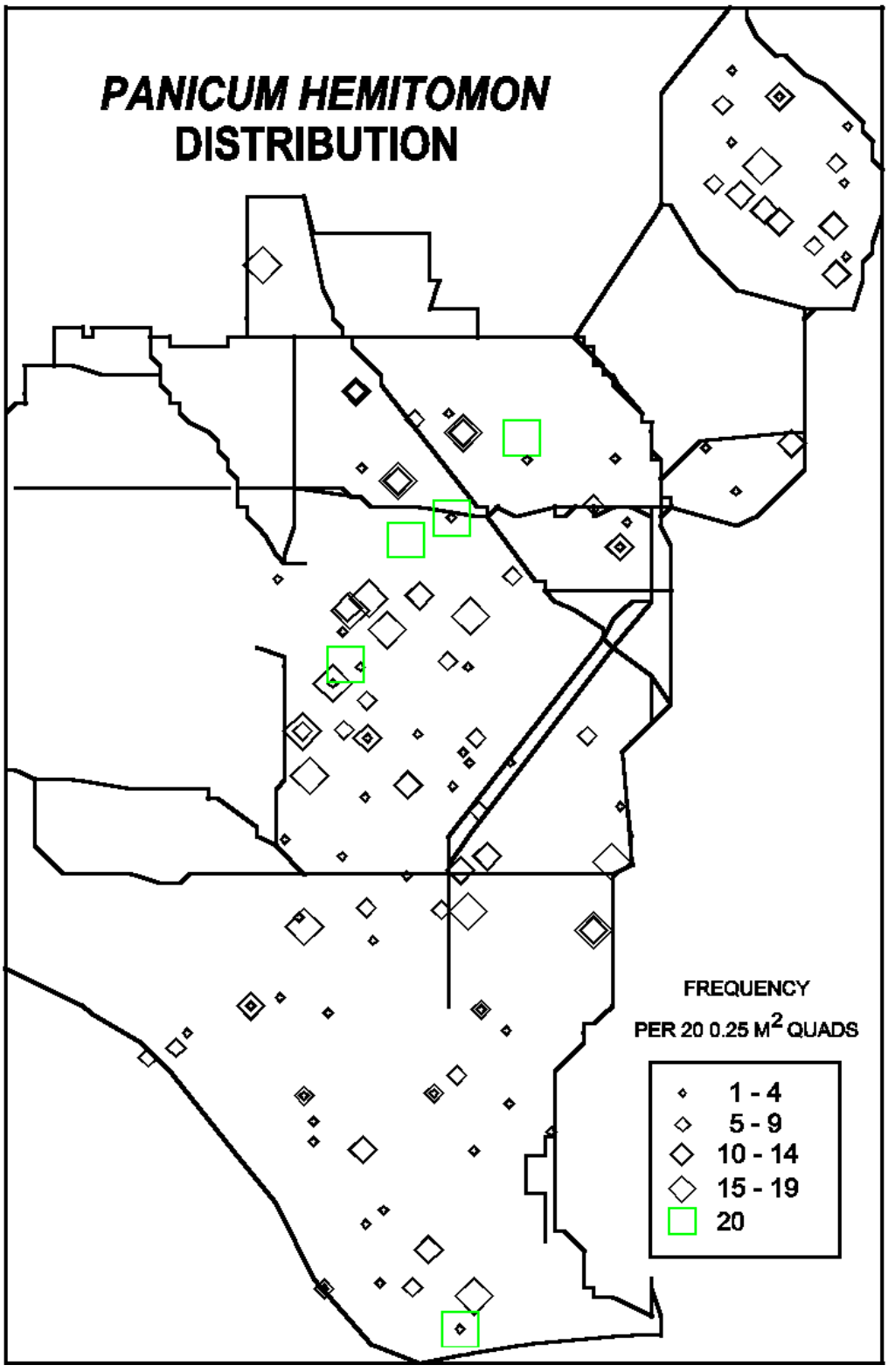


Figure 4.37. Distribution of *P. hemitomon* in the study area.

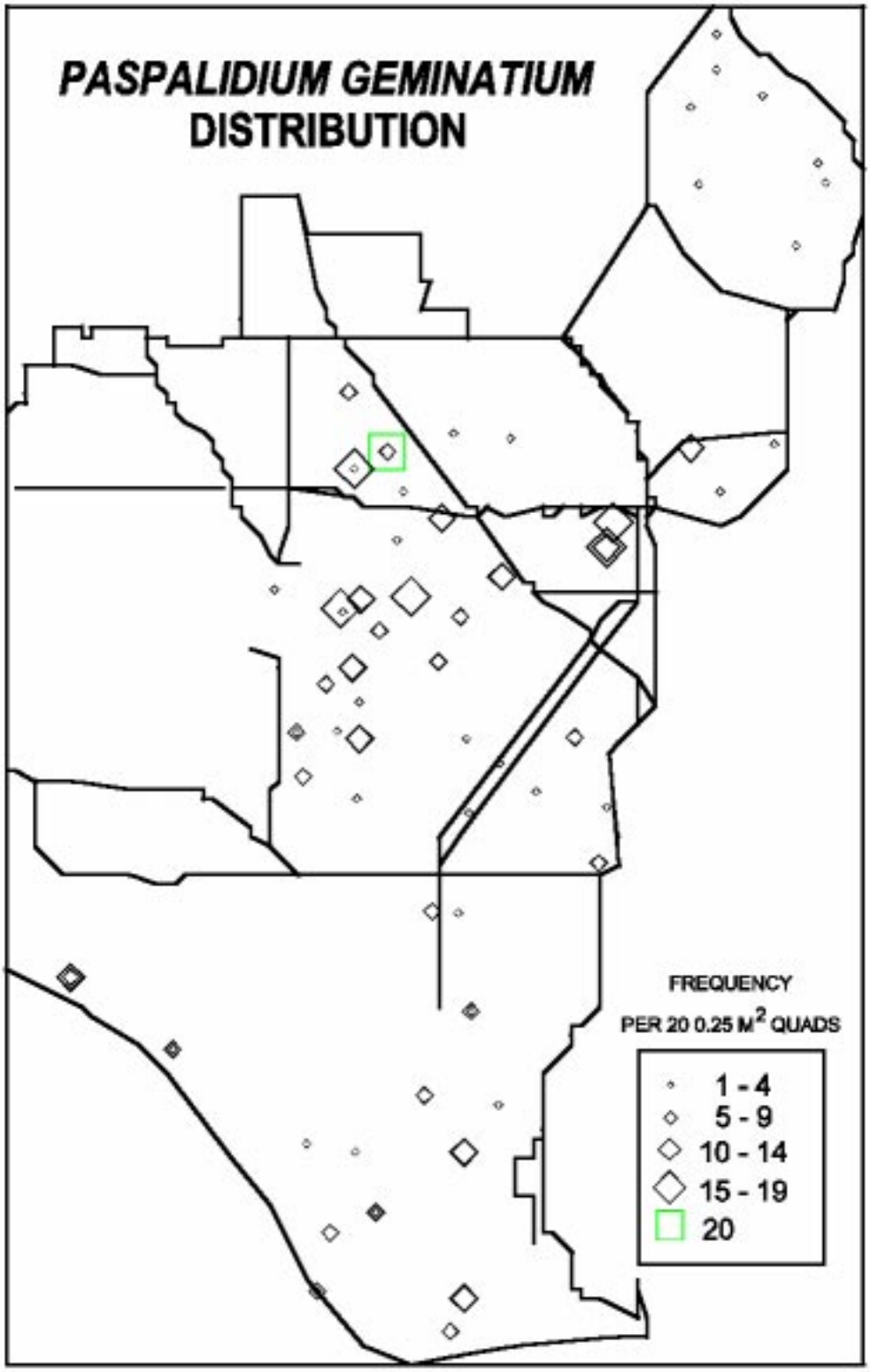
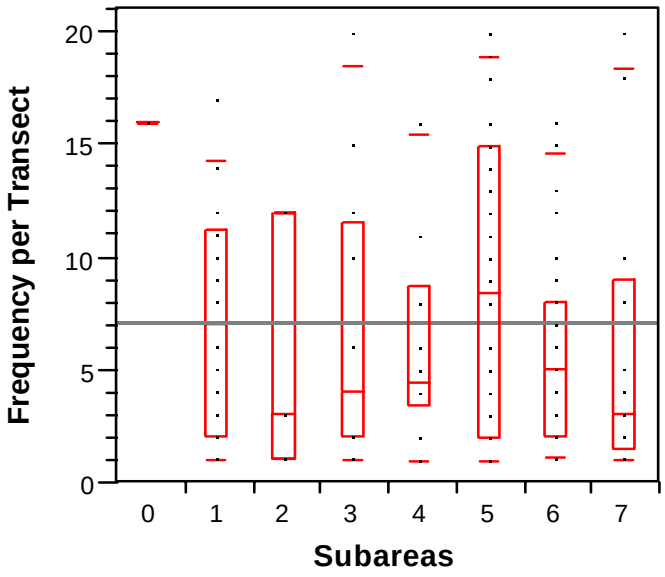


Figure 4.38. Distribution of *P. geminatium* in the study area.

A. *Panicum hemitomon*



B. *Paspalidium geminatum*

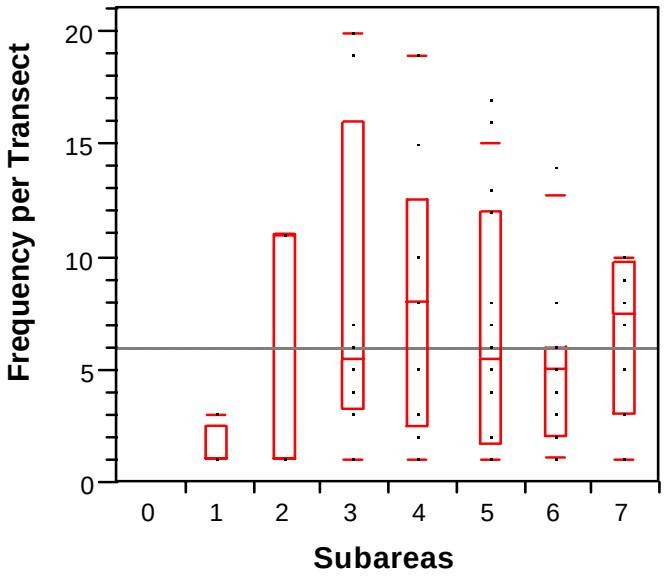


Figure 4.39. Distribution of *P. hemitomon* and *P. geminatium* by subarea.

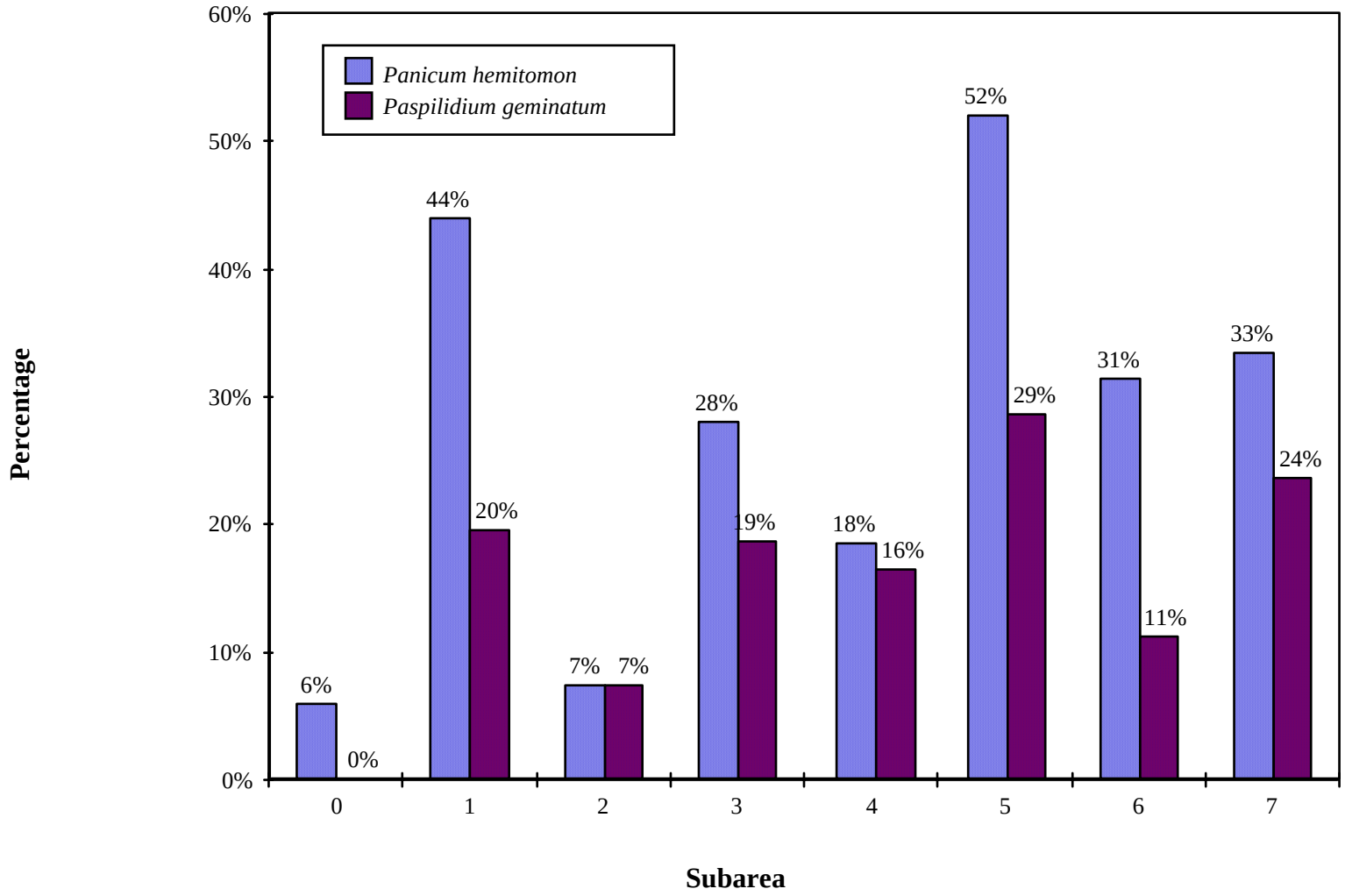


Figure 4.40. Percent of transects with *P. hemitomon* and *P. geminatium* by subarea.

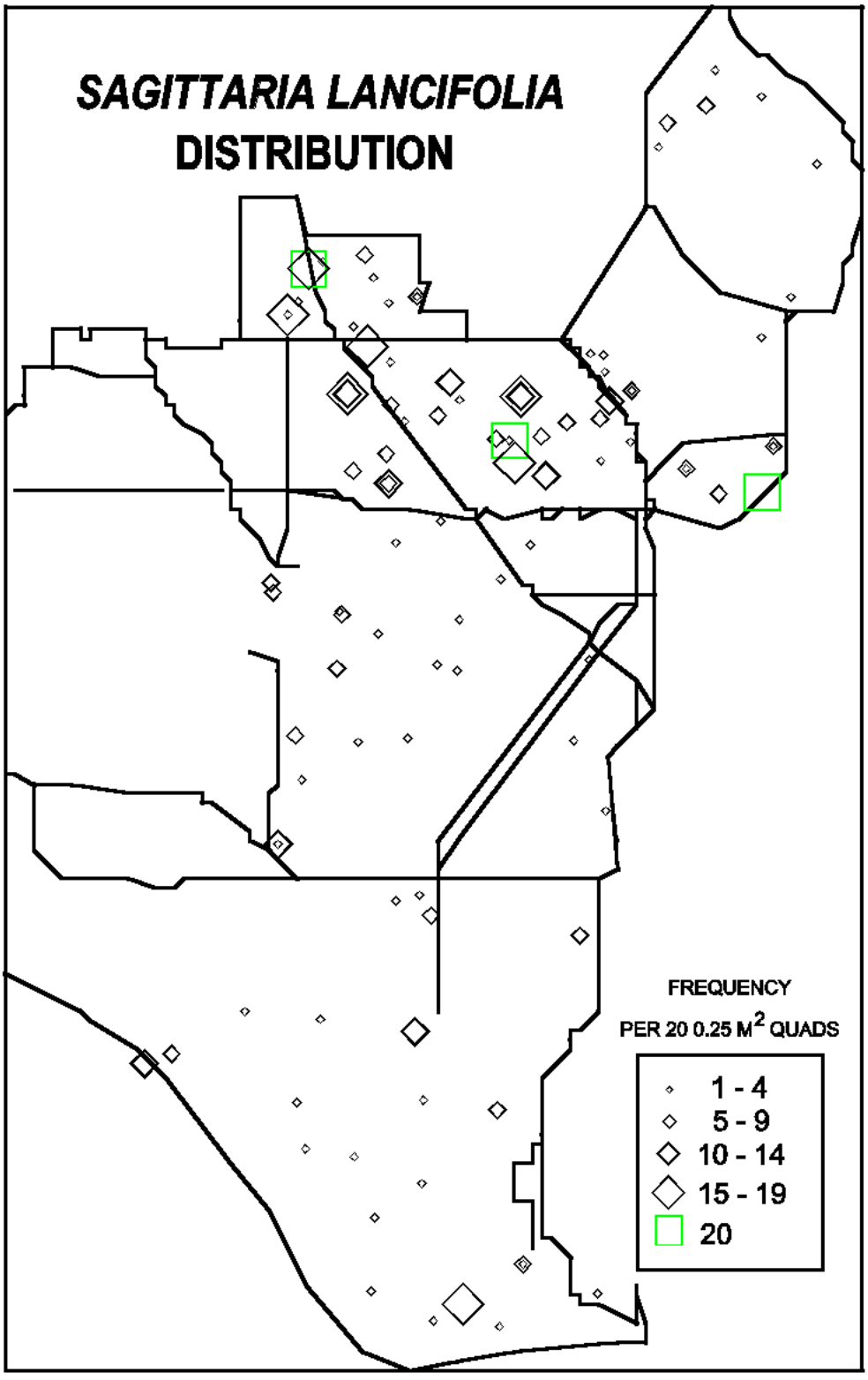


Figure 4.41. Distribution of *S. lancifolia* in study area.

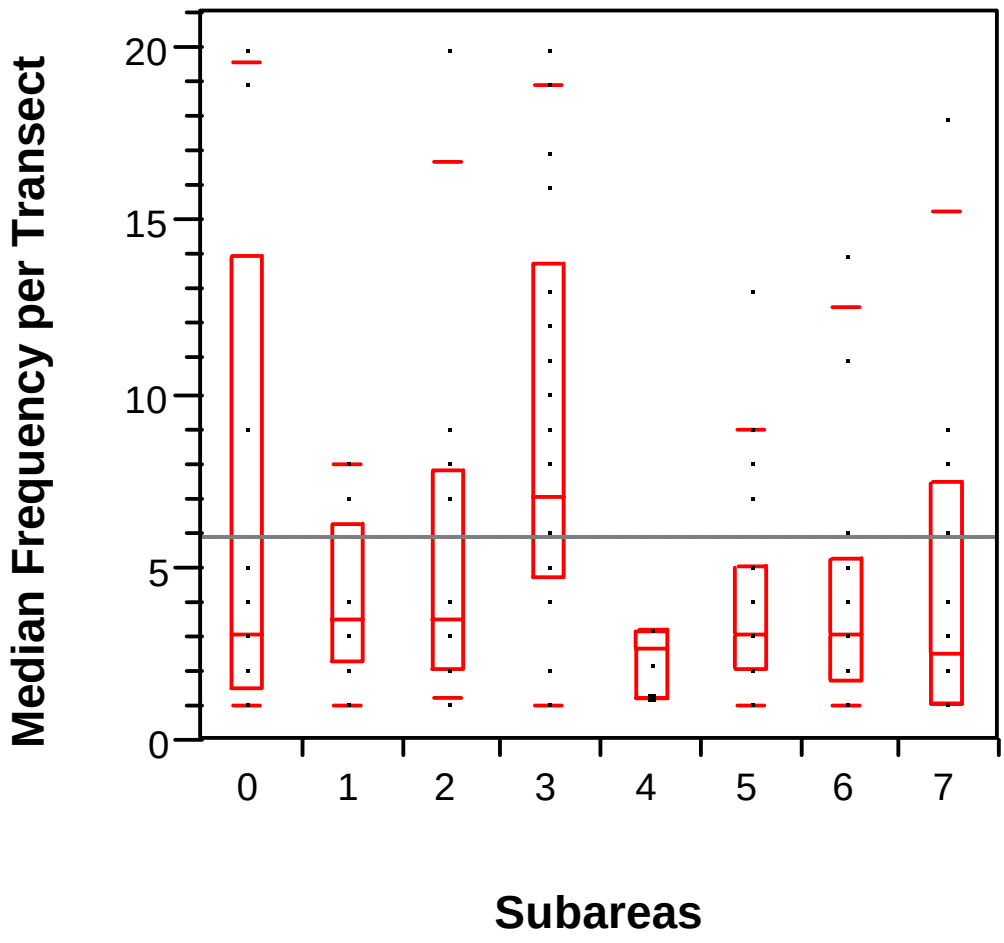


Figure 4.42. Distribution of *S. lancifolia* by subarea.

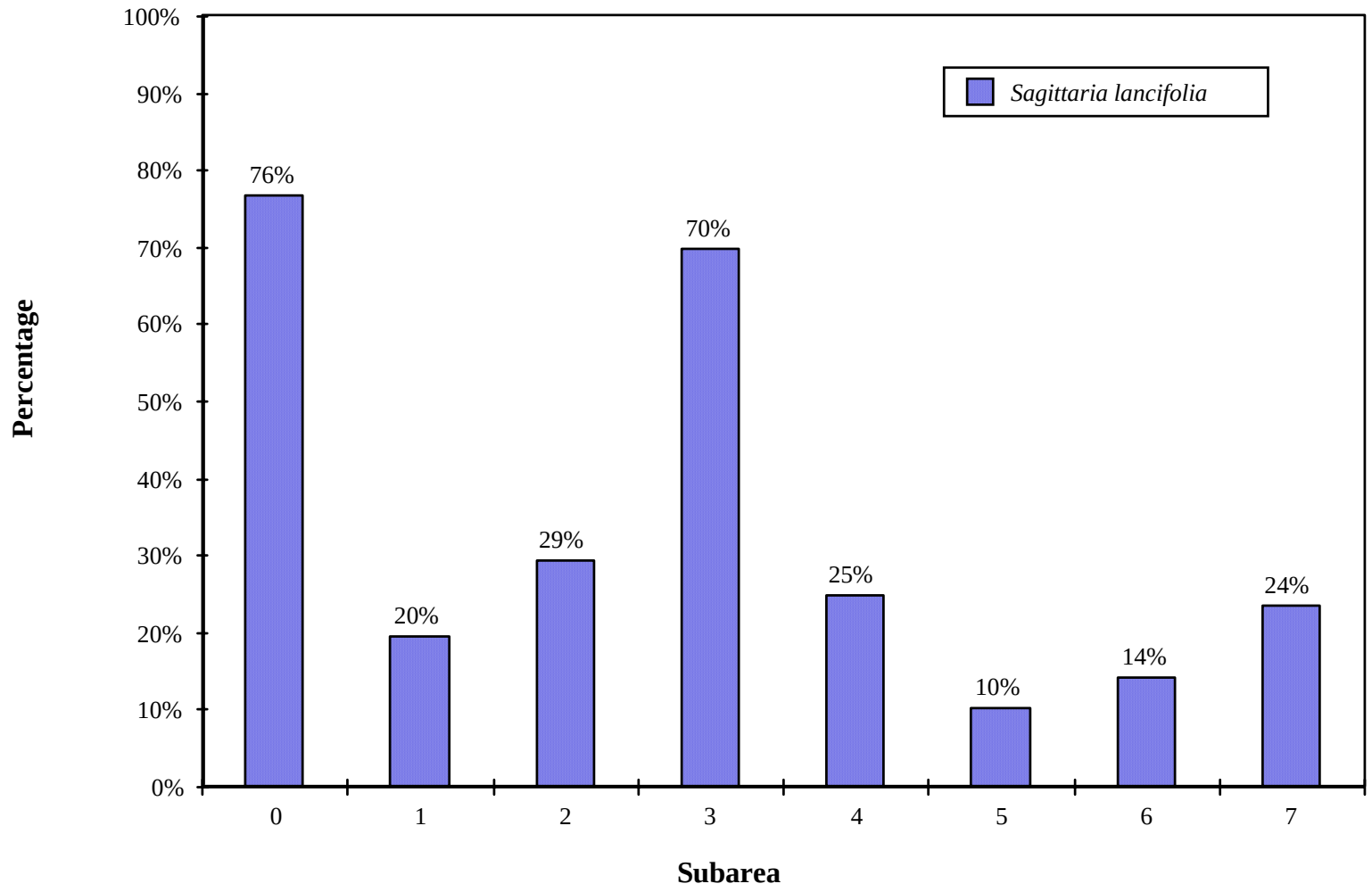


Figure 4.43. Percent of transects with *S. lancifolia* by subarea.

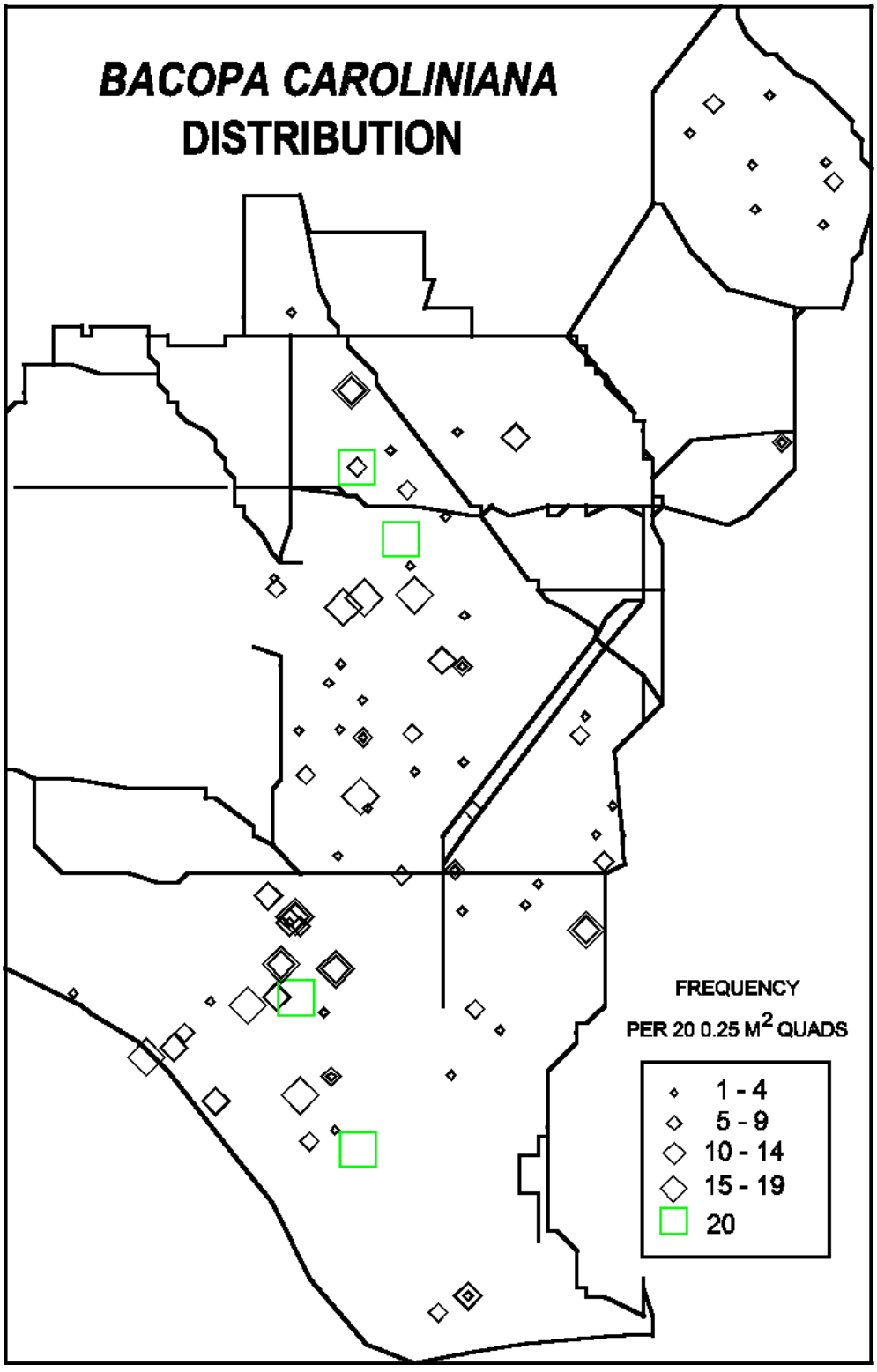


Figure 4.44. Distribution of *B. caroliniana* in study area.

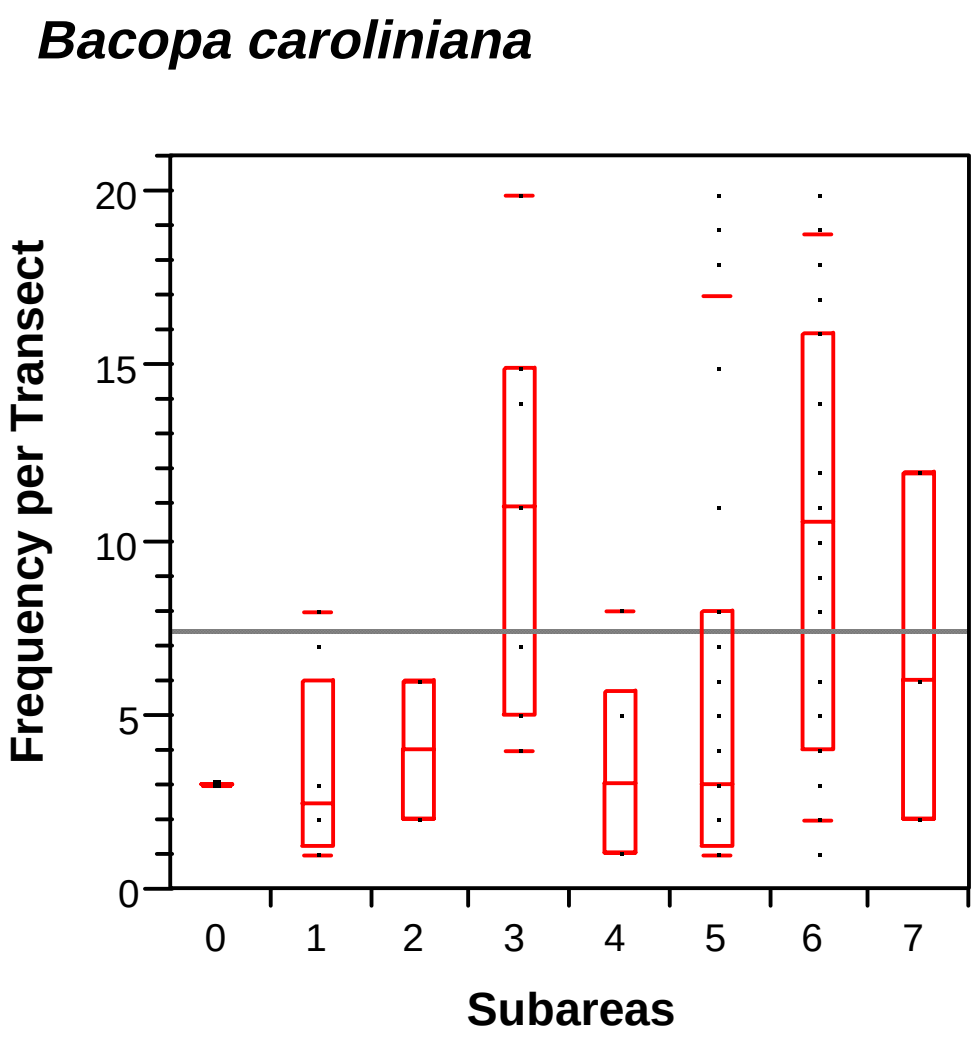


Figure 4.45. Distribution of *B. caroliniana* by subarea.

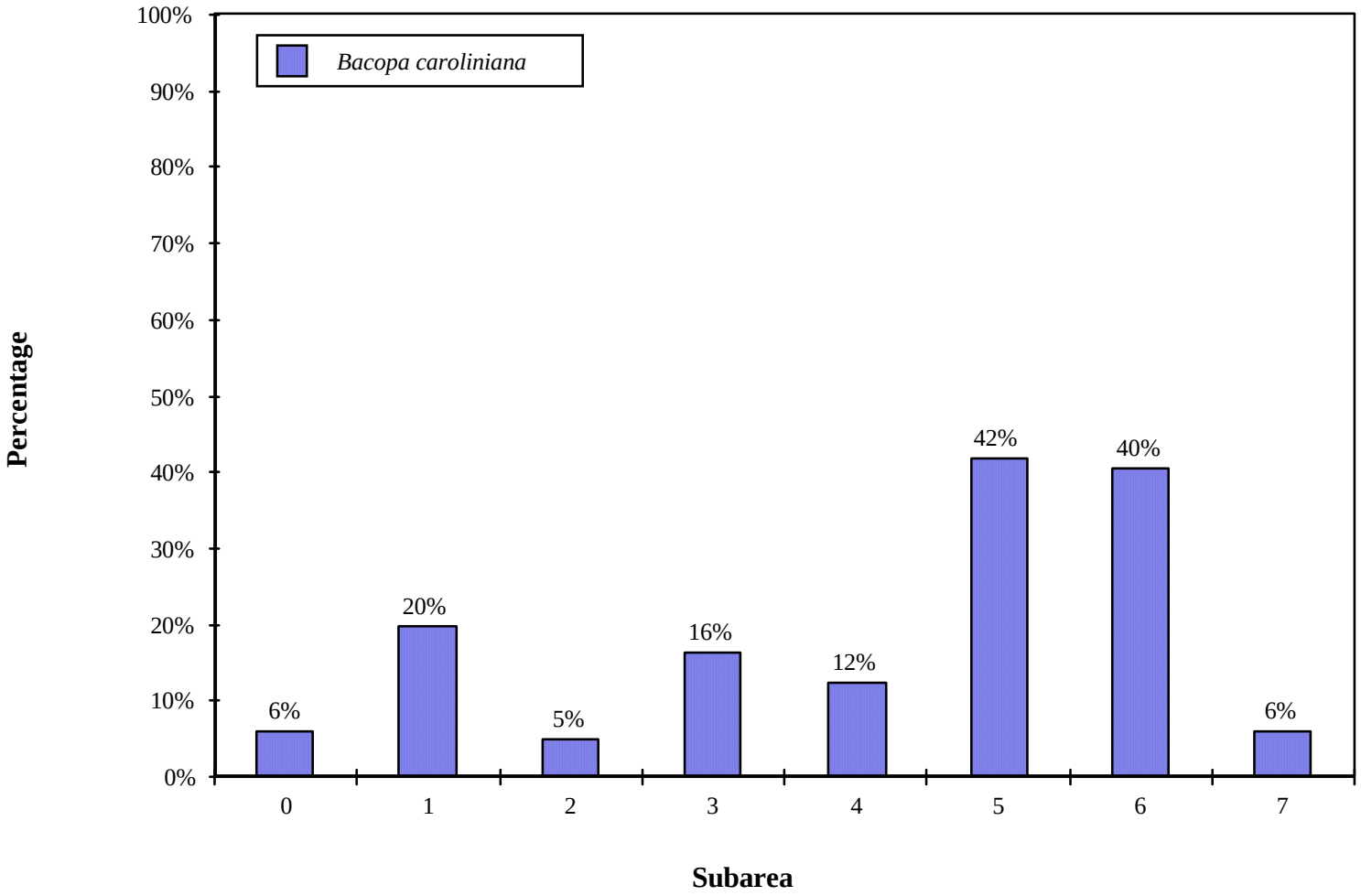


Figure 4.46. Percent of transects with *B. caroliniana* by subarea.

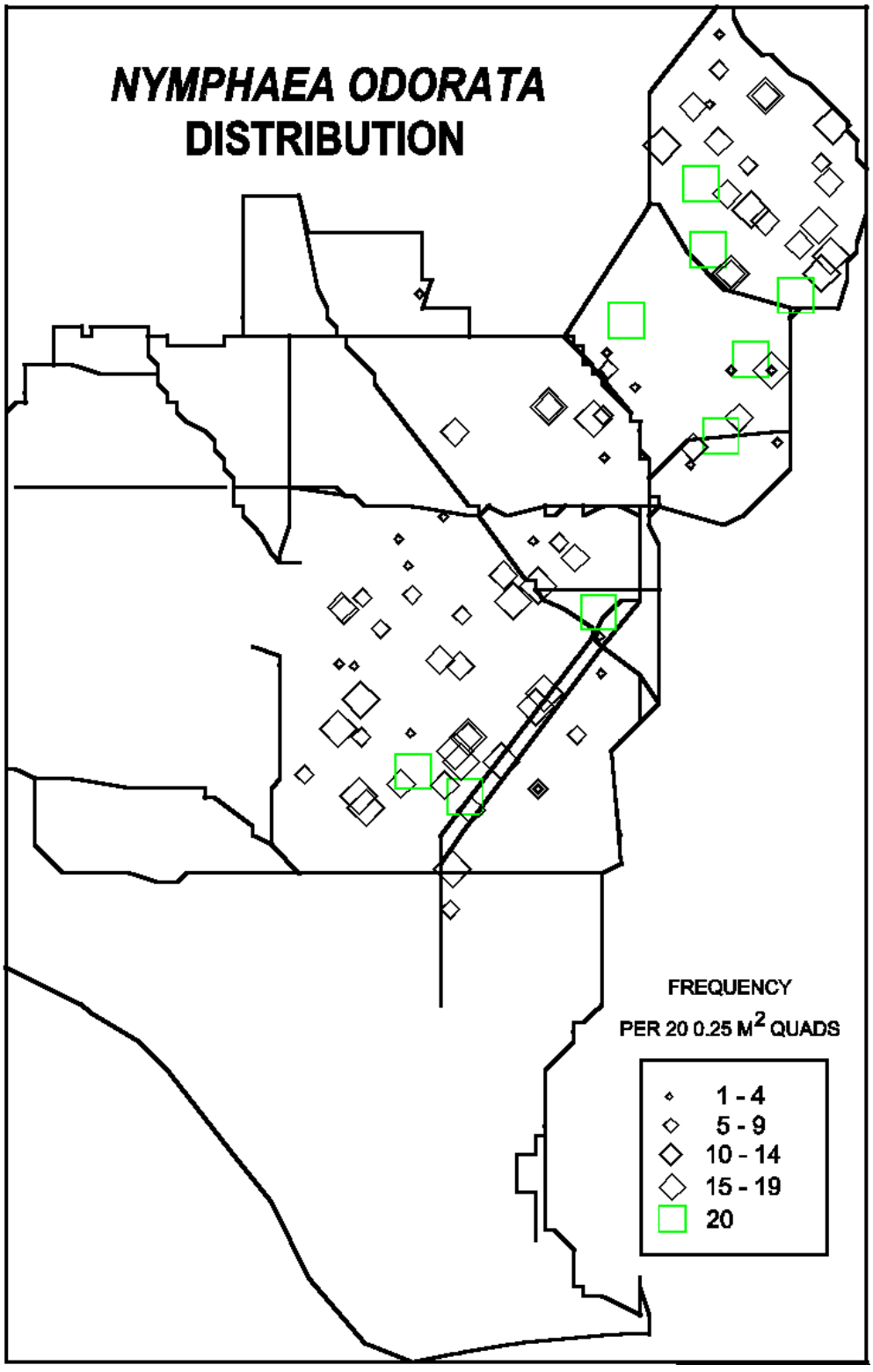


Figure 4.47. Distribution of *N. odorata* in study area.

Nymphaea odorata

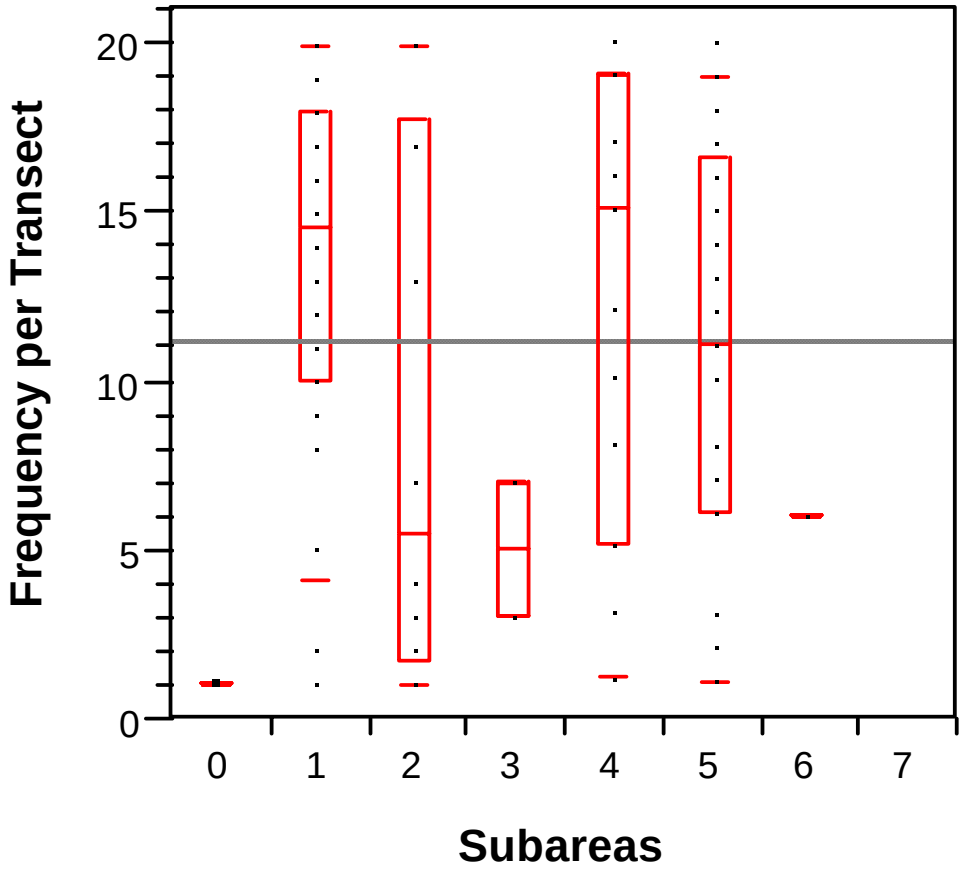


Figure 4.48. Distribution of *N. odorata* by subarea.

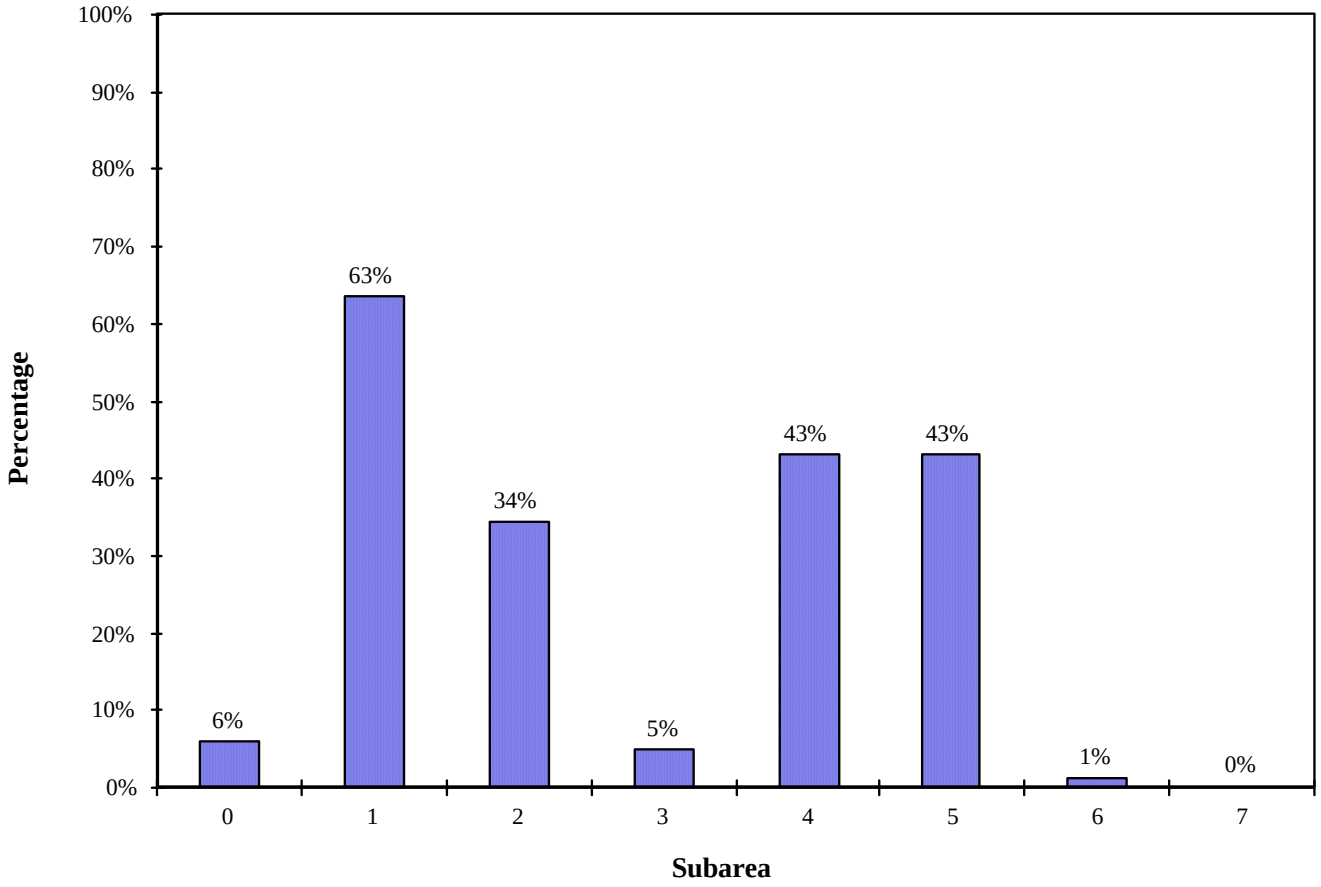


Figure 4.49. Percent of transects with *N. odorata* by subarea.

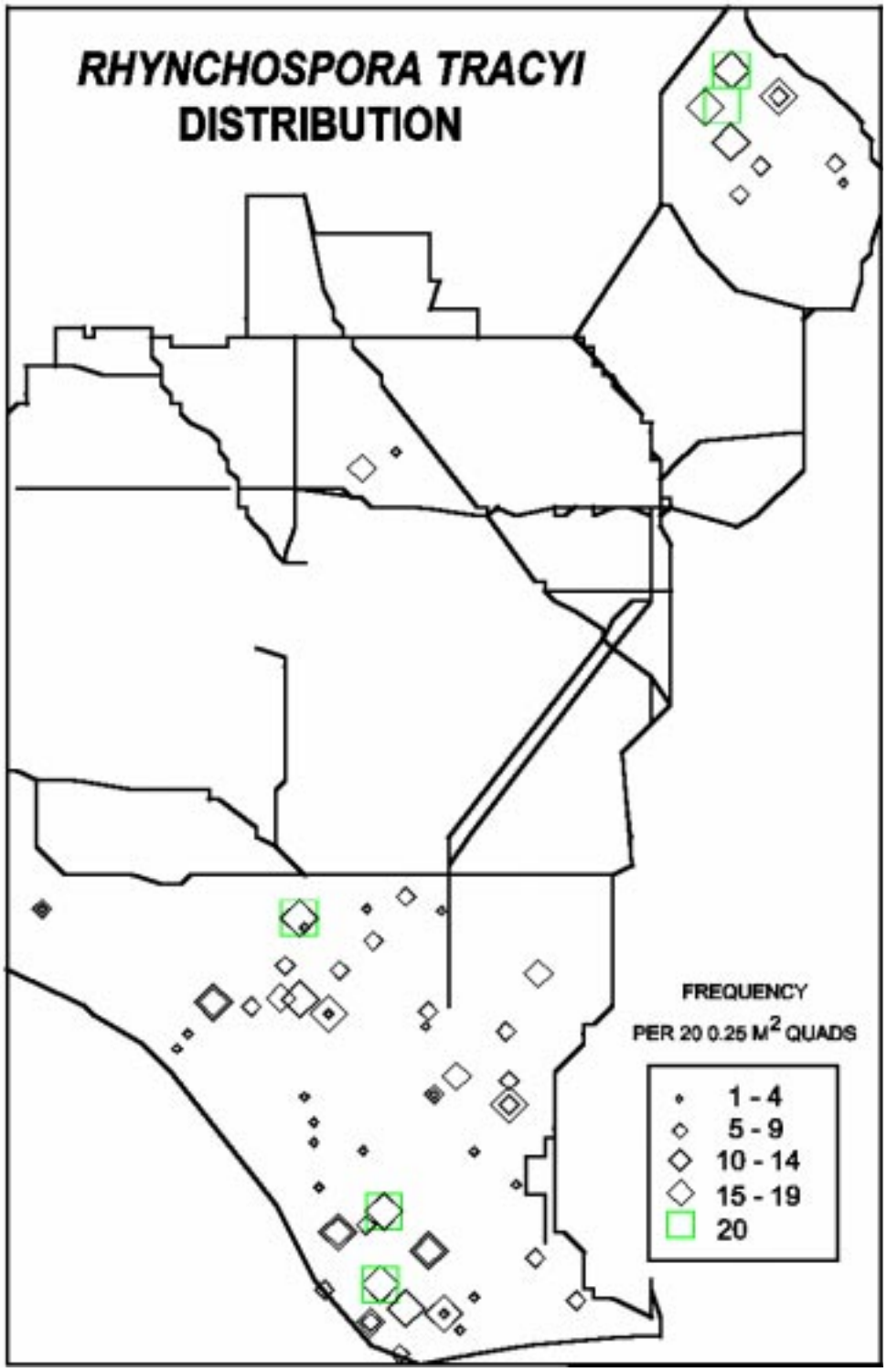


Figure 4.50. Distribution of *R. tracyi* by subarea.

Rhynchospora tracyi

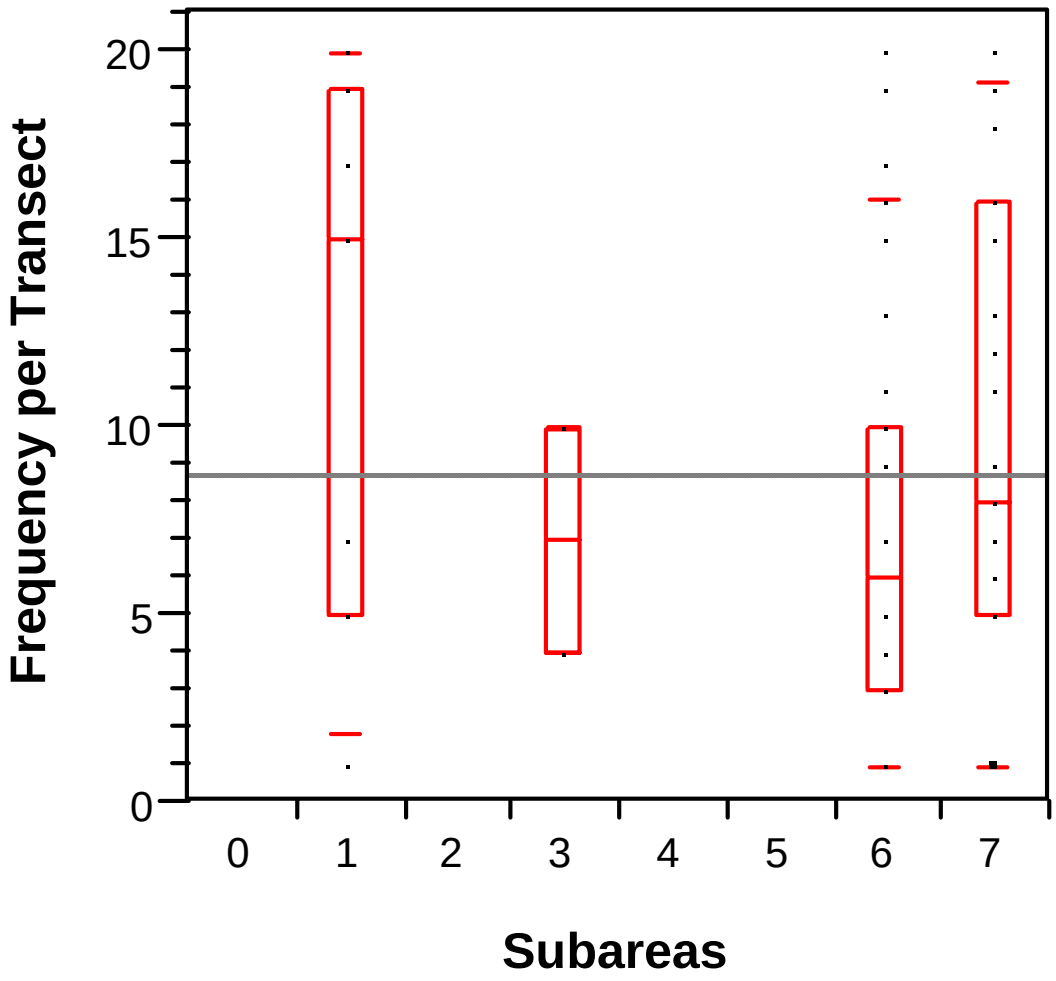


Figure 4.51. Distribution of *R. tracyi* by subarea.

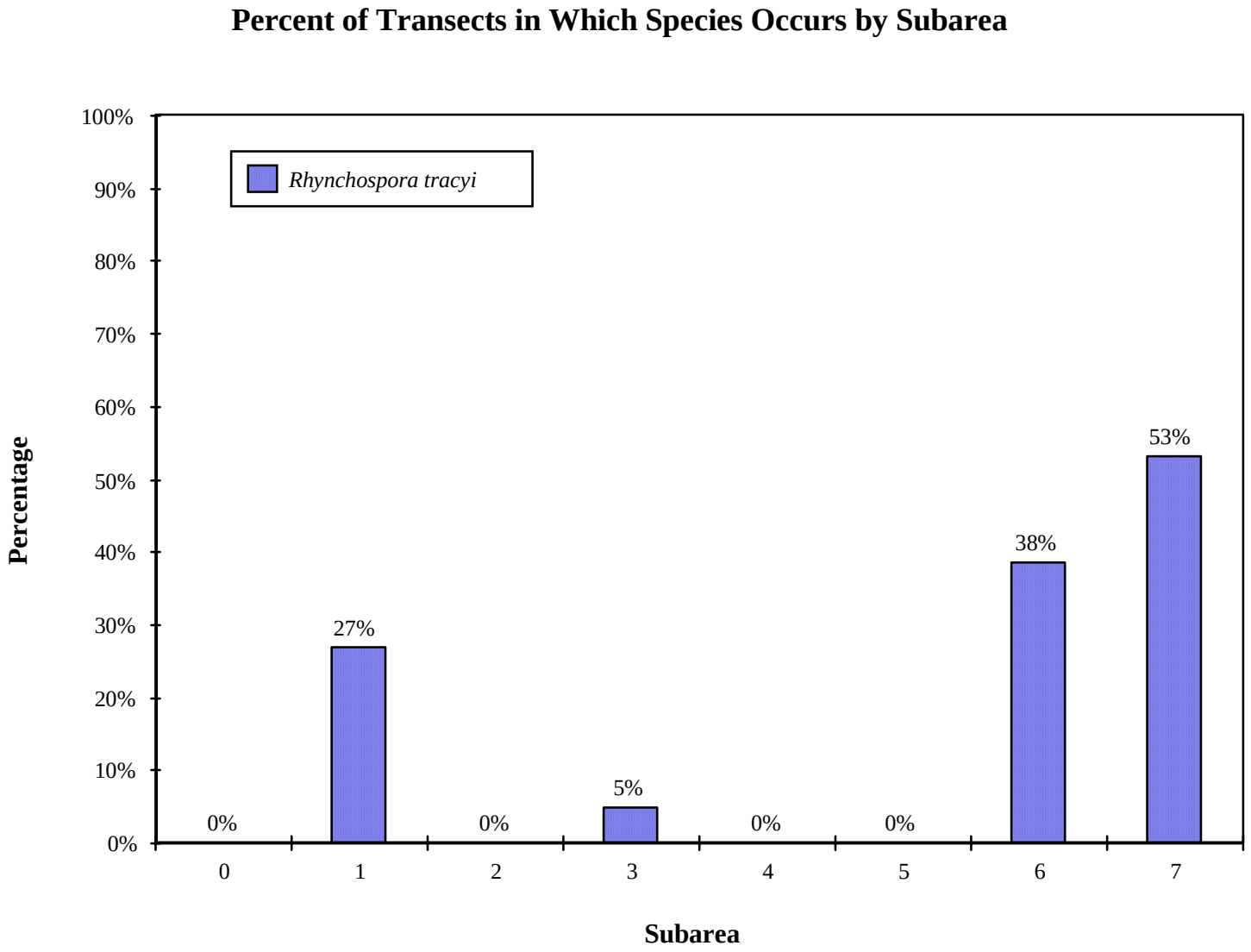


Figure 4.52. Percent of transects with *R. tracyi* by subarea.

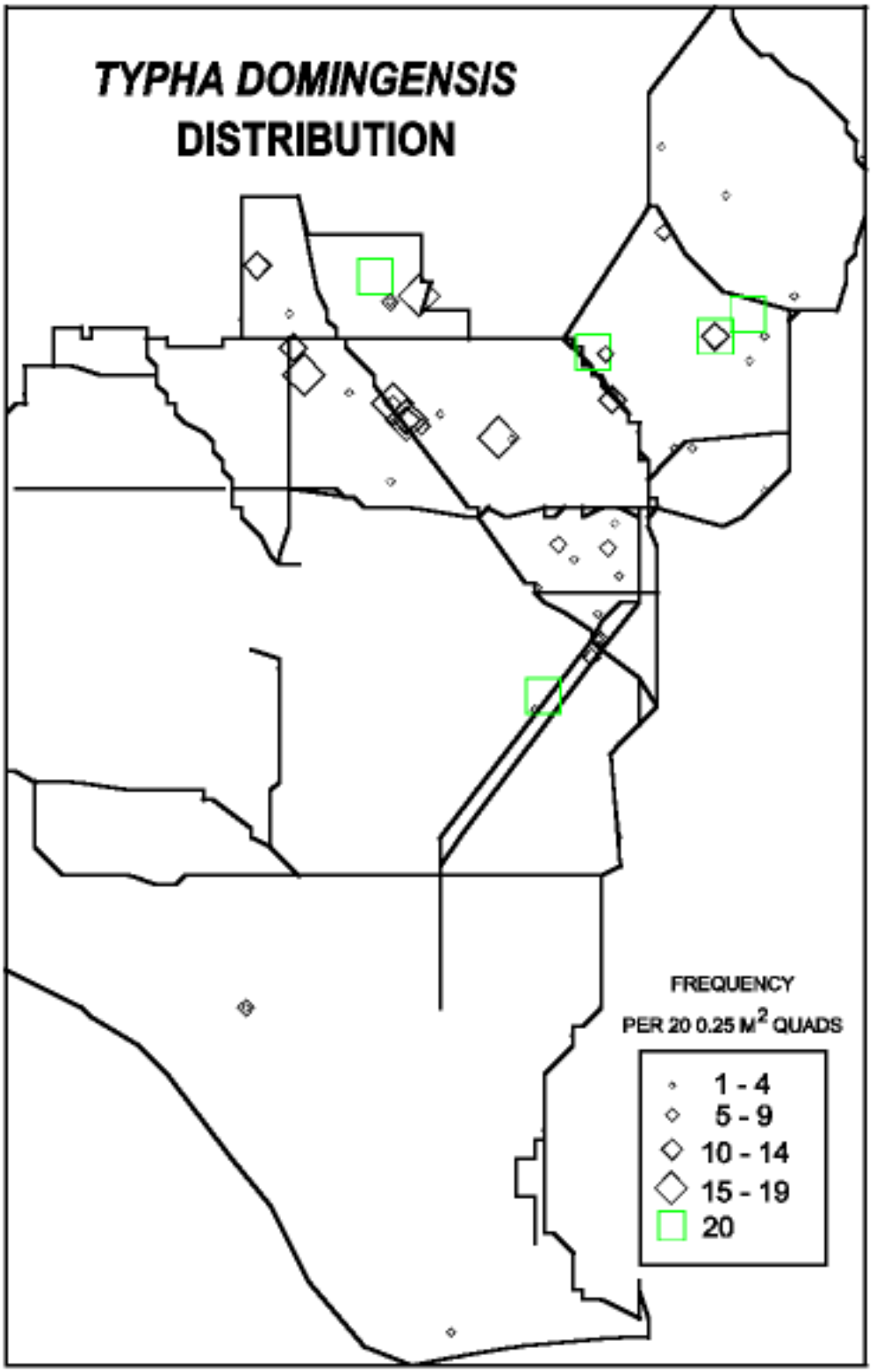


Figure 4.53. Distribution of *T. domingensis* in the study area.

Typha domingensis

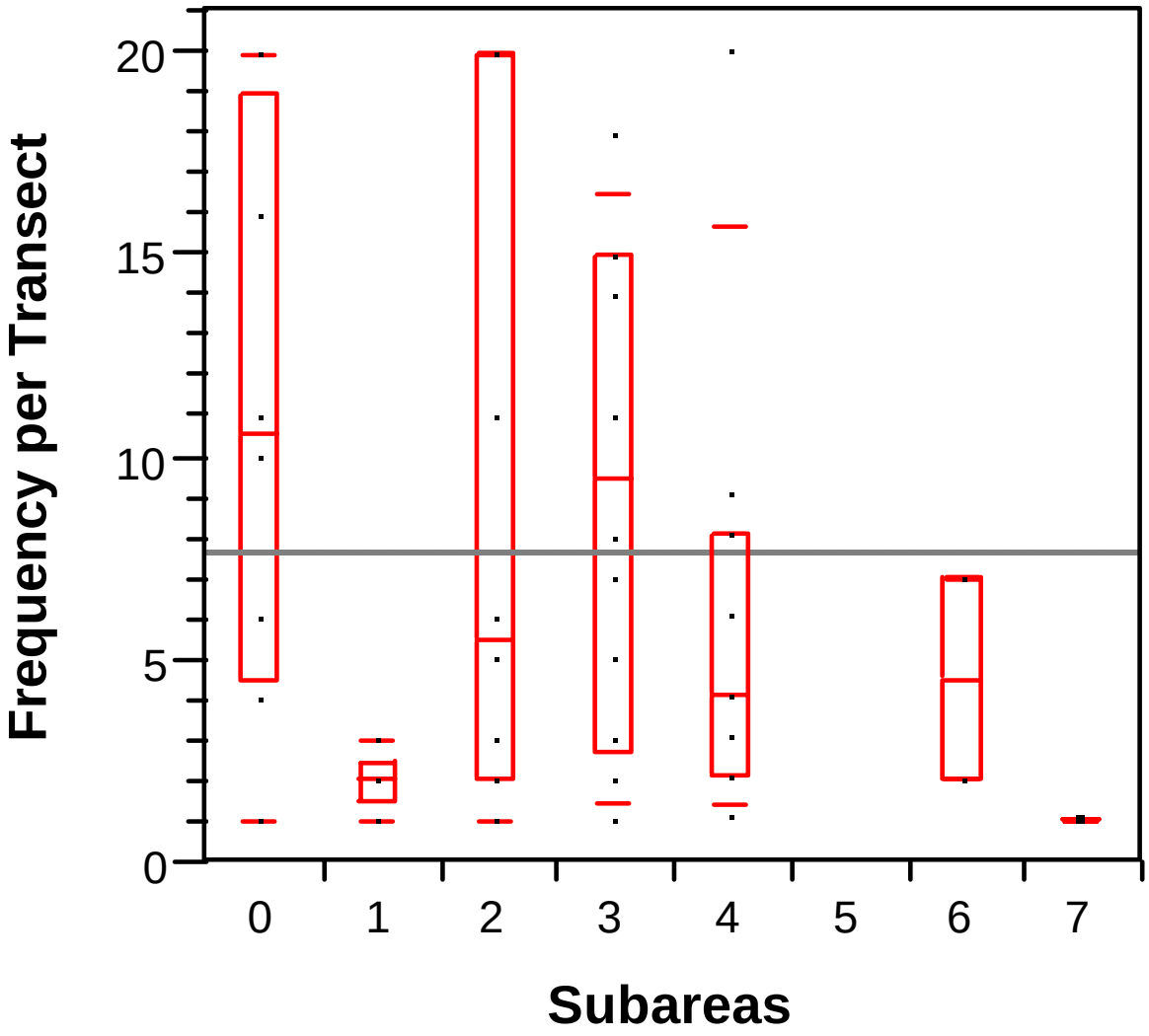


Figure 4.54. Distribution of *T. domingensis* by subarea.

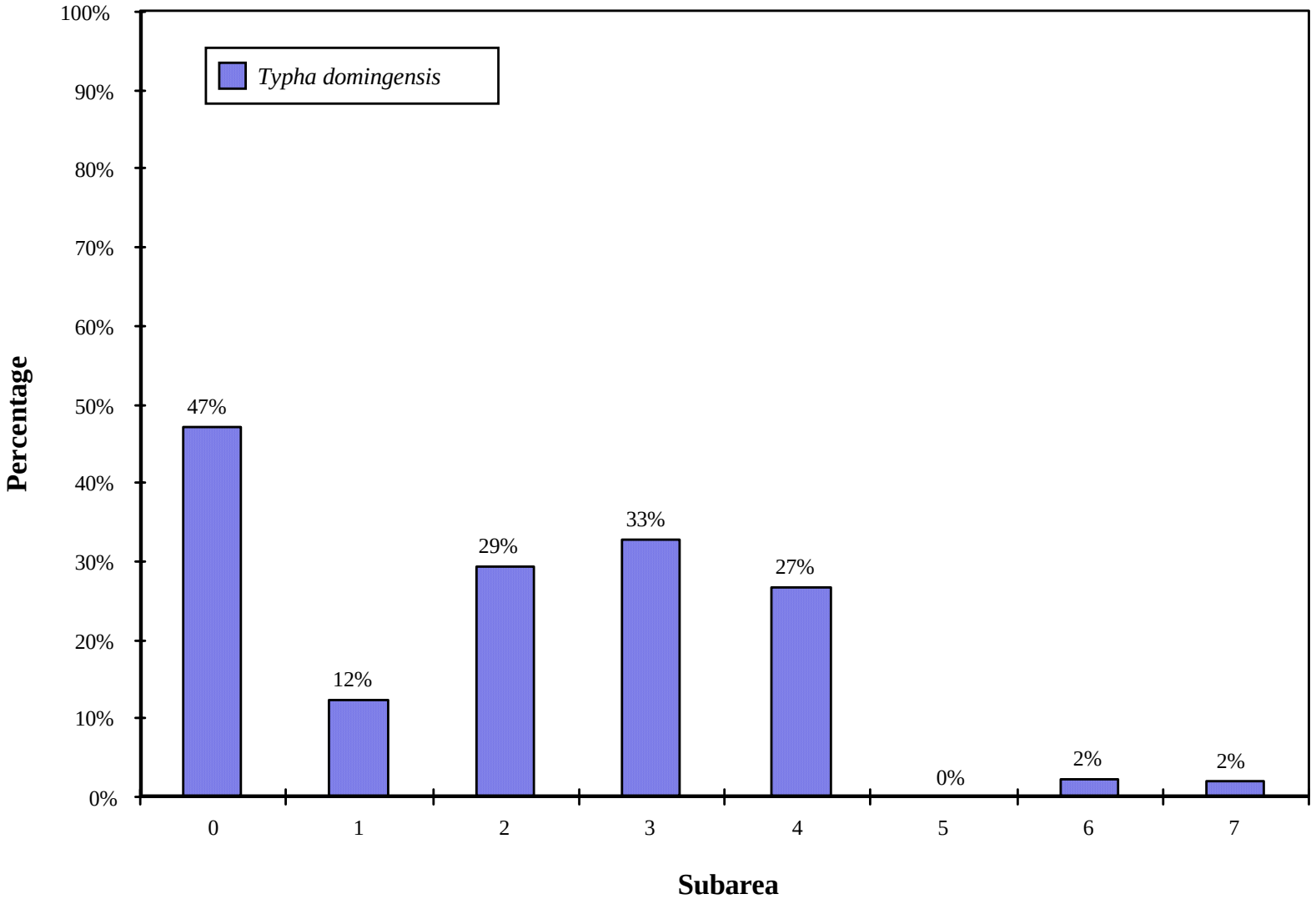


Figure 4.55. Percent of transects with *T. domingensis* by subarea.

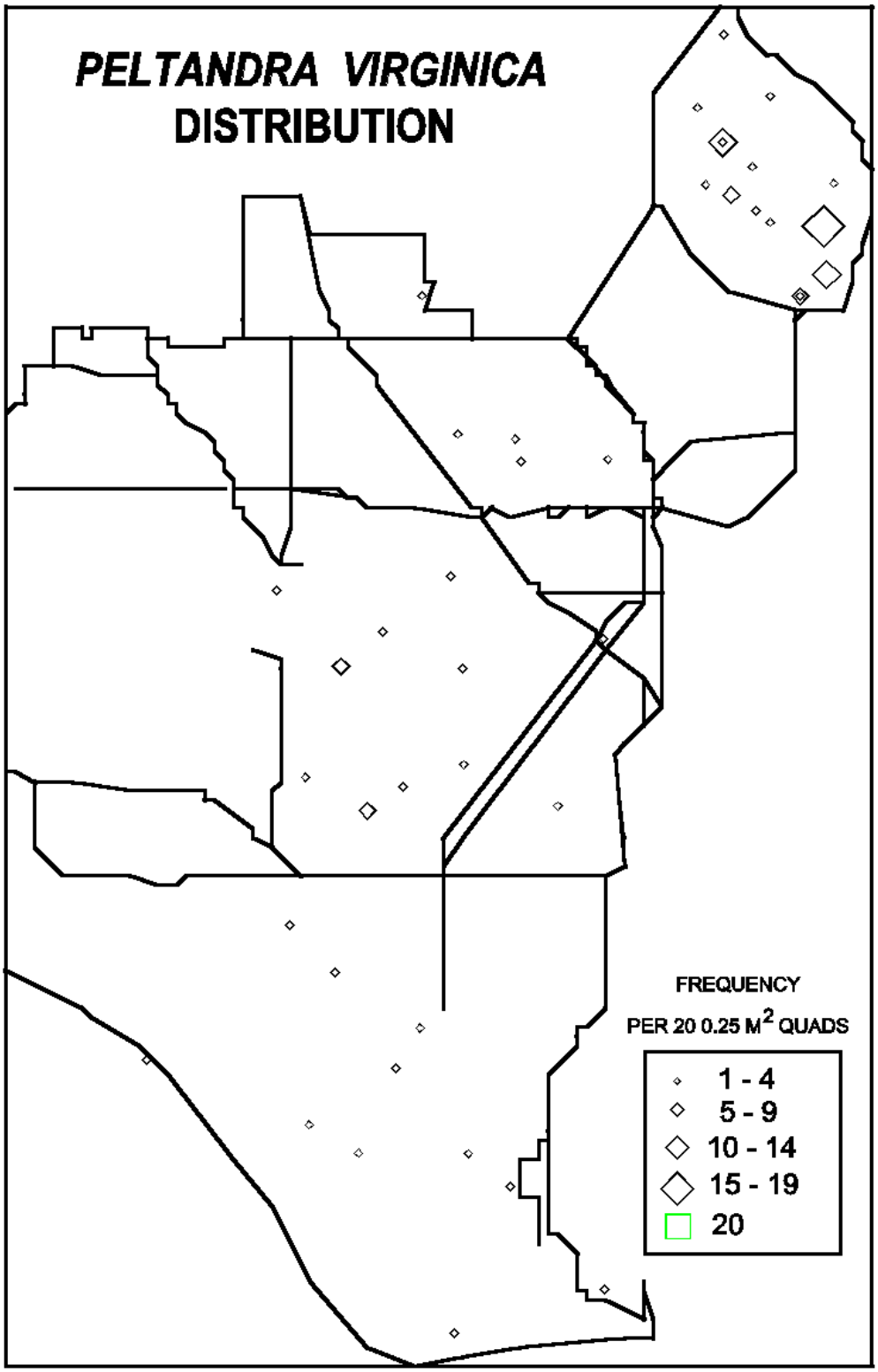


Figure 4.56. Distribution of *P. virginica* in the study area.

Peltandra virginica

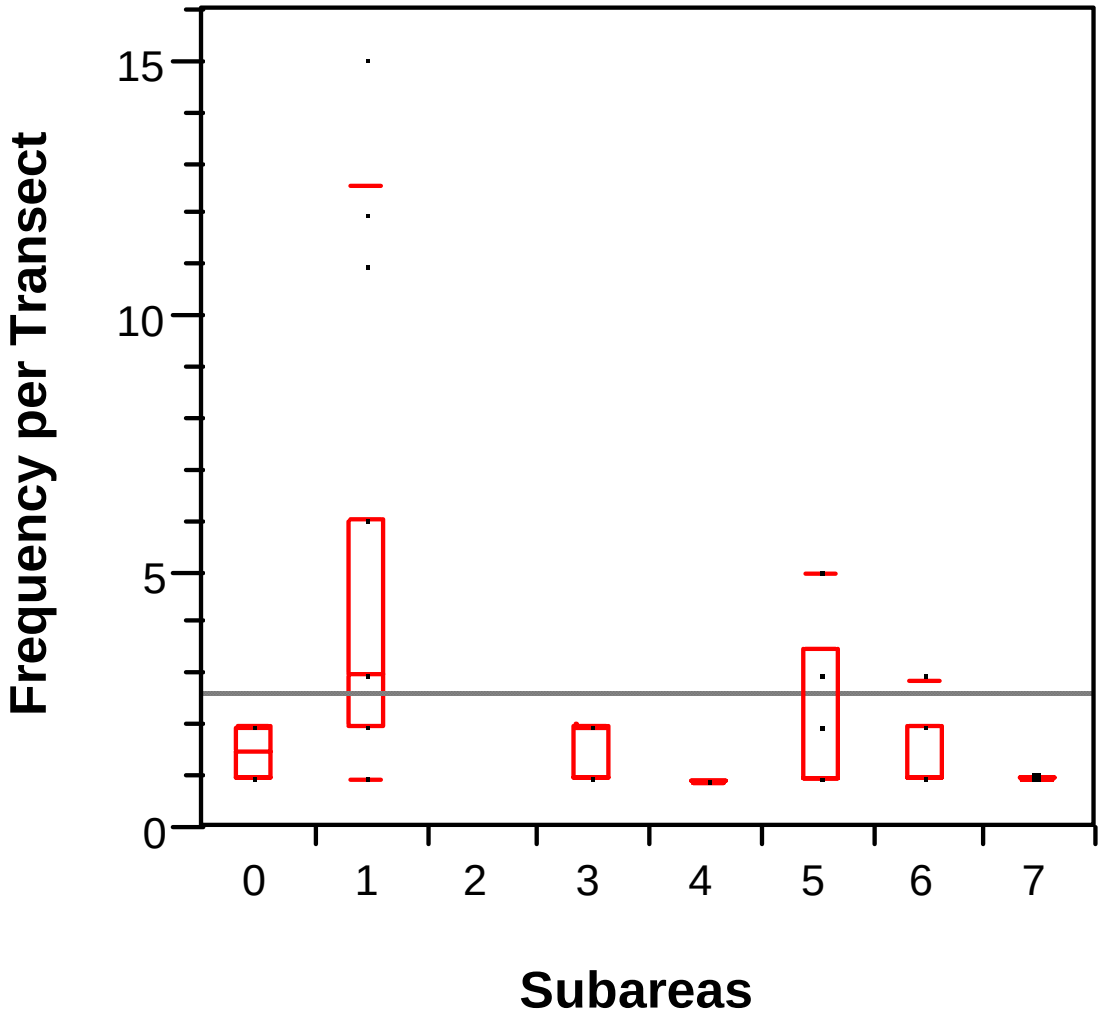


Figure 4.57. Distribution of *P. virginica* by subarea.

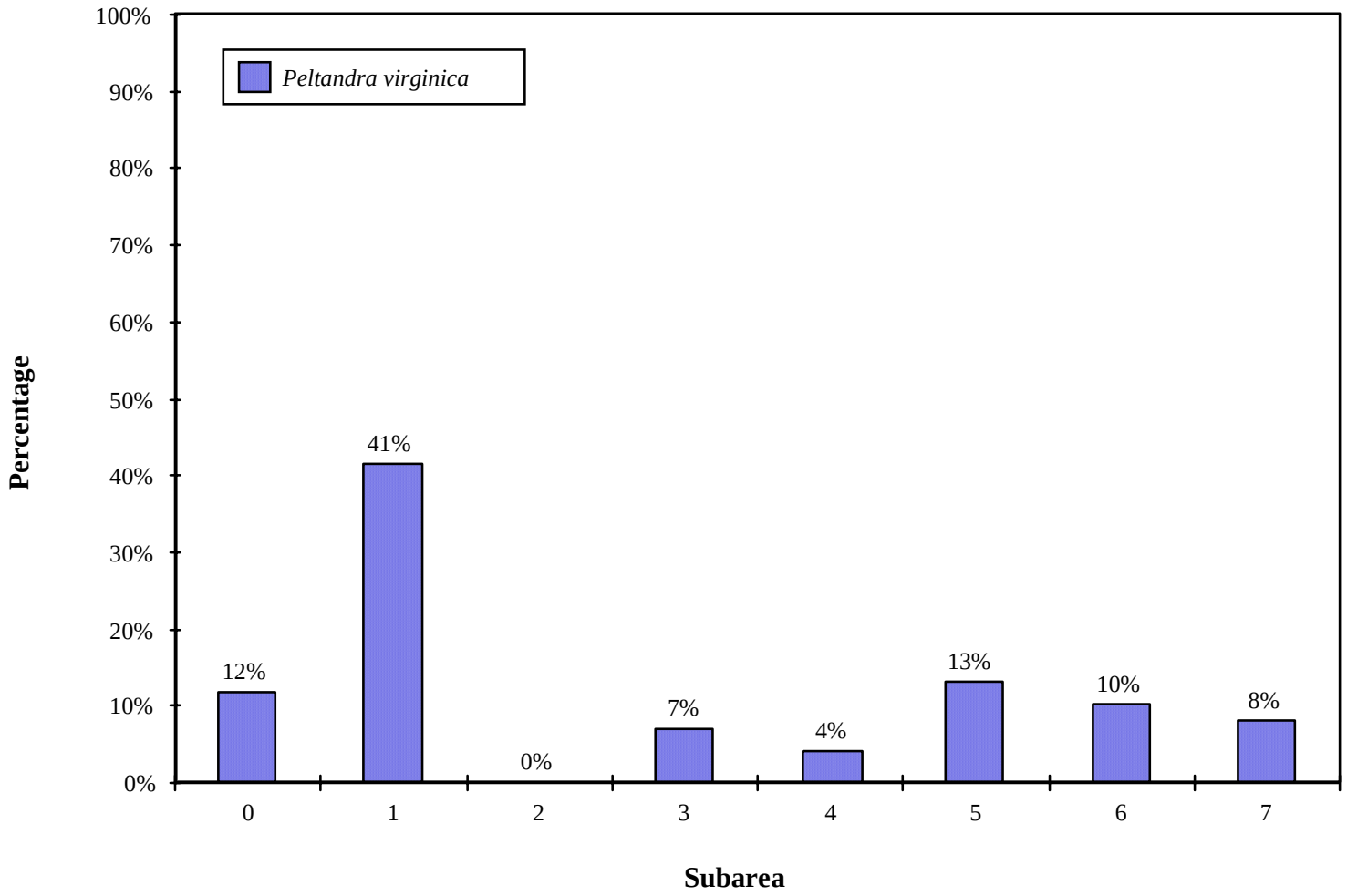


Figure 4.58. Percent of transects with *P. virginica* by subarea.

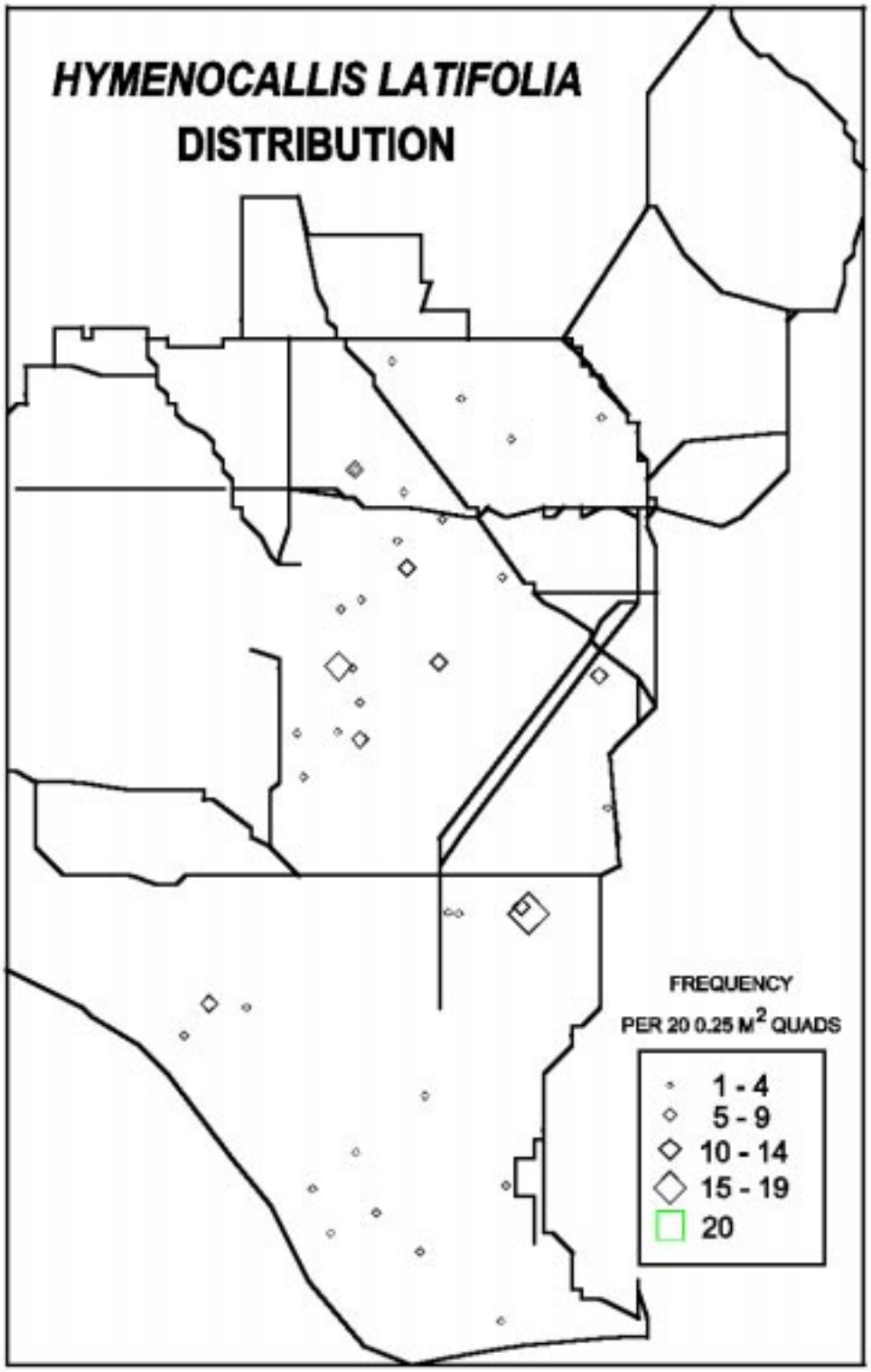


Figure 4.59. Distribution of *H. latifolia* in the study area.

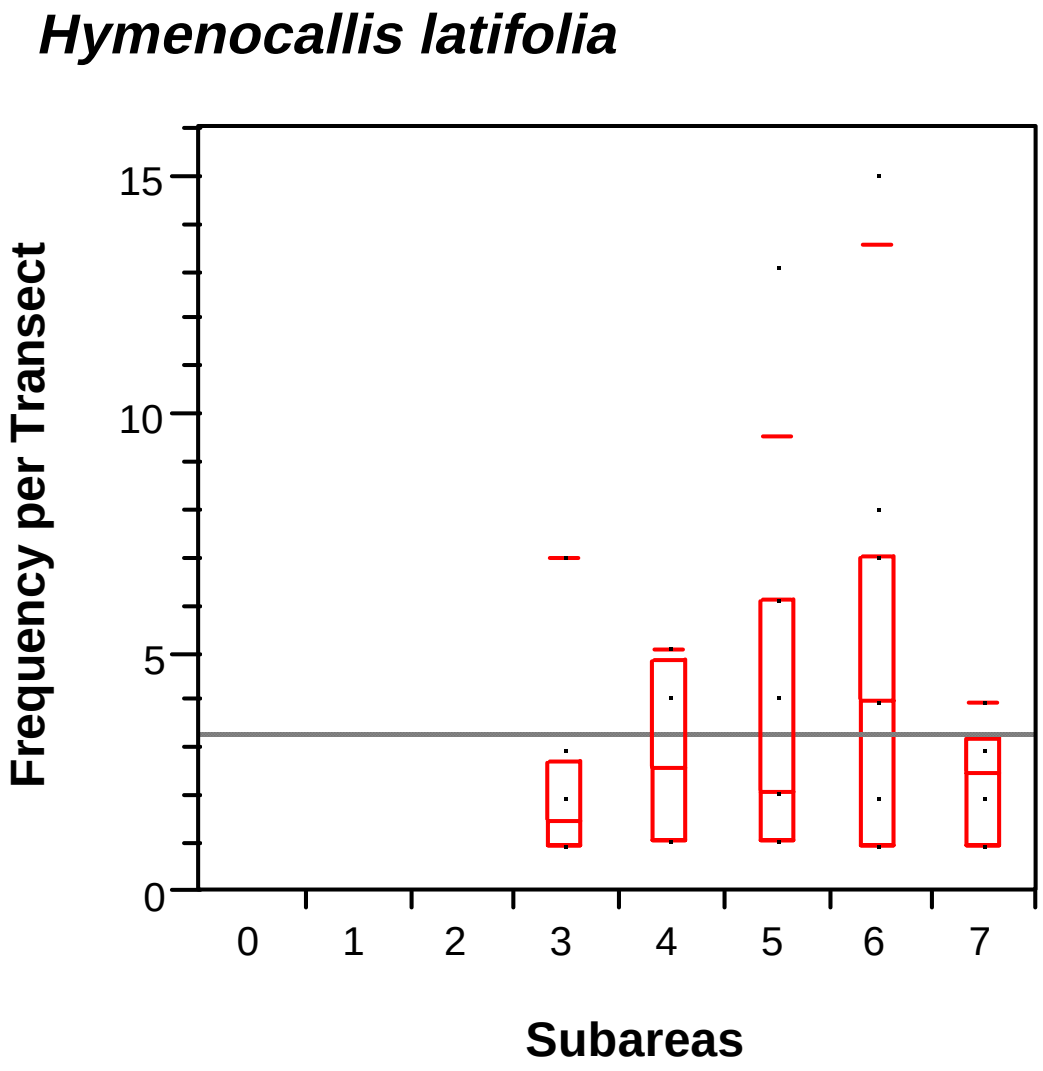


Figure 4.60. Distribution of *H. latifolia* by subarea.

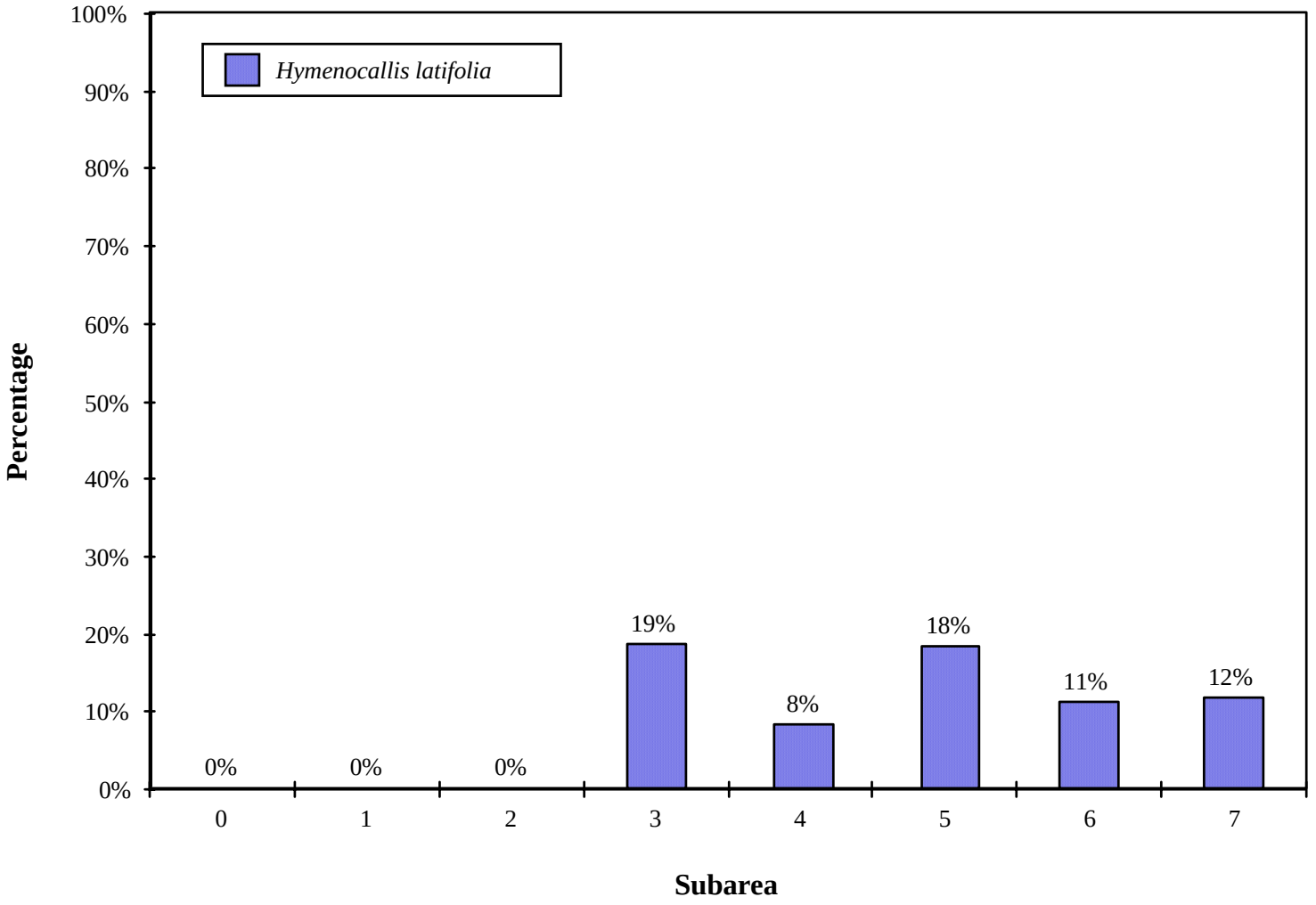


Figure 4.61. Percent of transects with *H. latifolia* by subarea.

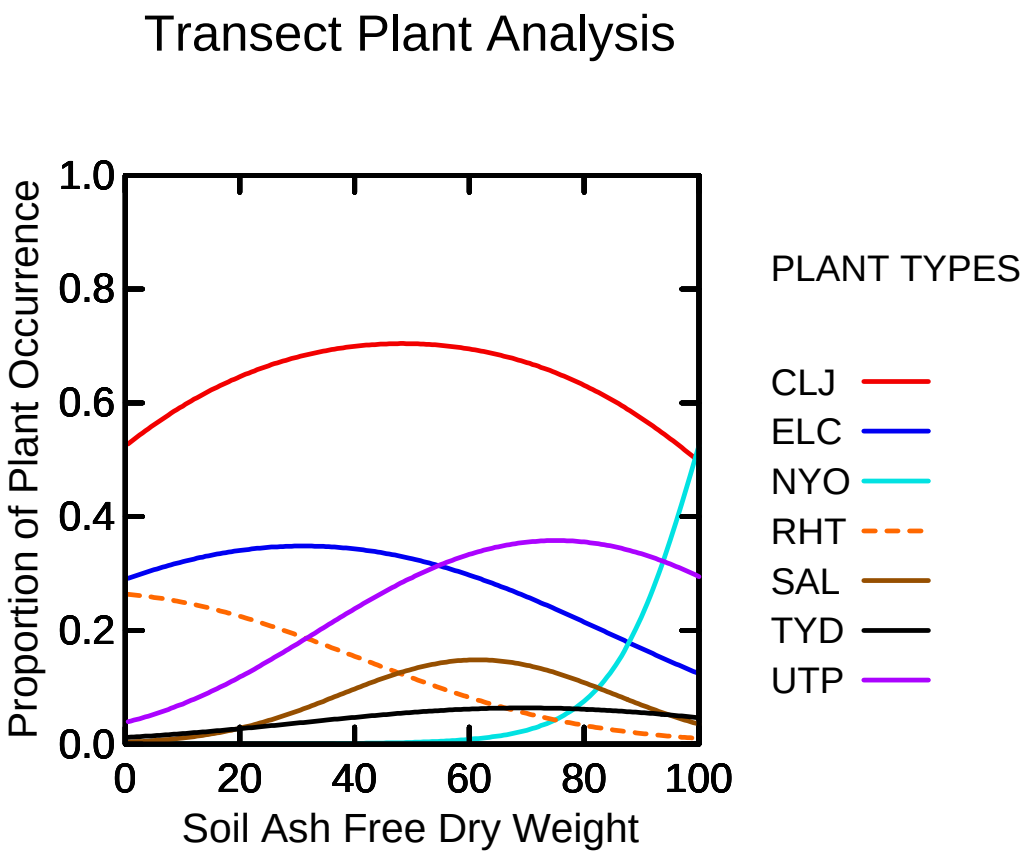


Figure 4.62. Logistic regression of AFDW to plant abundance.

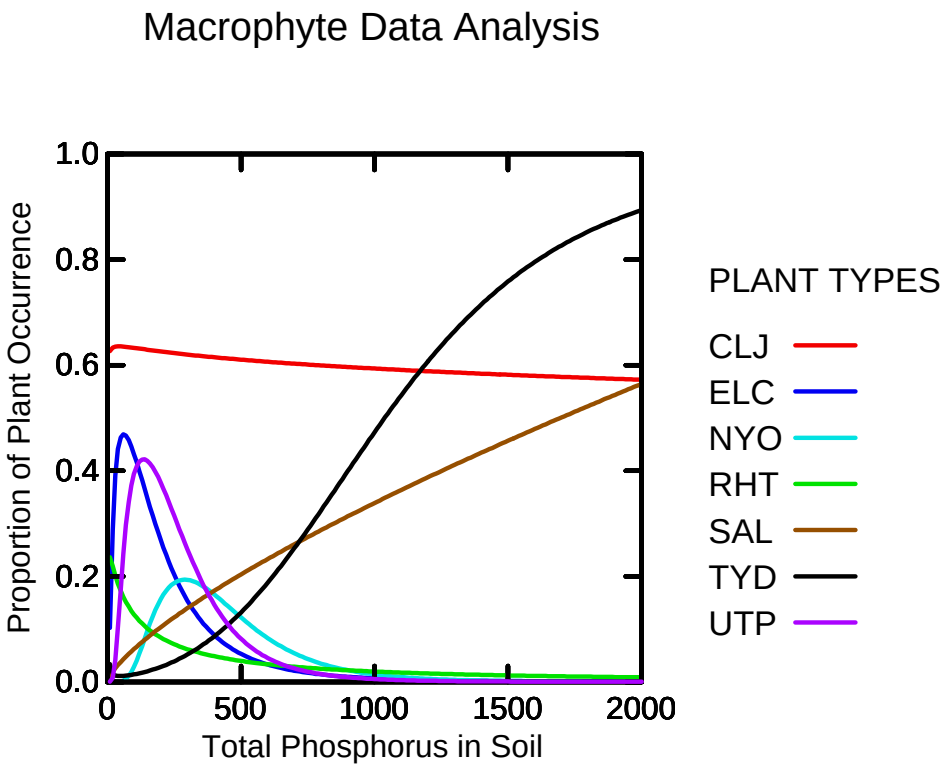
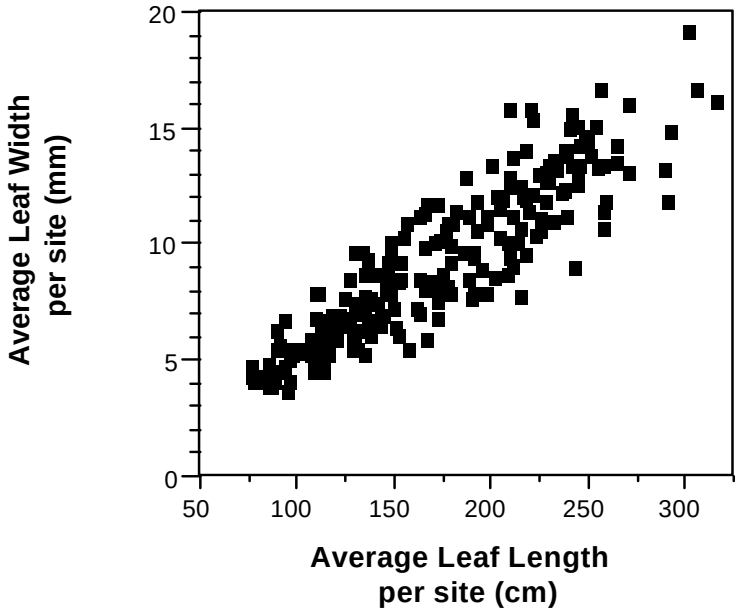


Figure 4.63. Logistic regression of soil TP to plant abundance.

A. ***Cladium jamaicense***



B. ***Sagittaria lancifolia***

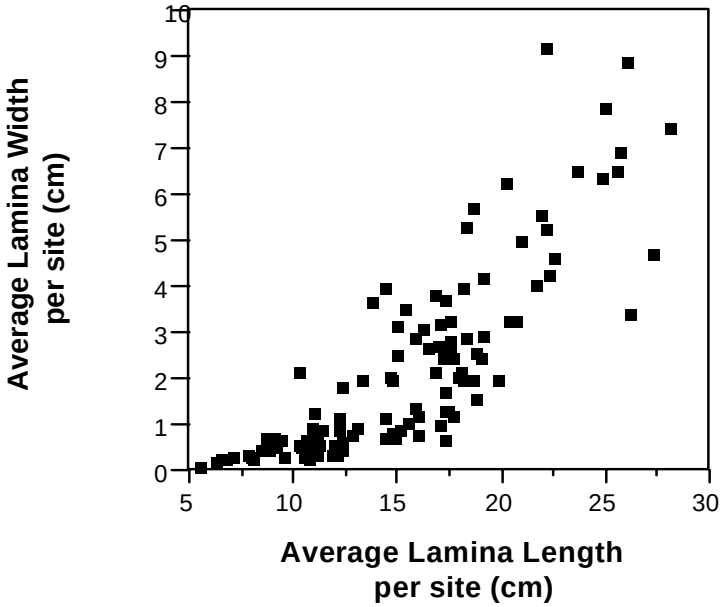


Figure 4.64. Scatterplot of average lamina length per site vs. lamina width per site for *Cladium jamaicense* (A) and *Sagittaria lancifolia* (B).

Sagittaria lancifolia

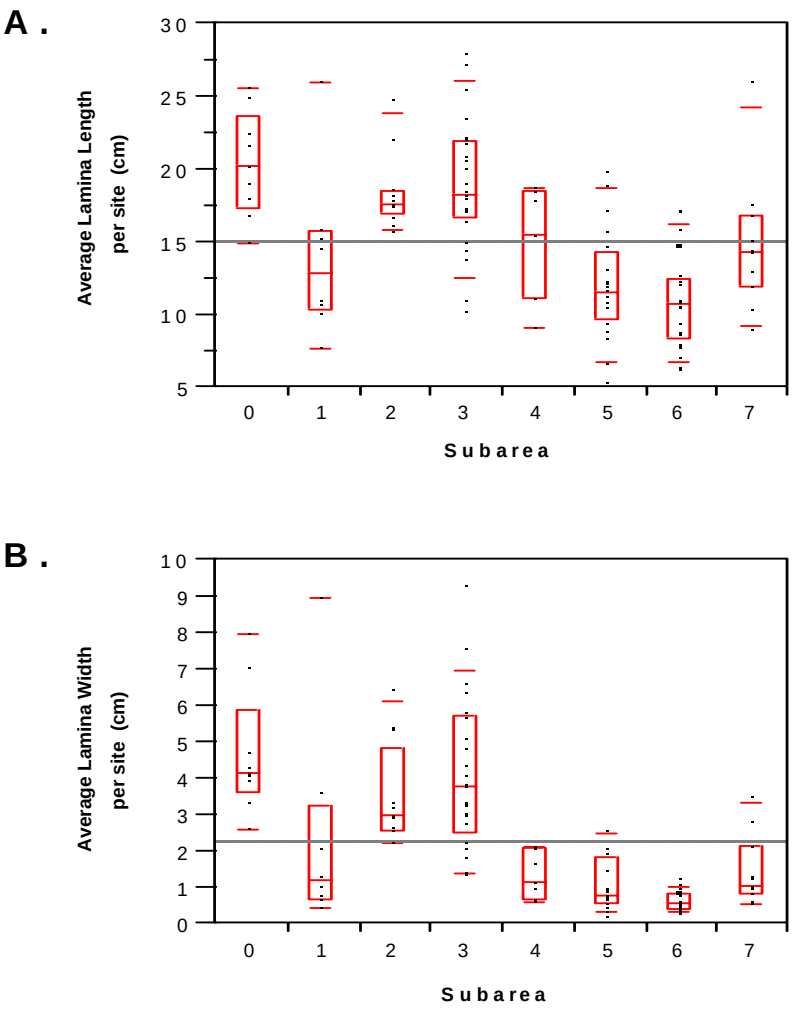


Figure 4.65. *Sagittaria lancifolia* average lamina length per site (A) and average lamina width per site (B) by subarea. Subarea 0 = Rotenberger-Holyland; 1 = Loxahatchee National Wildlife Refuge; 2 = WCA2; 3 = WCA3 north of Alligator Alley; 4 = southeastern WCA3; 5 = southwestern WCA3; 6 = Shark River Slough, Everglades National Park; 7 = Taylor Slough and southern Everglades National Park.

Sagittaria lancifolia

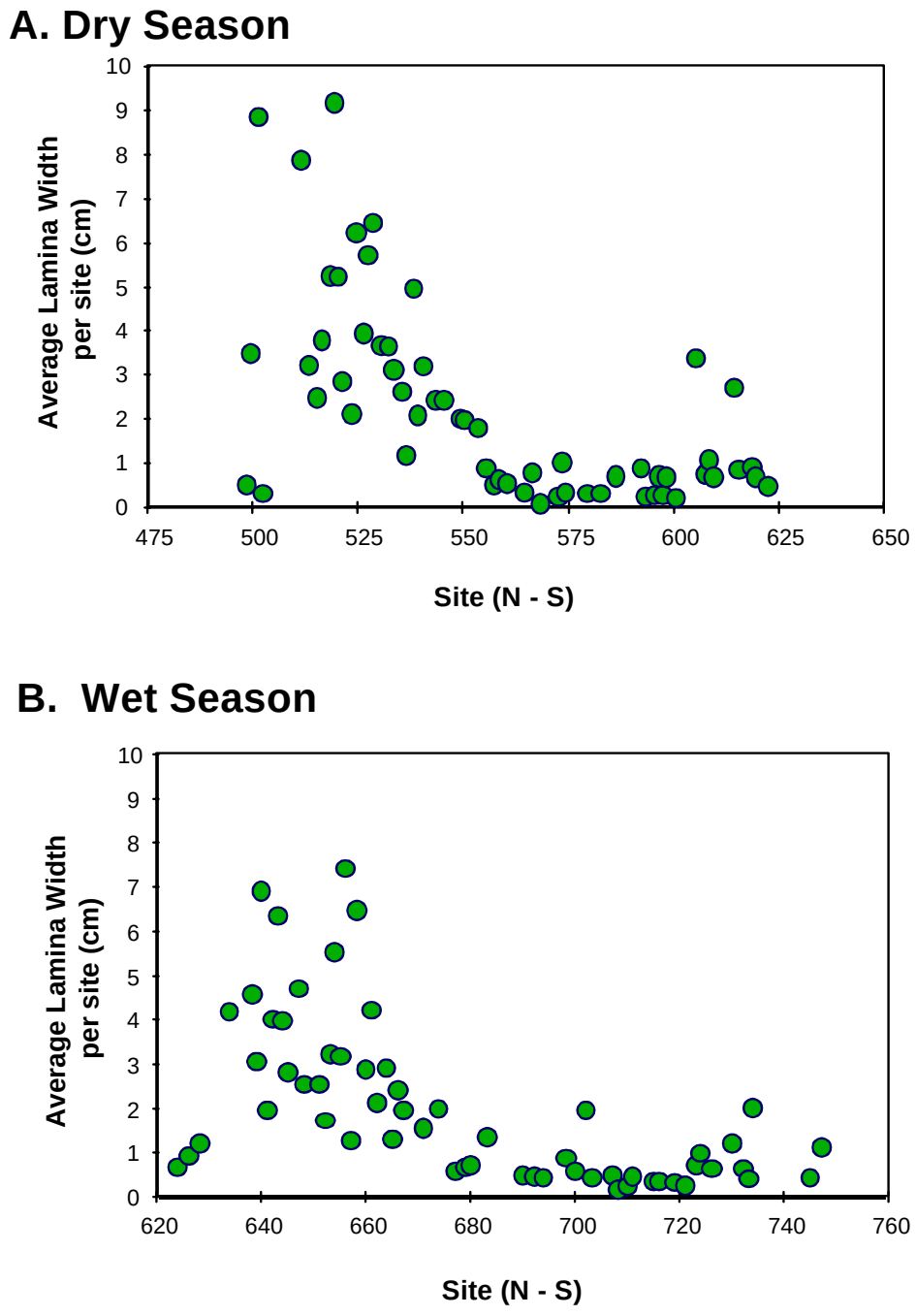


Figure 4.66. Scatterplot of *Sagittaria lancifolia* average lamina width per site for the May 1999, dry season sampling (A) and the Sept.-Oct., 1999, wet season sampling. Sites numbered north to south (cf.).

Cladium jamaicense

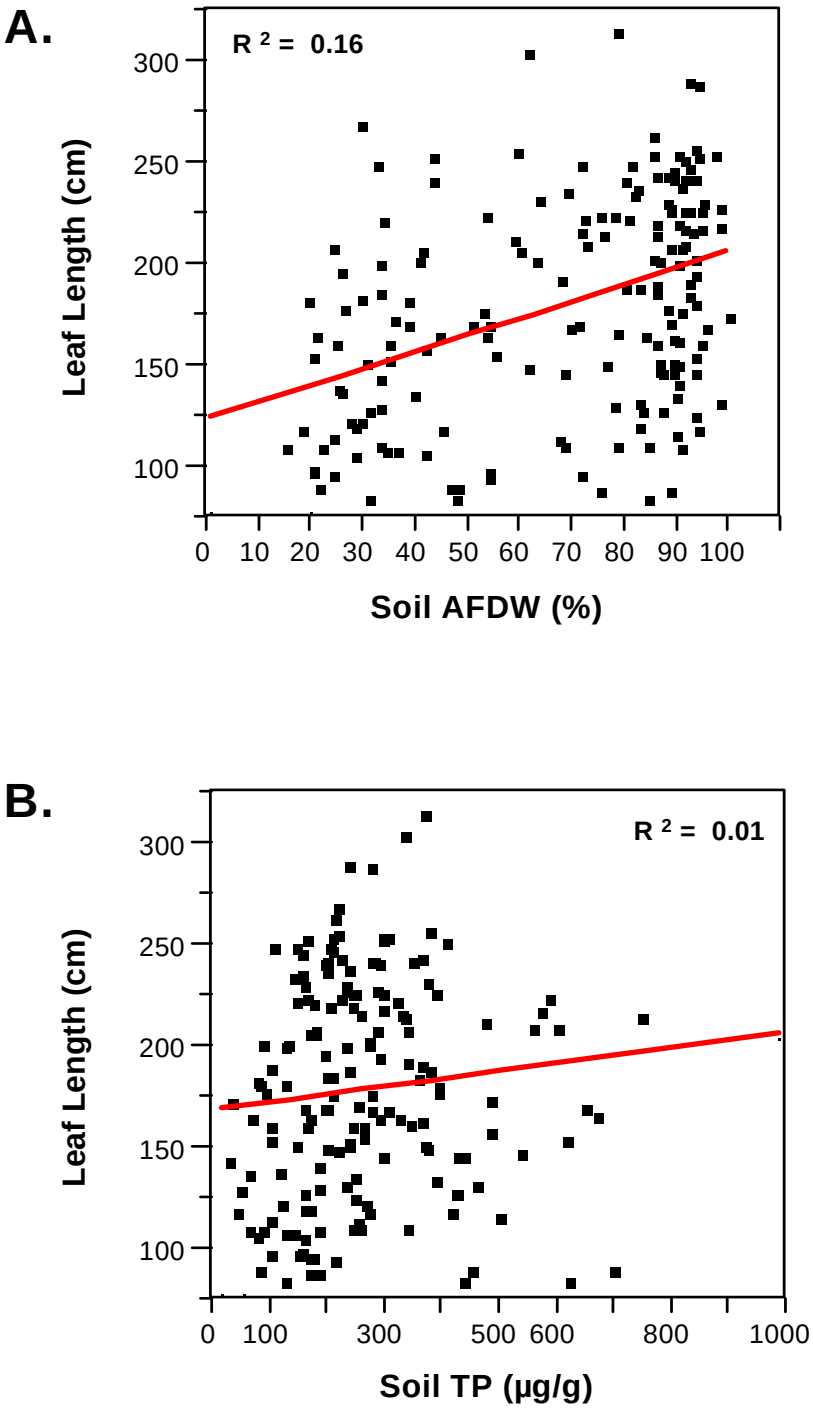


Figure 4.67. Scatterplot of (A.) soil ash-free dry weight vs. average leaf length per site and (B.) soil total phosphorus vs. average leaf length per site for *Cladium jamaicense.*

Sagittaria lancifolia

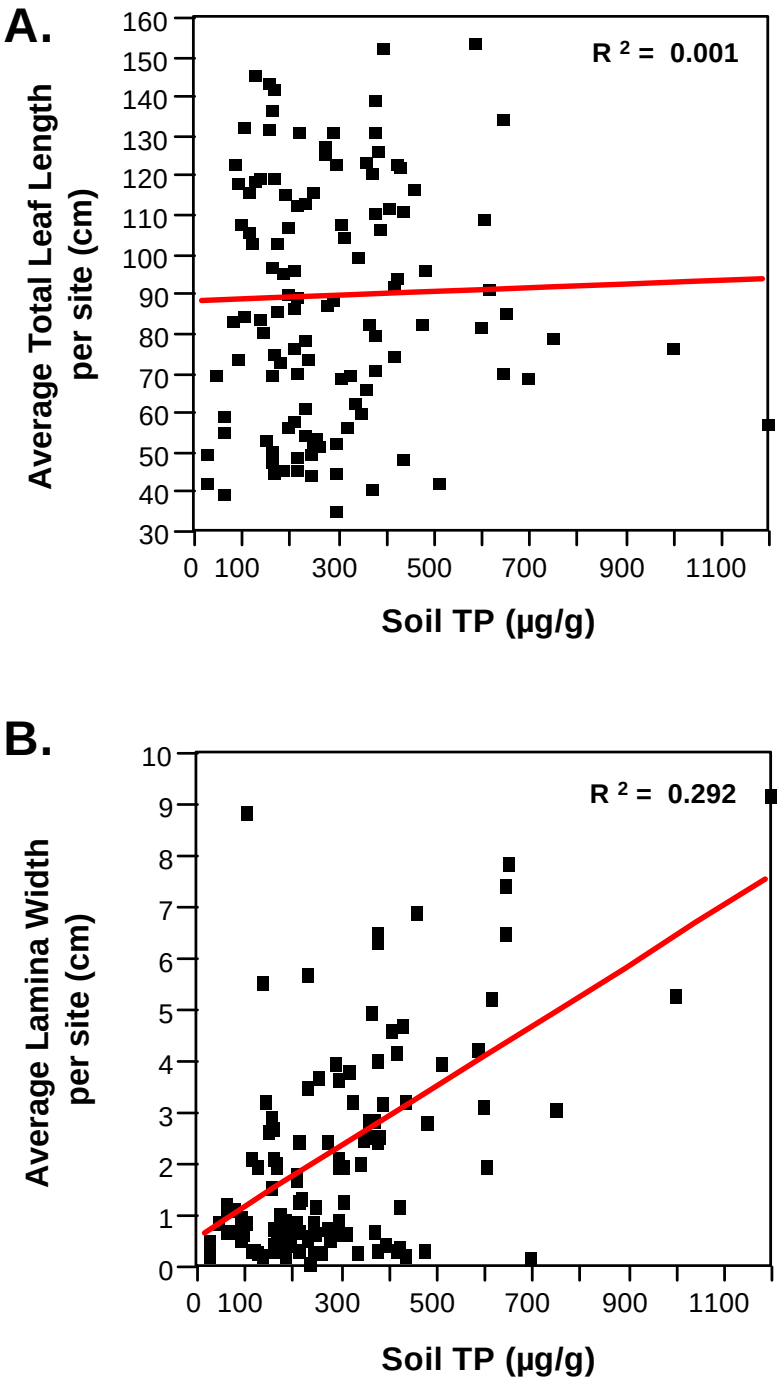


Figure 4.68. Scatterplot of (A.) soil total phosphorus vs. average total leaf length per site and (B.) soil total phosphorus vs. average lamina width per site for *Sagittaria lancifolia*.

Sagittaria lancifolia

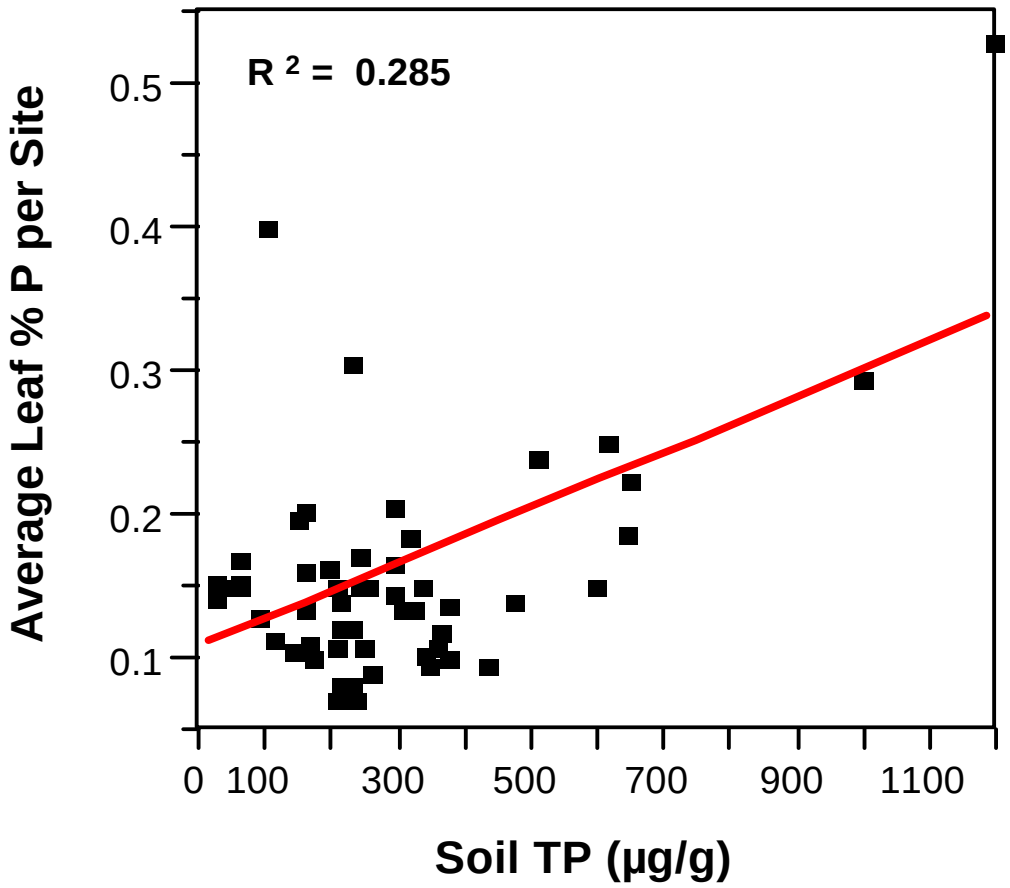


Figure 6.69. Scatterplot of soil total phosphorus vs. average leaf % phosphorus per site for *Sagittaria lancifolia*.

Sagittaria lancifolia

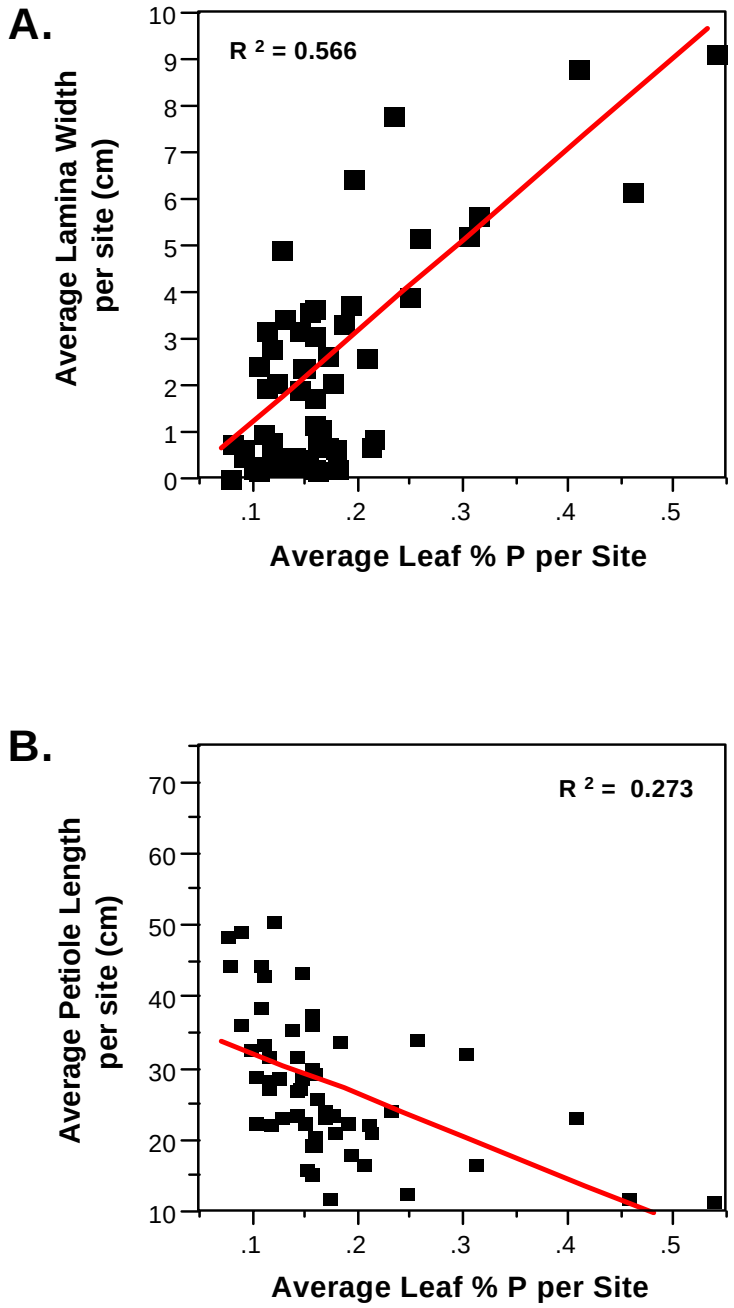


Figure 4.70. Scatterplot of (A.) average leaf % phosphorus per site vs. average lamina width per site and (B.) for average leaf % phosphorus per site vs. average petiole length per site for *Sagittaria lancifolia*.

Sagittaria lancifolia

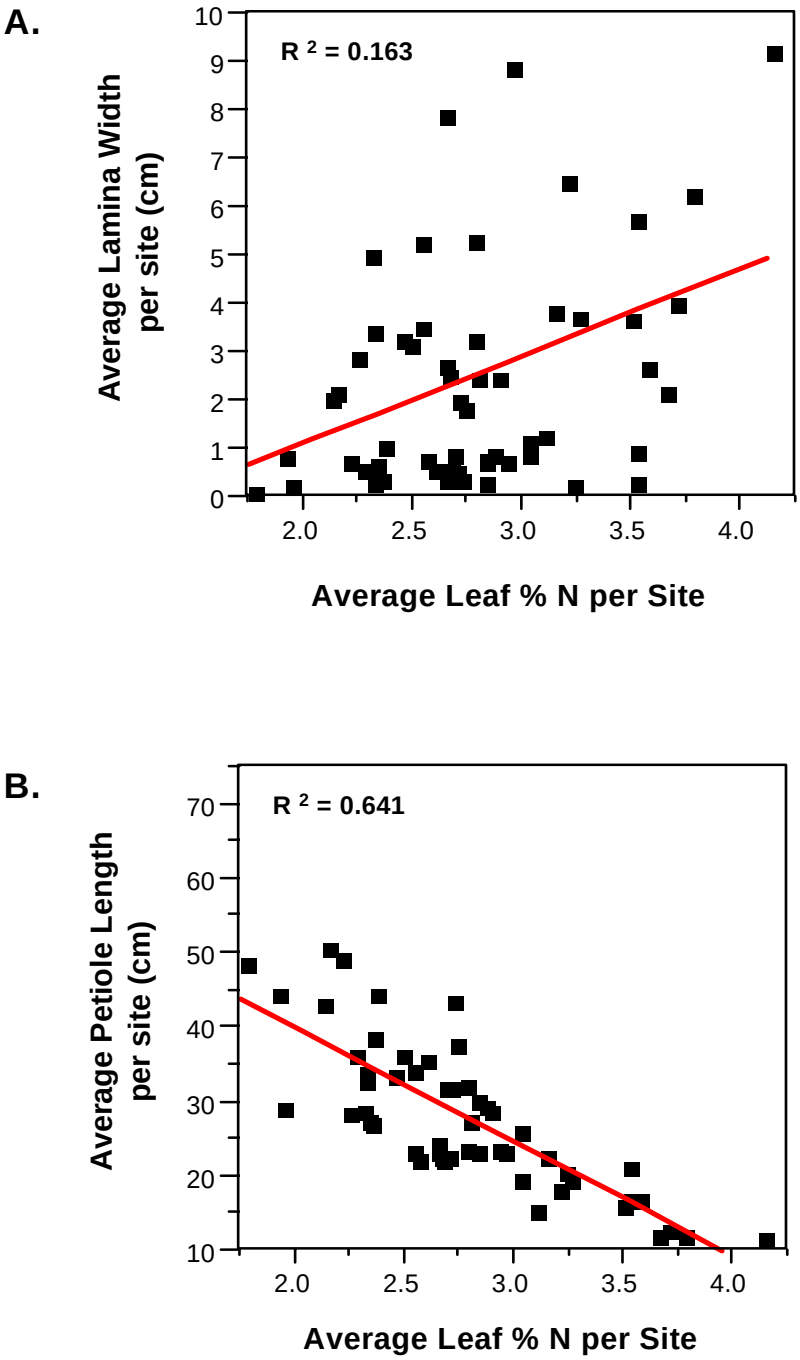
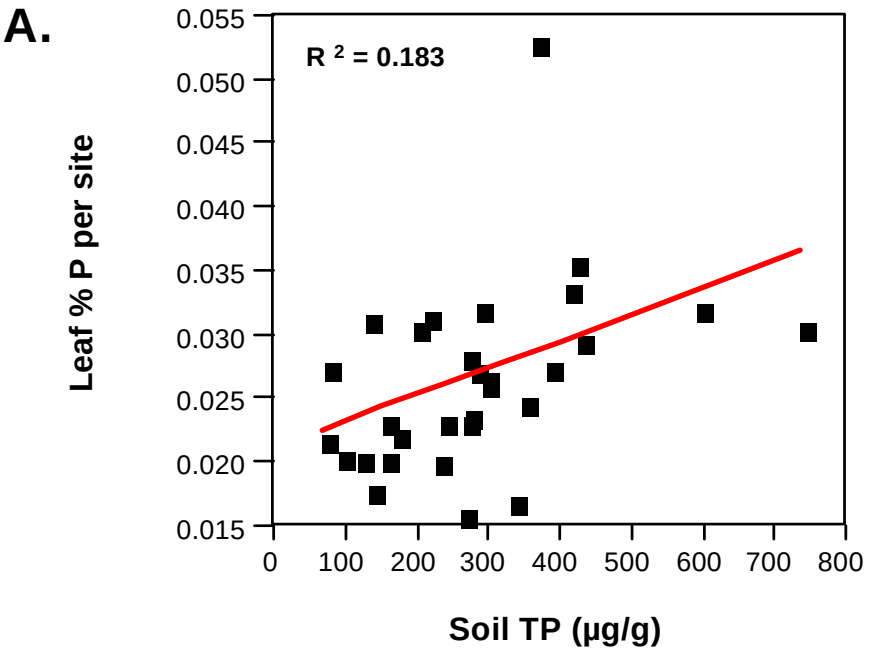


Figure 4.71. Scatterplot of (A.) average % leaf nitrogen per site vs. average lamina width per site and (B.) for average % leaf nitrogen per site vs. average petiole length per site for *Sagittaria lancifolia*.

Cladium jamaicense



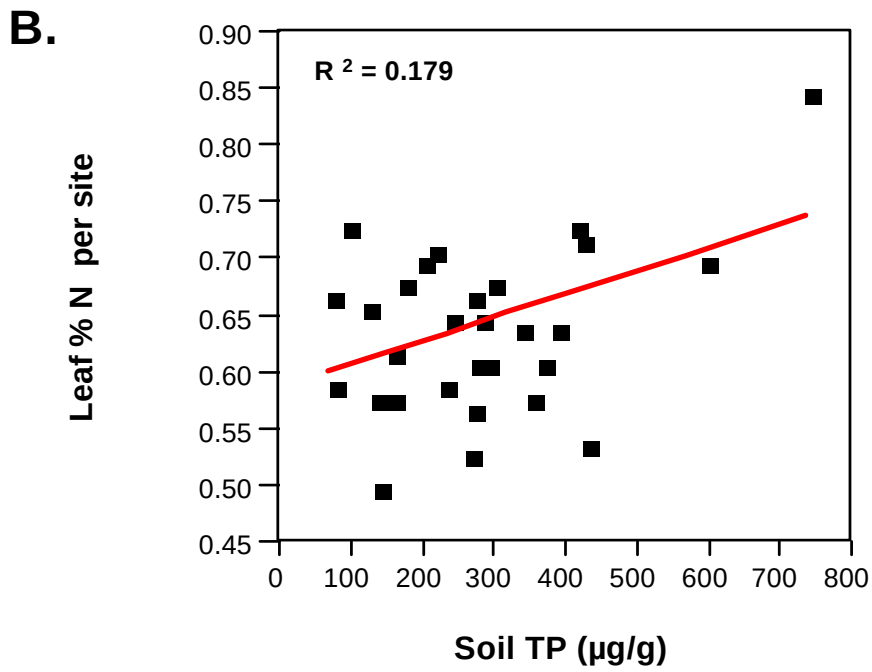


Figure 4.72. Scatterplot of (A.) soil total phosphorus per site vs. average leaf % phosphorus per site and (B.) soil total phosphorus per site vs. average leaf % nitrogen per site for *Cladium jamaicense.*

Cladium jamaicense

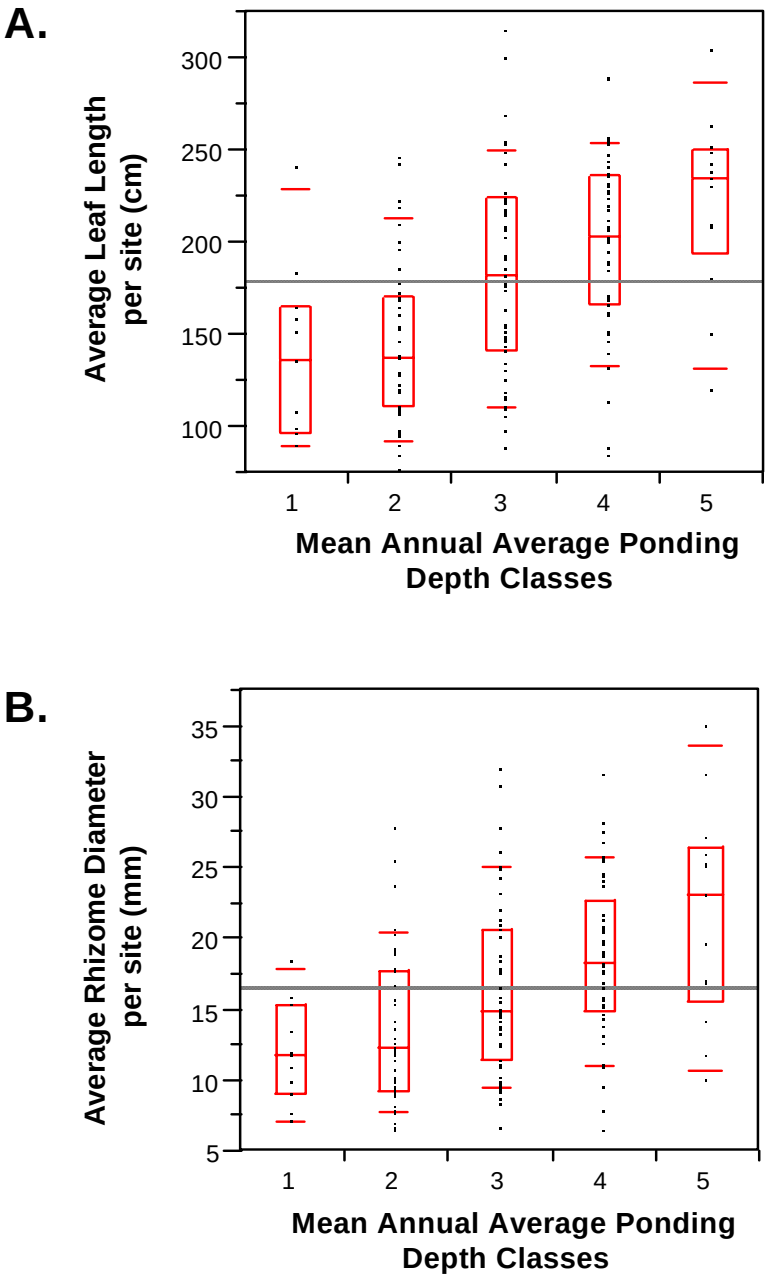


Figure 4.73. *Cladium jamaicense* average leaf length per site (A.) and average rhizome diameter per site (B) by mean annual average ponding depth class. 1 = 0 to 0.1 ft.; 2 = 0.1 to 0.5 ft.; 3 = 0.5 to 1.0 ft.; 4 = 1.0 to 2.0 ft.; 5 = 2.0 to 3.0 ft.; 6 = more than 3 ft.

Sagittaria lancifolia

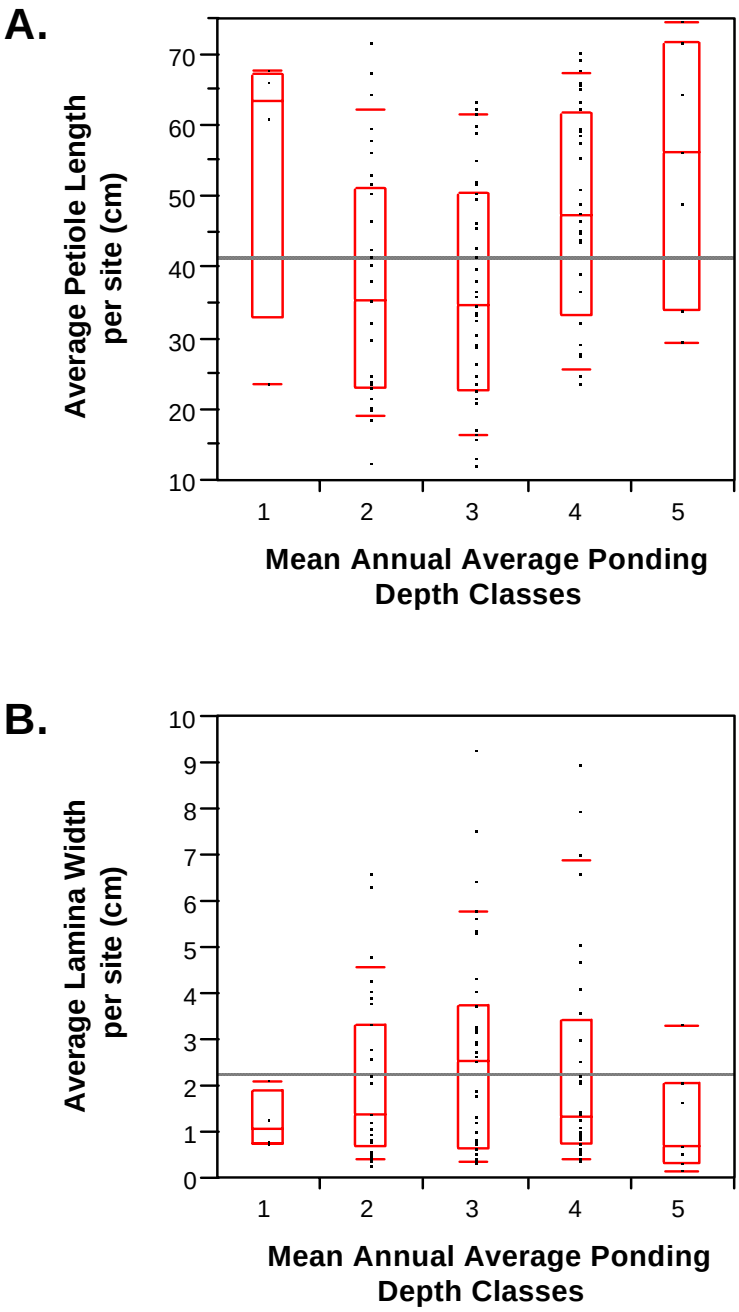


Figure 4.74. *Sagittaria lancifolia* average petiole length per site (A.) and average lamina width per site (B.) by mean annual average ponding depth classes. 1 = 0 to 0.1 ft.; 2 = 0.1 to 0.5 ft.; 3 = 0.5 to 1.0 ft.; 4 = 1.0 to 2.0 ft.; 5 = 2.0 to 3.0 ft.; 6 = more than 3 ft.

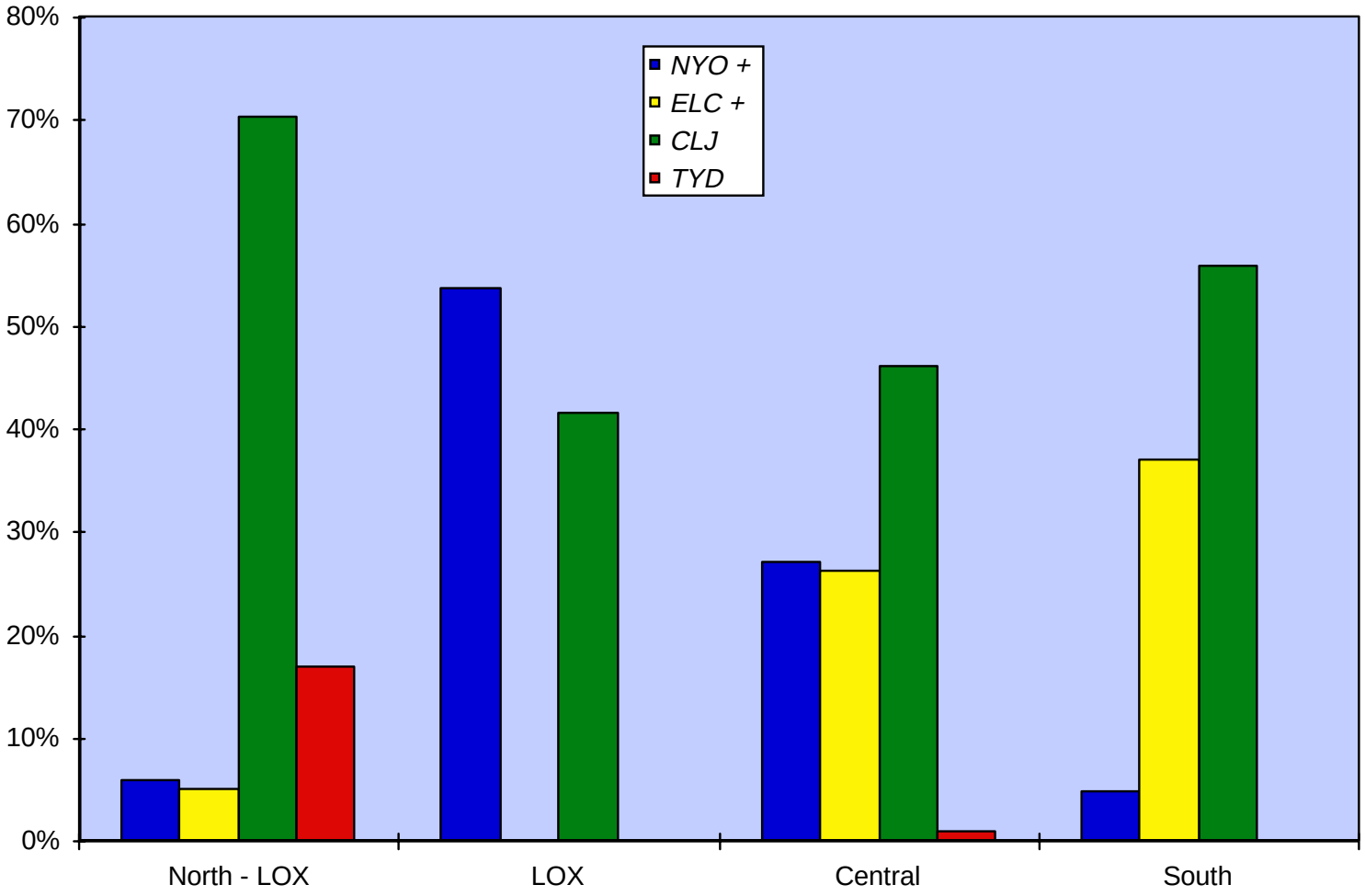


Figure 4.75. Frequency of major plant clusters in areas of the Everglades.

5.0 PERIPHYTON DISTRIBUTION

5.1 Periphyton Importance in the Everglades Ecosystem

Periphyton is a dominant and conspicuous component in most of the Everglades marsh. Periphyton mats contain a mixed and tightly organized assemblage of autotrophic microalgae, heterotrophic bacteria and associated macrophyte plants and detritus. Natural assemblages are productive (Browder et al. 1982), providing the primary source of fixed carbon to the food web (Turner et al. 1999) and influencing water and soil quality through their metabolism (Gleason & Spackman 1974).

Native Everglades periphyton communities are threatened by disturbances that alter their structure and disrupt functional processes that maintain their natural organization. Examples include documented compositional and physiological responses to nutrient enrichment (Swift & Nicholas 1987, Raschke 1993, Vymazal & Richardson 1995, McCormick et al. 1996, McCormick & O'Dell 1996) and structural responses to changes in hydrologic regimes (Browder et al. 1982). Because periphyton communities integrate short-term variation in their physical and chemical environment, measures of their condition in terms of productivity, biomass, species composition or nutrient content can provide more reliable assessments of water quality than single point physicochemical measures. While there have been several attempts to develop periphyton-based indices of nutrient enrichment in the Everglades (McCormick & Stevenson 1998), their application throughout the Everglades must be approached cautiously since their performance has not been evaluated outside the few localities where the data originated. Furthermore, the usefulness of periphyton in assessing other elements of water quality and quantity (i.e., ion content, mercury contamination, hydroperiod, water depth) has not been examined in the Everglades. However, numerous studies elsewhere have shown algae, particularly diatoms, to be useful in tracking changes in ion concentrations (i.e., acidification Dixit et al. 1992); hydroperiod (Gaiser et al. 1998), water depth (Pienitz et al. 1995), and salinity (Fritz et al. 1991) in addition to assessing nutrient enrichment (Dixit et al. 1992).

For these reasons, an analysis of diatom composition was incorporated into the 1999 REMAP assessment protocols. The specific goals were to: (1) provide spatially intensive baseline data from sites throughout the Everglades for future use in tracking natural or anthropogenic long-term environmental change; (2) describe current spatial patterns in diatom

species composition and their relationships to critical environmental parameters; and (3) use these relationships to make predictions about community change under different management scenarios. The specific sampling and analysis procedures were discussed in Chapter 3 *Materials and Methods*.

5.2 Periphyton Presence and Growth Form

Periphyton was found at 78 and 49 sites during cycles 4 and 5, respectively. Figures 5.1 and 5.2 show the distribution of periphyton in the 3 different growth forms across the Everglades. Periphyton aggregations were notably absent or rare in the Rotenberger, Holeylands, Lox, WCA-2 and WCA3-N. Sites in Everglades National Park (Shark River and Taylor Sloughs) were dominated by soil-associated benthic mats in the dry season (Cycle 4). During the wet season (Cycle 5), Taylor Slough was still dominated by benthic mats, while Shark Slough contained more floating periphyton, some of which was associated with *Utricularia purpurea*. Seasonal and spatial transitions from benthic to floating aggregations are common responses to variations in water depth and substrate availability (Browder et al. 1982). Periphyton in the Water Conservation Areas was confined mostly to floating mats and *Utricularia*, although benthic communities were found at some sites during the dry season.

5.3 Diatom Species Composition

A total of 104 diatom taxa representing 30 genera were collected during cycles 4 and 5 (Table 5.1). Diatoms with alphanumeric designations (18 taxa) could not be identified after extensive searches of relevant literature. Representative specimens of each taxon were archived on permanent slides in our collection, but for troublesome taxa, we also digitally photographed representative specimens and collected data on taxonomically significant morphometric characters. This helped to maintain taxonomic consistency and will support future plans for a more rigorous taxonomic analysis. Another 21 taxa are listed as "unidentifiable" because they were represented by poor specimens that precluded accurate taxonomic designation. The most common and widespread taxa were *Brachysira neoexilis, Brachysira neoexilis* var. 02*, Encyonema* ftsp02*, Encyonema evergladianum, Fragilaria synegrotesca, Mastogloia smithii, Navicula radiosafallax, Nitzschia palea* var. *debilis* and *Nitzschia serpentiraphe*. These include commonly reported periphyton mat species (Browder et al. 1982, Swift & Nicholas 1987,

Raschke 1993, McCormick & O'Dell 1996), although a variety of nomenclatural techniques have been applied in Everglades literature, so it is difficult to validate numerous suspected synonyms. At the generic level the flora is typical of nutrient poor, hardwater, shallow systems, and includes mostly benthic, rather than planktonic taxa. At the specific level, several of the dominant species have been considered endemic, and the flora includes an abundance of taxa restricted to the tropical and subtropical environments (Slate 1991). To determine diatom distribution patterns and environmental correlates we took both an assemblage and species-based approach.

5.4 Environmental Associations of Diatom Assemblages

We used the Bray-Curtis dissimilarity metric (D) to measure differences among samples based on their diatom assemblages. Relationships among sites were then visualized in one dimension using hierarchical, farthest-neighbor clustering of the dissimilarity matrix. Data from cycles 4 and 5 were analyzed separately to determine temporal consistencies in compositional trends, and species abundances were relativized to totals prior to analysis. Five clusters of related sites (within-cluster D #0.50) could be identified from cluster dendrograms for each cycle (Figures 5.3, 5.4). Diatom taxa that significantly influenced site assignment to the 5 clusters were identified using Dufrene & Legendre's (1997) Indicator Species Analysis and are marked in Table 5.1.

To determine the type and extent of environmental influence on the 5 diatom assemblage clusters, means of each environmental parameter were calculated among samples in each cluster and compared to the other 4 clusters using pairwise post-hoc contrasts (adjusting probabilities for multiple comparisons, $P < 0.01$). Variables that differed significantly between one or more clusters are shown in Tables 5.2 and 5.3. Two clusters differed in mean latitude and longitude in both cycles, although the subdivision designations showed little pattern in relation to the cluster dendrograms. Certain ion measurements, including pH, conductivity, Cl, F, S^{2-} and SO_4 differed among species clusters, particularly during the wet season (Cycle 5). These parameters varied greatly across the sites sampled for periphyton and, given their influence on cellular processes, it is not surprising that species would assort along these gradients. Effects of water depth and associated parameters including soil depth, mineral content, bulk density, and hydroperiod, were more readily detected in dry than wet season samples. Diatom response to soil TP (and

correlated alkaline phosphatase activity) was also strongest during the dry season. During the wet season, nutrient (TP, TN) effects were only detected in water column measurements. Two diatom clusters also differed with respect to methyl mercury concentrated in periphyton tissues during the wet season. These data have provided the first means of identifying effects of multiple environmental parameters on diatom species composition. They will be instrumental in the development of calibration models for predicting compositional change given certain scenarios of environmental modification.

5.5 Indicator Species

Whereas multi-species assemblage data often provides a more precise measure of environmental conditions than a single-species approach, trends in the abundances of select species can be informative if those species are easy to find and are particularly sensitive to a critical environmental parameter. We classified diatoms collected in this survey as environmental indicators if they (1) were present in more than 20 % of the sites, (2) had a mean relative abundance greater than 0.5%, and (3) were significantly correlated with one or more environmental parameters. Eleven taxa met these criteria; photographs of representative specimens and relationships to influential environmental parameters are shown in Figures 5.5 to 5.15.

Some of the 11 taxa were sensitive to a number of environmental parameters while others responded to only one or two. Six taxa showed geographic pattern in their relative abundances. *Encyonema evergladianum*, *Encyonema* egsp01, and *Navicula cryptotenella* were more abundant at western sites, *Encyonema* ftsp02 and *Mastogloia smithii* were more abundant in the north and *Nitzschia serpentiraphe* to the south. These geographic patterns are probably correlated with underlying E-W and N-S environmental gradients.

Six taxa responded strongly to pH. *Fragilaria synegrotesca*, *Encyonema* ftsp02, *Brachysira neoexilis*, and *Nitzschia serpentiraphe* were most abundant at sites with pH >7.5, while *Encyonema silesiacum* var. *elegans* and E. *microcephala* indicate sites with lower pH (<7.5). Similarly, *Brachysira neoexilis*, *Nitzschia serpentiraphe*, *Navicula cryptotenella* and *Encyonema* egsp01 were abundant at low conductivity, low chloride sites while *Mastogloia smithii*, abundant everywhere, attained highest abundances at the highest conductivity sites. In general, conductivity and pH are lowest in WCA-1 and highest near canals where limerock has

been recently exposed. Chloride gradients often parallel conductivity gradients, being highest near canals and in areas of the northern Everglades that receive seepage from the marine aquifer. A gradient analysis would likely reveal predictable assortment of these species according to distance from these ion sources.

Eight taxa can be considered indicators of water depth, and associated parameters such as soil depth, mineral content, and hydroperiod. *Encyonema* ftsp02 and *Encyonema silesiacum* var. *elegans* were more common in deep sites and were infrequently encountered at sites that dry regularly. *Nitzschia palea* var. *debilis*, *Encyonema microcephala*, *Nitzschia serpentiraphe* and *Encyonema* egsp01 were more common in Taylor Slough and in other shallow areas that dry regularly. *Fragilaria synegrotesca* was more abundant in sites with high mineral content, yet the distribution of this taxon in Shark River Slough suggests that it prefers deeper water of longer hydroperiod than other Everglades taxa (Gottleib, unpubl. data). Together, changes in the abundances of these taxa might be used to indicate the ecological effectiveness of water level manipulations. Further studies to define the extent and mechanisms of these effects are warranted, because diatom response to hydroperiod is poorly understood, particularly for hardwater wetland assemblages (Gaiser et al. 1998).

In contrast to other surveys in the Everglades, we found relatively few strong taxonomic responses to nutrient concentrations. This is probably because sites that have been highly enriched in nutrients for a long time tend to have reduced periphyton communities, and were therefore excluded from the periphyton survey. Only 7 periphyton sampling sites had water column TP concentrations in excess of 20 ppb, and most were below 10 ppb, a level considered ambient for the native Everglades. Two species, *E. silesiacum* and *E. microcephala*, were found in greater abundance in relatively more enriched sites, while *Brachysira neoexilis* and *Nitzschia serpentiraphe* were correspondingly rare at these sites. Most of the species that can be considered good indicators of nutrient enrichment were not encountered in this study, but their nutrient optima have been well defined elsewhere (McCormick & O'Dell 1996).

5.6 Conclusions and Recommendations

This study has shown that diatom community analysis can be a useful tool in environmental monitoring and should continue to be integrated into Everglades assessment protocols for the following reasons:

1. Diatoms are ubiquitous in the Everglades yet species have non-random distributions. Baseline distribution data is now available for use in detecting environmental change.

2. Diatoms are sensitive to environmental variation. Assemblage and species responses to spatial variation in ion content, nutrient availability and hydroperiod have been identified. Temporal models can be built from these spatially explicit data to predict community change under different management scenarios with a measurable degree of accuracy.

3. Diatoms respond quickly to environmental change. Unlike many other biotic indicators, changes in diatom assemblage composition can happen over very short time scales (days to weeks) and, therefore, can provide sensitive early warning signals of impending ecosystem change.

4. The taxonomic reference base generated from this survey will increase efficiency of future diatom inventories. Many surveys exclude diatom analyses because of perceived technical difficulties in collection and assessment. While this may have been the case at one time in the Everglades, currently available taxonomic databases should substantially reduce allocation of time and resources to identification. There are fewer species of diatoms in the Everglades than vascular plants so their identification is no more of a task than more commonly employed vegetation monitoring. Given currently available reference materials, lack of technical expertise in this field is no longer a viable argument against diatom assessments, especially given their potential in environmental monitoring.

Because this is the first broad survey to incorporate diatom assessments, the data provide several suggestions for future monitoring efforts, including:

1. Future assessments of diatom community composition should be accompanied by measurements of TP sequestered in the periphyton mat. Difficulties in this study in detecting species responses to nutrient gradients are, in part, due to the lack of a measure of nutrient availability that integrates the appropriate time scale. Diatom communities do not strongly reflect local water column nutrient concentrations because of luxury uptake of nutrients and because of the variability inherent in water column concentrations (Gaiser et al., submitted ms). Even less likely is a response to soil nutrients, which are often recalcitrant and reflect a much longer period of accumulation than the life histories of these organisms. Mat tissue phosphorus concentration is perhaps the most reliable measure of nutrient availability on a weekly or monthly time scale (Gaiser et al., submitted ms).

2. Further studies of diatom response to water depth and hydroperiod are warranted. This and other concomitant studies (Gaiser et. al., submitted ms) are the first to show a strong response of diatom assemblages to hydroperiod, a critical

environmental parameter that is a fundamental component of most restoration programs. Certain diatoms may provide a very accurate assessment of the success of hydrologic restoration. This study suggests hydrologically sensitive species that should be the target of more explicit survey or experimental studies. These efforts are especially critical because of the lack of general knowledge of the response of wetland diatom assemblages to water depth change.

3. Interpretations of environmental change based on diatom assessments must not ignore the fact that a given diatom assemblage reflects a suite of correlated environmental parameters. This study shows that diatoms respond very strongly to pH and conductivity, two parameters that are often correlated with nutrient availability in this system. Experiments that control for the effects of these environmental correlates could clarify interpretations of environmental change based on descriptive data from diatom surveys.

4. In the future, collections should include scrapings of periphyton from any available surface at all sites. Periphyton tends only to be abundant in unimpacted areas of the Everglades, because of the detrimental effects of excess nutrients on mat integrity. However, reduced communities exist in enriched areas (ie., on stems of cattail, in benthic muds) that can contain a very different assemblage of species than neighboring unimpacted areas. Samples from these diatom communities can provide extreme values for developing more generally applicable diatom-based nutrient indexes. Also, because fossil diatoms are retained in wetland sediments, knowledge of species responses along the full length of existing environmental gradients are necessary for retrospective analyses of change.

Table 5.1. Diatom taxa collected during 1999 REMAP sampling and their associated mean relative abundance (percent), frequency of occurrence (of 153 sites) and cluster group affiliation from sample cycles 4 and 5. Diatoms having a significant ($p<0.05$) cluster affiliation are designated with an *.

Taxon	Relative Abundance	Frequency	Cycle 4 Cluster	Cycle 5 Cluster
Achnanthidium lanceolata Bréb.	0.01	1		
Achnanthidium minutissima Kütz.	0.01	3	3	
Achnanthidium minutissima var. *scotica* (Carter) L-Bert.	0.44	65	3*	3
Amphora ovalis (Kütz.) Kütz.	0.01	1		3
Amphora sulcata A. Schmidt	0.96	29	1	3*
Amphora veneta Kütz.	0.01	1		2
Aulacoseira islandica O. Müll. Simon.	0.01	1		
Brachysira brebissonii Ross	0.34	20	3	4
Brachysira neoexilis L.-Bert.	3.14	137	4*	4*
Brachysira neoexilis L.-Bert. var. 01	1.36	96	4	4
Brachysira serians (Bréb.) Round & Mann	0.03	4		3
Caloneis bacillum (Grun.) Cl.	0.02	4		1
Caloneis macedonica Hust.	0.01	1		1
Caponea caribbea Podz.	0.02	9	2	3
Craticula cuspidata (Kütz.) Mann	0.03	3		1
Cyclotella meneghiniana Kütz.	0.12	42	4	4
Desmogonium rabenhorst var. *elongatum* Patr.	0.01	1		4
Diploneis oblongella (Naeg.) Cl.-Eul.	0.21	36	2	3
Diploneis parma Cl.	1.59	95	1	4
Encyonema egsp01	0.74	24	5*	4*
Encyonema ftsp01	0.37	65	5	3
Encyonema ftsp02	4.47	144	5*	4
Encyonema sjsp03	0.41	50	2	2
Encyonema ftsp04	0.01	5	1	
Encyonema evergladianum Krammer	22.55	145	4*	5*
Encyonema microcephala Grun. *ex*. V.H.	1.20	97	4*	4*
Encyonema pusilla (Grun.) Cl.	0.02	1		
Encyonema silesiacum (Bleisch *ex*. Rabh.) Mann	0.01	6		4
Encyonema silesiacum var. *elegans* (Bleisch) Mann	0.68	34	5*	5*
Eunotia flexuosa (Bréb.) Kütz.	0.12	39	5*	5
Eunotia incisa Greg. var. 01	0.14	21	5	4
Eunotia monodon Ehr. var. 01	0.04	8	4	3
Eunotia naegeli Migula	0.05	10		1
Fragilaria ctsp01	0.02	3		4
Fragilaria ctsp02	0.01	4	3*	5*
Fragilaria nanana L.-Bert.	0.18	31	4	1
Fragilaria synegrotesca L.-Bert.	11.50	149	3*	1*
Frustulia rhomboides var. *crassinervia* (Bréb.) Ross	0.34	27	5*	4

Table 5.1 continued

Taxon	Relative Abundance	Frequency	Cycle 4 Cluster	Cycle 5 Cluster
Gomphonema acuminatum Ehr.	0.03	6	5*	4
Gomphonema affine Kütz. var. 01	0.44	86	5*	4
Gomphonema clavatum Ehr.	0.20	34	4	5
Gomphonema gracile Ehr.	0.01	1		
Gomphonema parvulum (Kütz.) Kütz.	0.11	4		
Gomphonema egsp01	0.02	4		1
Gomphonema sdsp01	0.18	31	4	5
Hantzschia amphioxys (Ehr.) Grun.	0.01	4	4	1
Luticola mutica (Kütz.) Mann	0.01	3		3
Mastogloia lanceolata Thwaites *ex*. W. Sm.	0.02	8	1*	
Mastogloia smithii Thwaites	34.03	149	1*	2*
Navicula brasiliana Cl. var. 01	0.01	1		
Navicula cryptocephala var. *exilis* (Kütz.) Grun.	0.01	3		4
Navicula cryptotenella L.-Bert.	0.75	76	5*	2
Navicula cutiformis Grun.	0.01	1	2	
Navicula cryptolyra Brock.	0.01	1		
Navicula digitoradiata (Greg.) Ralfs	0.01	4	4	3
Navicula radiosafallax L.-Bert.	0.78	89	5	5
Navicula subtilissima Cl.	0.25	35	4*	4
Neidium ampliatum (Ehr.) Krammer	0.01	3	4	2
Neidium floridanum Reim.	0.01	1		
Nitzschia amphibiodes Hust.	0.03	5		5
Nitzschia amphibia Grun.	0.24	37	3	5
Nitzschia amphibia var. *elongata* Grun.	0.71	19	1	2
Nitzschia intermedia Hantz.	0.01	3		2
Nitzschia lacunarum Hust.	0.01	3	3	3
Nitzschia nana Grun.	0.09	7		1
Nitzschia palea (Kütz.) W. Sm.	0.46	26	2	2
Nitzschia palea var. *debilis* (Kütz.) Grun.	3.17	143	4*	3
Nitzschia scalaris (Ehr.) W. Sm.	0.01	1		
Nitzschia semirobusta L.-Bert.	0.01	2		2
Nitzschia serpentiraphe L.-Bert.	2.11	122	2*	4
Nitzschia serpentiraphe L.-Bert. var. 01	0.32	26	1	2
Pinnularia acrosphaeria Rabh.	0.02	4		
Pinnularia gibba Ehr. var. 01	0.08	15		1
Pinnularia maior Boyer var. *pulchella*	0.02	9	1	
Pinnularia microstauron (Ehr.) Cl.	0.33	14	1	3
Pinnularia rupestris Hantz. var. 01	0.03	5		
Pinnularia streptoraphe Cl. var. 01	0.02	5		2
Pinnularia viridis (Nitz.) Ehr.	0.01	1		
Rhopalodia gibba (Ehr.) O. Müll.	0.13	10	1*	2
Rhopolodia musculus (Kütz.) O. Müll.	0.01	1		
Sellaphora laevissima (Kütz.) Round	0.24	66	4	3

Table 5.1 continued

Taxon	Relative Abundance	Frequency	Cycle 4 Cluster	Cycle 5 Cluster
Sellaphora pupula (Kütz.) Round	0.33	5	2	4
Stauroneis anceps var. *subrostrata* Gaiser & Johansen	0.01	1		
Stauroneis phoenicentron (Nitzsch.) Ehr.	0.01	6	4	3
Stenopterobia curvula (W. Sm.) Krammer	0.01	1		4
Achnanthes unidentifiable	0.01	1		
Amphora unidentifiable	0.01	4		
Anomoneis unidentifiable	0.01	1		
Aulacoseira unidentifiable	0.01	3		
Brachysira unidentifiable	0.13	11		
Caloneis unidentifiable	0.01	2		
Cyclotella unidentifiable	0.08	21		
Diploneis unidentifiable	0.11	29		
Encyonema unidentifiable	1.79	114		
Eunotia unidentifiable	0.14	27		
Fragilaria unidentifiable	0.01	3		
Gomphonema unidentifiable	0.57	79		
Hantzschia unidentifiable	0.01	1		
Navicula unidentifiable	0.10	22		
Nitzschia unidentifiable	0.12	13		
Pinnularia unidentifiable	0.40	35		
Rhopalodia unidentifiable	0.01	1		
Stauroneis unidentifiable	0.01	1		
Stephanodiscus unidentifiable	0.01	1		
Unidentifiable valve	0.03	8		
Unidentifiable girdle	0.05	5		

Table 5.2. Means of environmental parameters for sites with diatom assemblage clusters 1-5 identified from Bray-Curtis, farthest-neighbor distance analysis of relative abundances of diatom taxa collected during Cycle 4. Only environmental parameters that differed significantly among clusters are shown. Highest and lowest mean values among the 5 clusters are shown in boldface type for each parameter. Numbers in superscript designate clusters with significantly higher or lower values than the given mean.

Cycle 4

Diatom Cluster		1	2	3	4	5
Latitude	Mean	**26.12** [2,3,4,5]	25.81 [1]	25.74 [1]	**25.60** [1]	25.81 [1]
(Decimal)	SD	0.14	0.29	0.26	0.34	0.20
Longitude	Mean	**-80.48** [2,3,4,5]	-80.62 [1,5]	-80.65 [1]	-80.72 [1]	**-80.77** [1]
(Decimal)	SD	0.07	0.15	0.16	0.11	0.05
pH	Mean	7.33	7.32	**7.48** [4,5]	**7.12** [3]	7.15 [3]
	SD	0.22	0.37	0.26	0.01	0.22
Conductivity	Mean	687.20 [5]	632.88 [5]	**700.30** [5]	480.50	**343.75** [1,2,3,4]
(FS)	SD	138.79	214.71	118.80	47.38	86.99
Water Depth	Mean	0.21	0.11 [3]	**0.24** [2,4]	**0.08** [3]	0.19
(m)	SD	0.15	0.15	0.13	0.14	0.12
Soil Depth	Mean	**1.08** [3]	0.70	**0.56** [1]	0.70	0.76
(m)	SD	0.42	0.48	0.36	0.39	0.95
Cl (water)	Mean	74.60 [4,5]	68.33	**90.55** [4,5]	**27.00** [1,3]	27.89 [1,3]
(mg l^{-1})	SD	8.23	48.70	35.22	24.02	13.14
TP (soil)	Mean	197.62	216.86	**238.32** [4]	**148.42** [3]	256.89
(Fg g^{-1})	SD	185.70	114.32	64.55	87.30	148.45
AP (floc)	Mean	79.10	**82.45** [3]	**18.71** [2,5]	34.98	57.55 [5]
(Fmole g^{-1})	SD	92.06	62.64	21.21	24.99	35.24
Mineral content	Mean	**56.49** [2,3]	**19.74** [1]	26.72 [1]	30.36	24.41
(% in floc)	SD	29.20	18.60	12.81	38.34	22.87
Bulk density	Mean	**0.32** [2,3]	**0.12** [1]	0.15 [1]	0.20	0.15
(g cm^{-3})	SD	0.15	0.07	0.04	0.19	0.09
Hydroperiod	Mean	**3.29** [5]	3.92 [5]	4.17 [5]	3.57 [5]	**5.90** [1,2,3,4]
(dry season)	SD	1.38	2.17	1.53	1.90	1.29

Table 5.3. Means of environmental parameters for sites with diatom assemblage clusters 1-5 identified from Bray-Curtis, farthest-neighbor distance analysis of relative abundances of diatom taxa collected during Cycle 5. Only environmental parameters that differed significantly among clusters are shown. Highest and lowest mean values among the 5 clusters are shown in boldface type for each parameter. Numbers in superscript designate clusters with significantly higher or lower values than the given mean.

Cycle 5

Diatom Cluster		1	2	3	4	5
Latitude	Mean	25.89	**26.03** ^{3,4}	25.71 ^{2}	**25.62** ^{2}	25.80
(Decimal)	SD	0.37	0.26	0.23	0.31	0.41
Longitude	Mean	-80.62	-80.59	-80.67	**-80.67** ^{5}	**-80.53** ^{4}
(Decimal)	SD	0.15	0.15	0.18	0.12	0.03
pH	Mean	7.74	7.69	7.73	**7.85** ^{5}	**7.52** ^{4}
	SD	0.45	0.27	0.29	0.32	0.34
Conductivity	Mean	492.38	**548.50** ^{3,4}	341.00 ^{2}	**297.64** ^{2}	452.00
(FS)	SD	269.40	231.66	128.55	77.18	194.13
Water Depth	Mean	**0.80** ^{3}	0.75	**0.55** ^{1}	0.57	0.74
(m)	SD	0.23	0.24	0.20	0.43	0.43
SO_4 (water)	Mean	14.10	**19.68** ^{3,4}	**2.50** ^{2}	3.03 ^{2}	7.26
(mg l^{-1})	SD	21.68	16.47	2.57	6.05	10.25
TOC (water)	Mean	20.11 ^{3,4}	**22.57** ^{3,4}	**13.04** ^{1,2}	11.36 ^{1,2}	14.88
(mg l^{-1})	SD	9.87	7.83	4.16	4.64	5.35
TP (water)	Mean	7.09 ^{3}	**7.43** ^{3}	**5.11** ^{1,2}	5.48	5.52
(Fg l^{-1})	SD	3.14	3.16	0.77	1.18	0.70
TN (water)	Mean	0.80 ^{3,4}	**0.96** ^{3,4}	0.65 ^{1,2}	**0.48** ^{1,2}	0.49
(mg l^{-1})	SD	0.31	0.34	0.27	0.18	0.19
Cl (water)	Mean	47.88	**52.88** ^{3,4}	26.34 ^{2}	**18.35** ^{2}	33.33
(mg l^{-1})	SD	41.03	24.00	17.63	8.67	20.79
F (water)	Mean	0.24	**0.29** ^{4}	0.17	0.10	**0.18** ^{2}
(mg l^{-1})	SD	0.18	0.17	0.16	0.05	0.13
H_2S (porewater)	Mean	0.67	0.51 ^{2}	**0.10** ^{2,5}	0.14	**1.17** ^{3}
(mg S2-l^{-1})	SD	1.54	0.62	0.06	0.22	1.82
MeHg (PUF)	Mean	2.78	**5.59** ^{3}	**1.97** ^{2}	3.66	2.47
(Fg kg^{-1})	SD	1.44	0.82	0.83	1.45	2.33

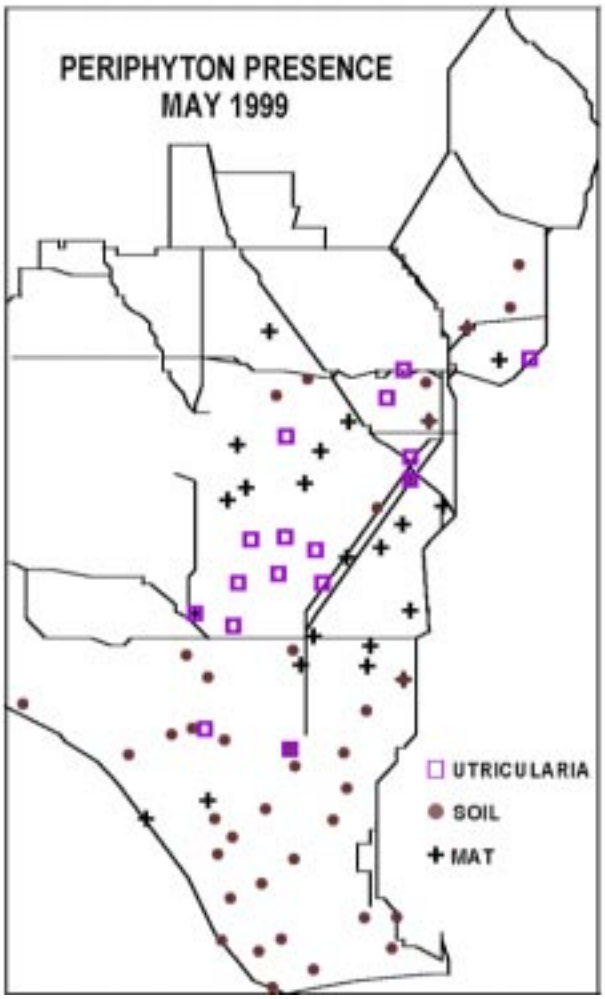


Figure 5.1. Distribution and substrate associations of periphyton during sample Cycle 4.

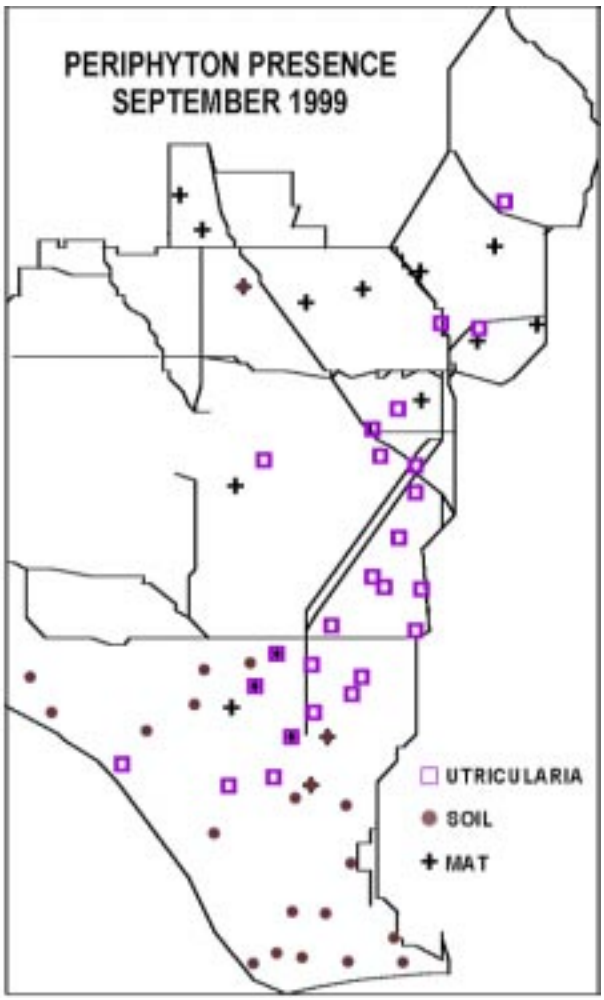


Figure 5.2. Distribution and substrate associations of periphyton during sample Cycle 5.

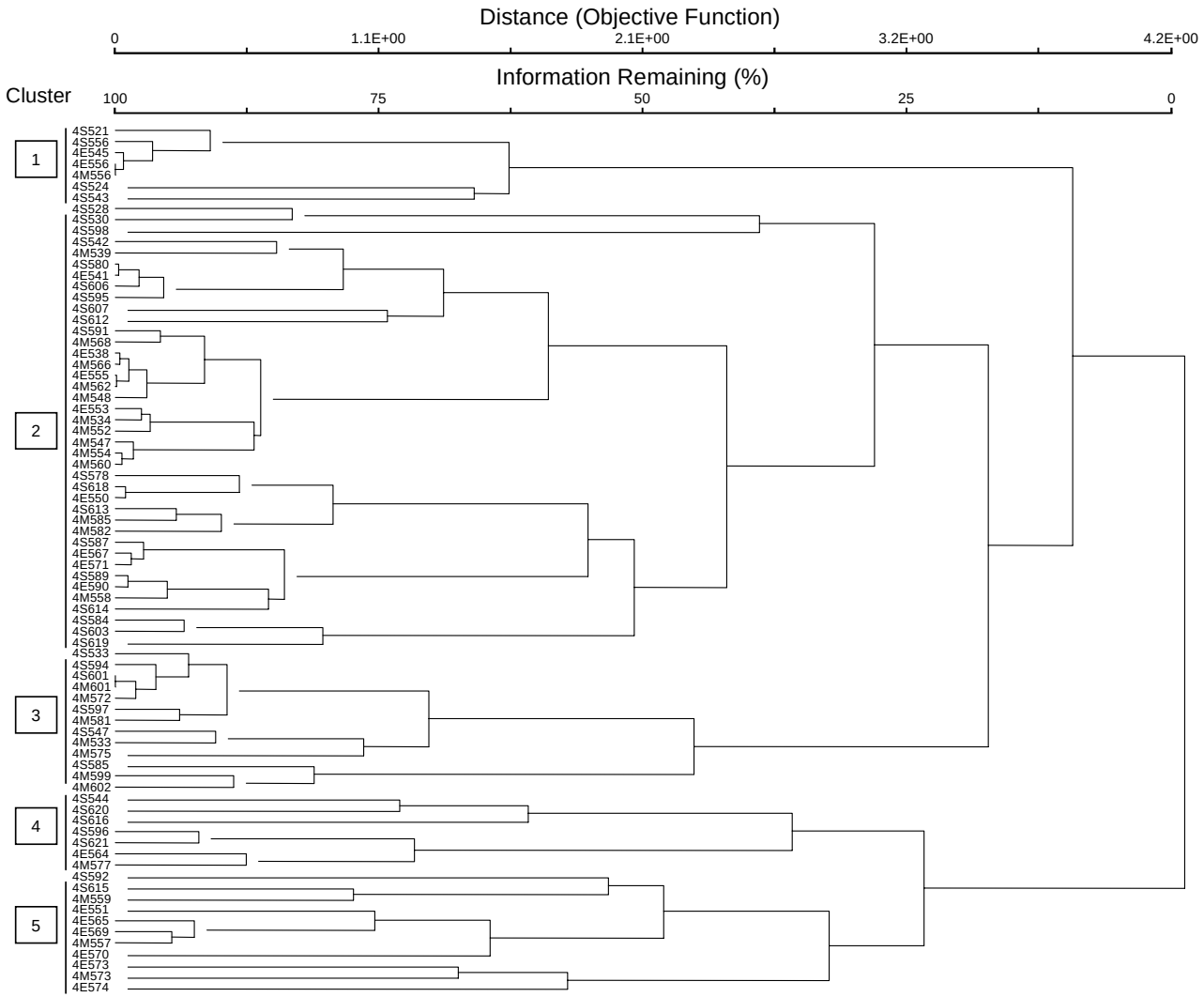


Figure 5.3. Cycle 4 cluster dendogram.

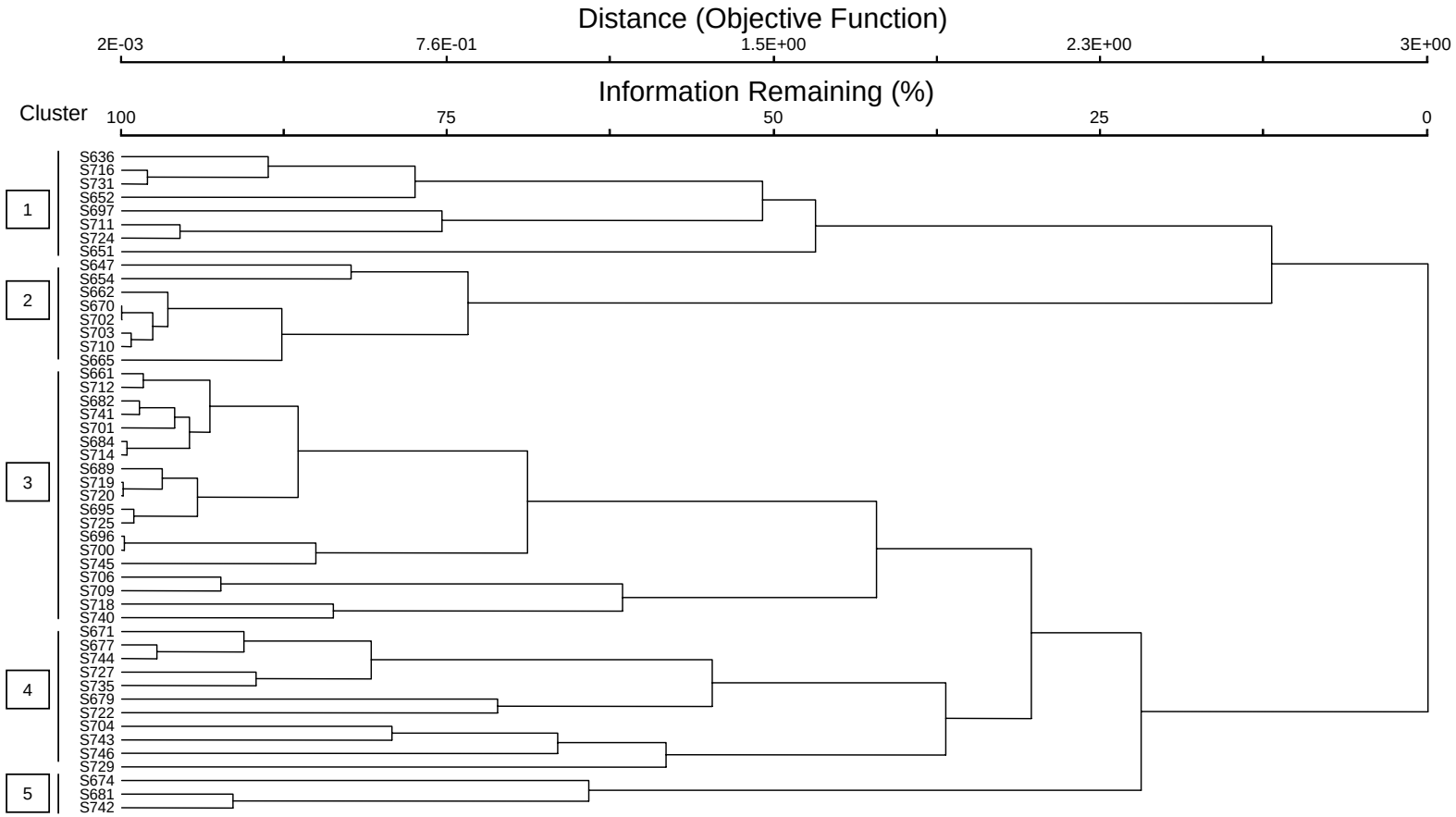


Figure 5.4. Cycle 5 cluster dendogram.

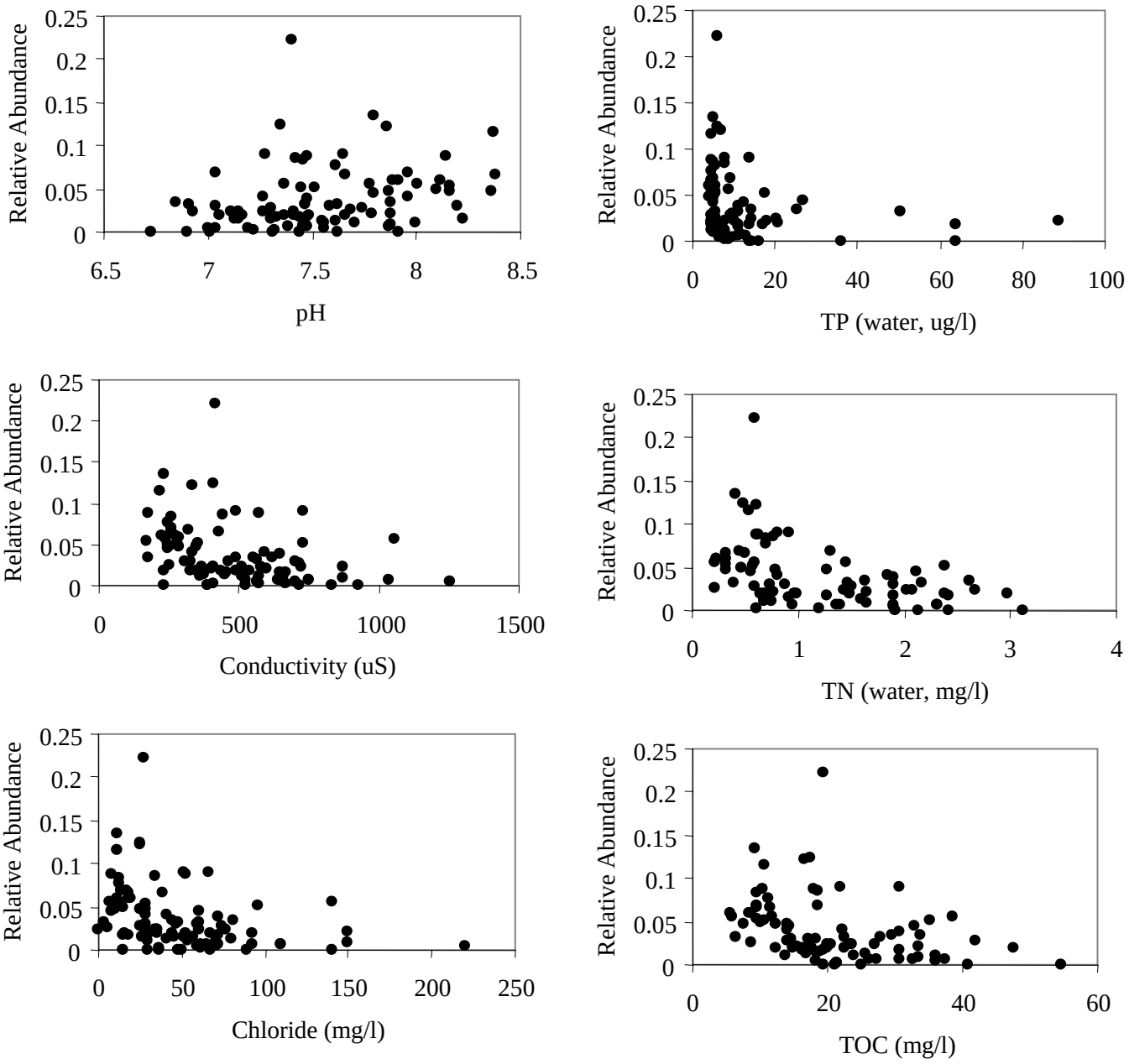
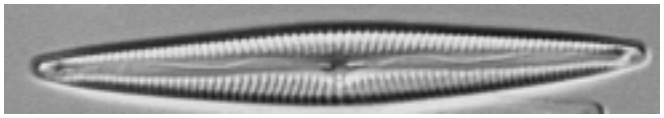


Figure 5.5. Relationships of *B. neoexilis* to influential environmental parameters.



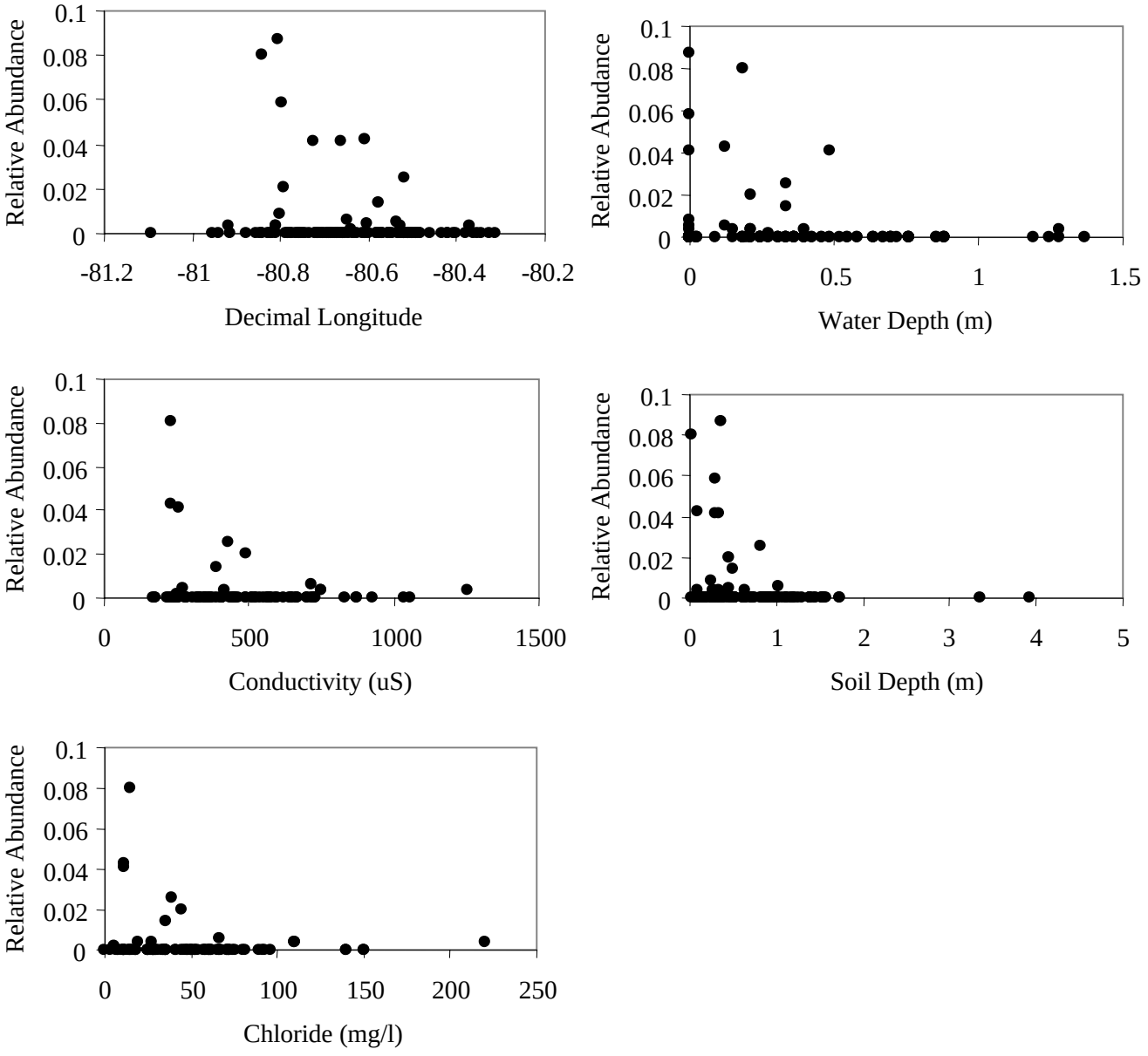


Figure 5.6. Relationships of *E. egsp01* to influential environmental parameters.

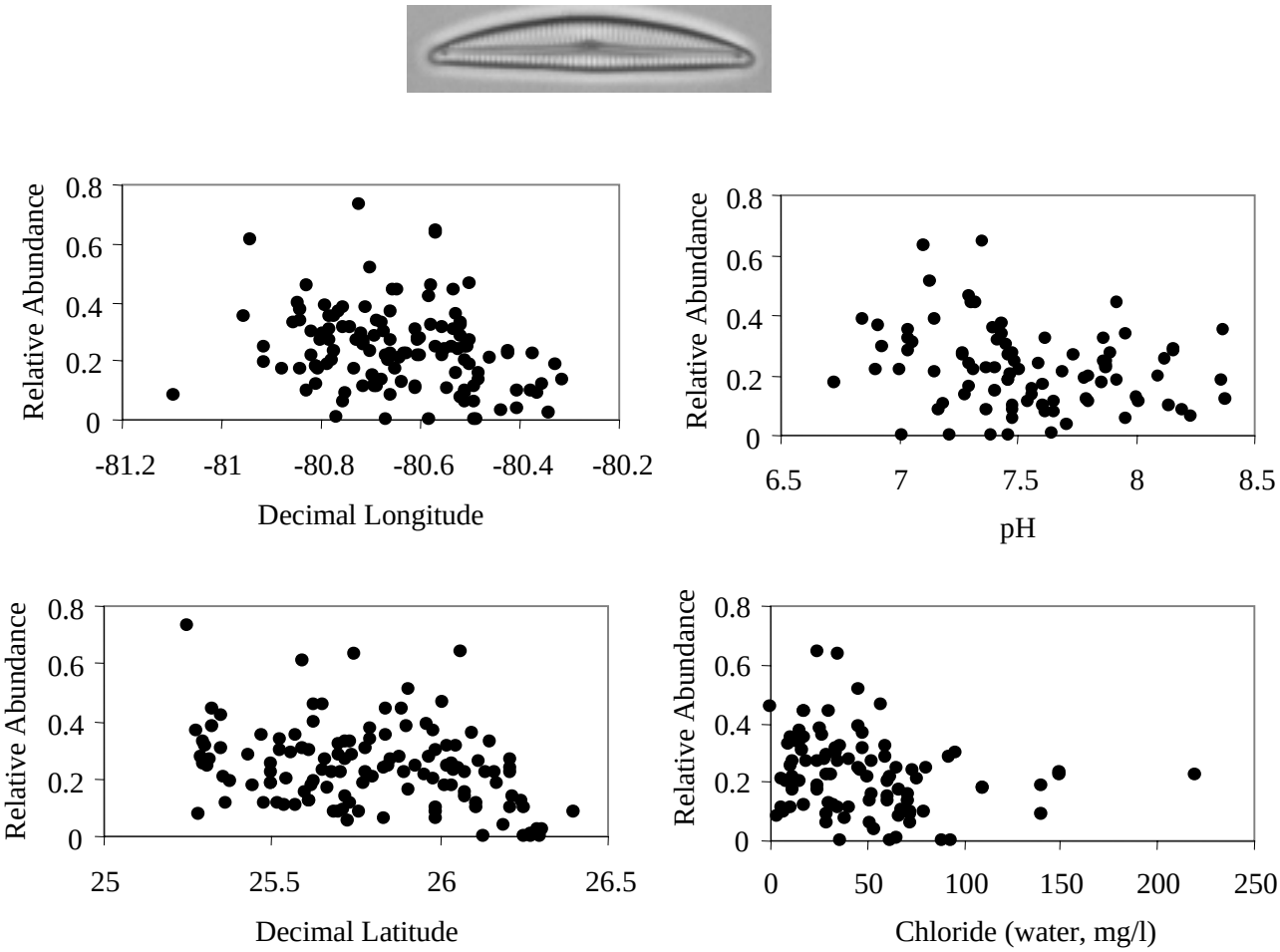


Figure 5.7. Relationships of *E. evergladianum* to influential environmental parameters.

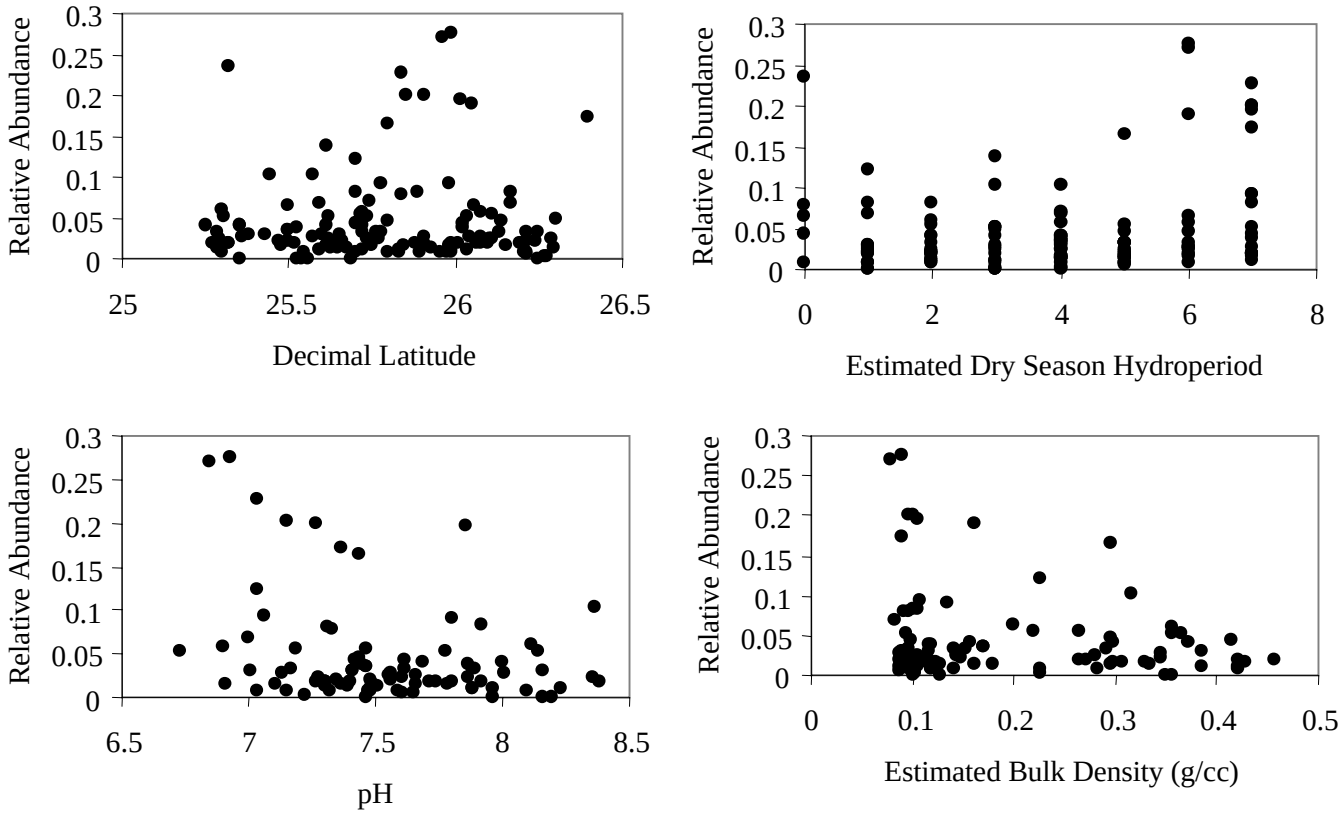
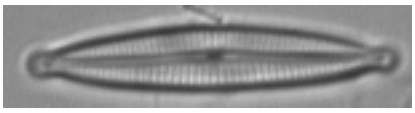
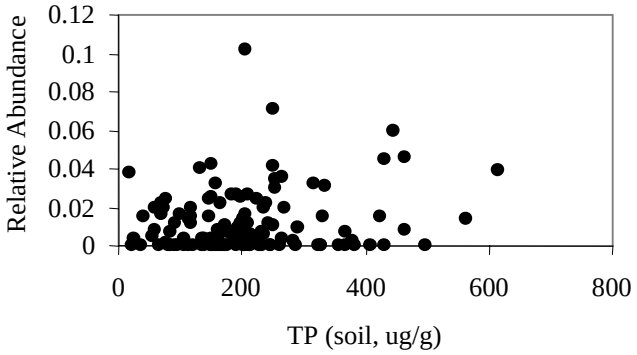


Figure 5.8. Relationships of *E. ftsp02* to influential environmental parameters.





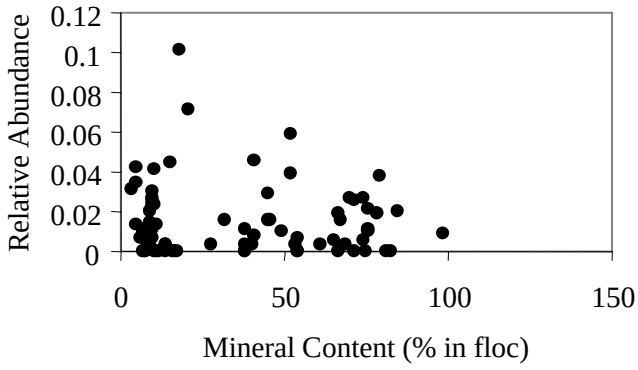
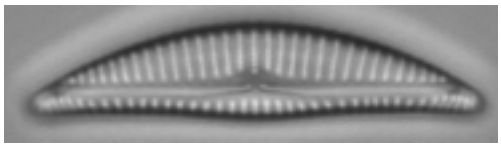
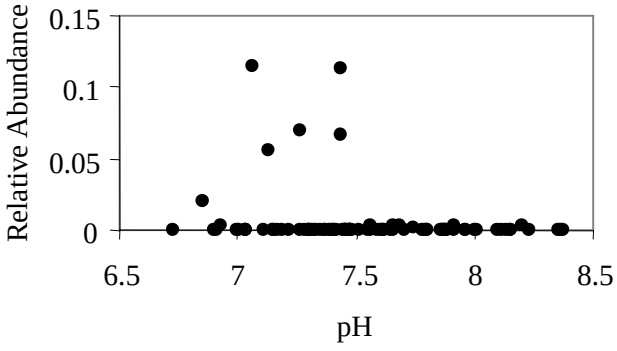
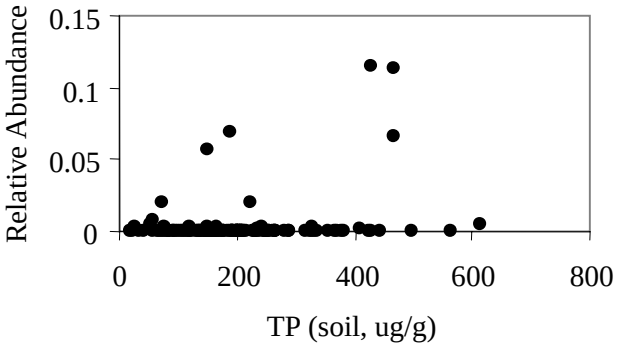
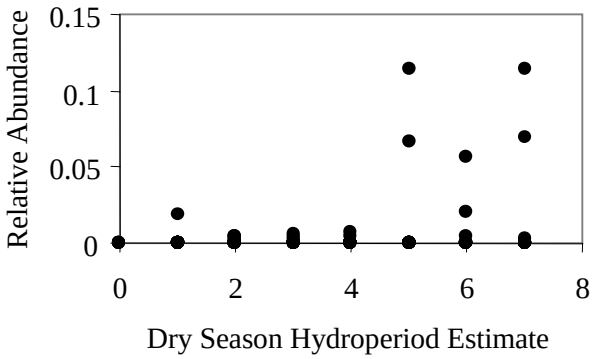


Figure 5.9. Relationships of *E. microcephala* to influential environmental parameters.









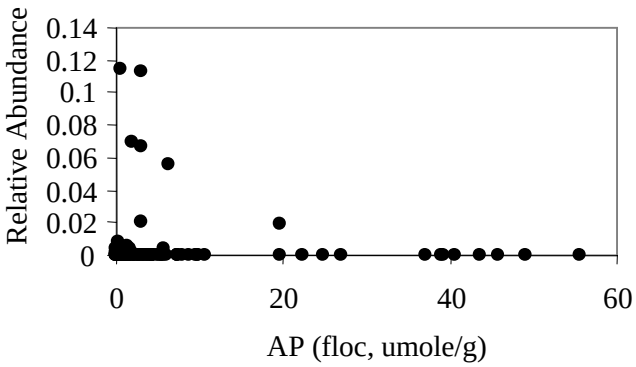
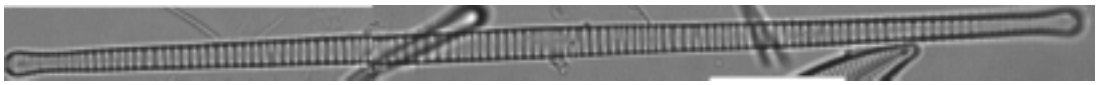
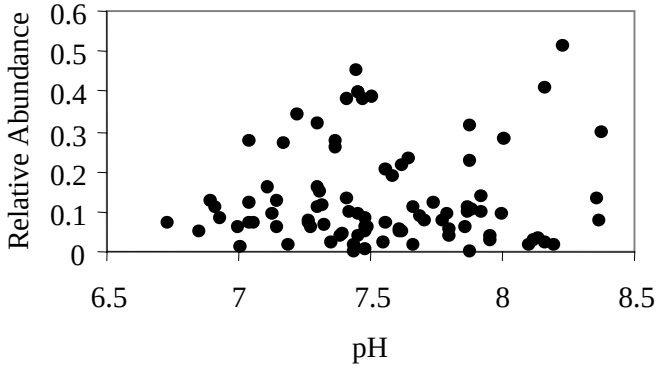
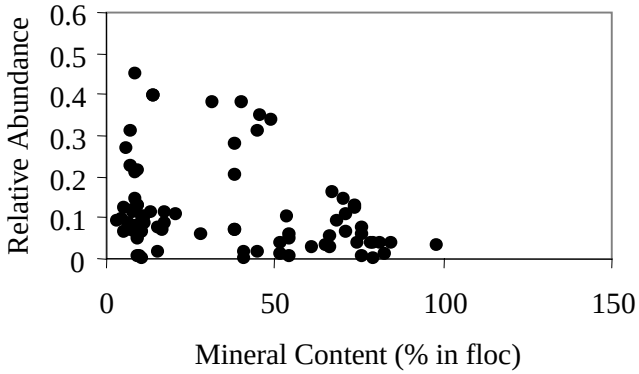


Figure 5.10. Relationships of *E. silesiacum* to influential environmental parameters.







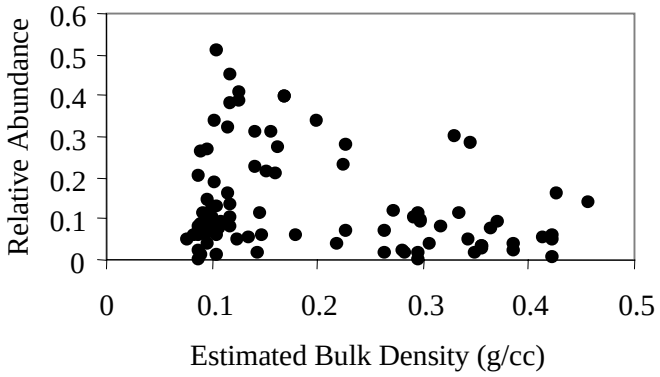
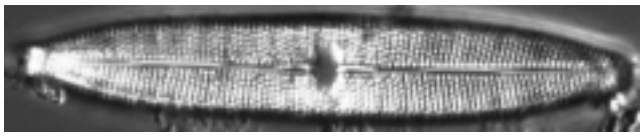
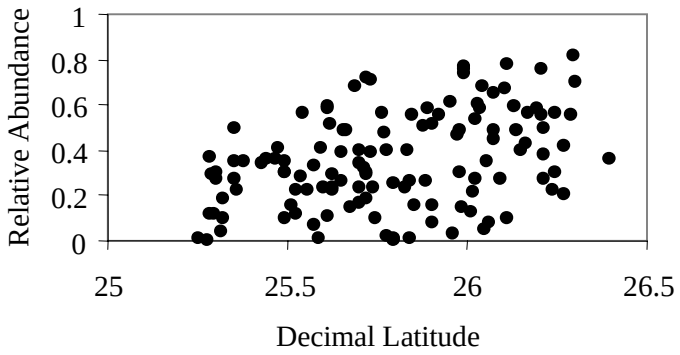
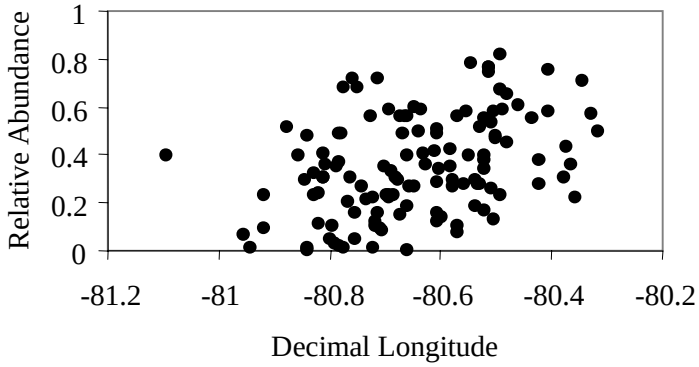


Figure 5.11. Relationships of *F. synegrotesca* to influential environmental parameters.







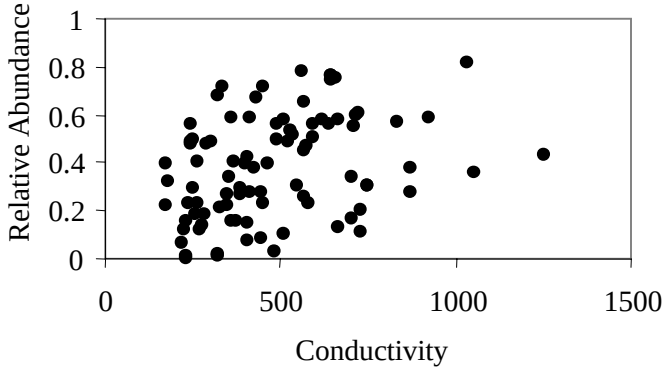


Figure 5.12. Relationships of *M. smithii* to influential environmental parameters.

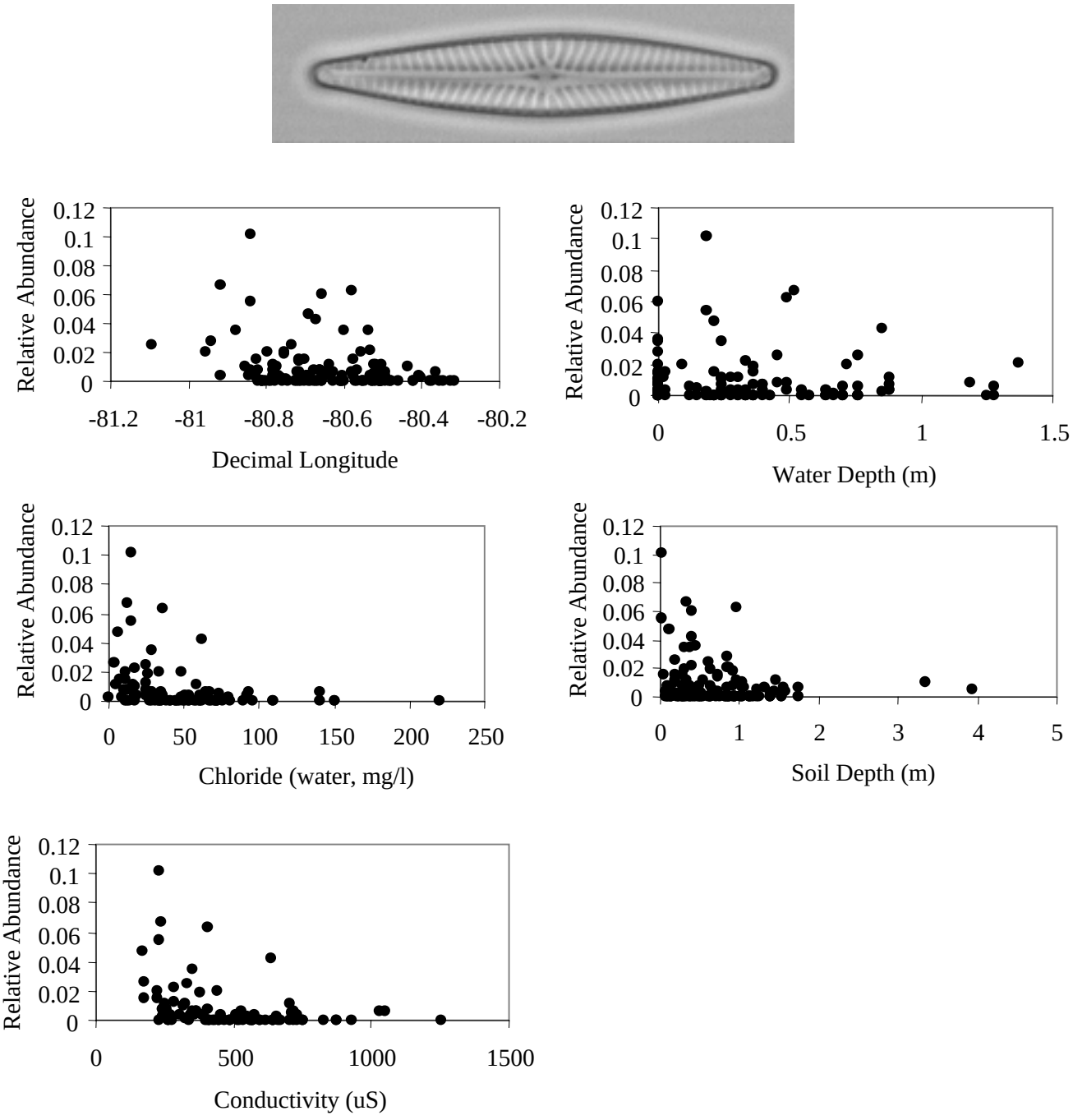
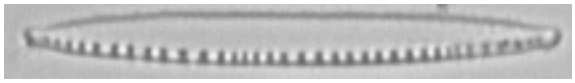
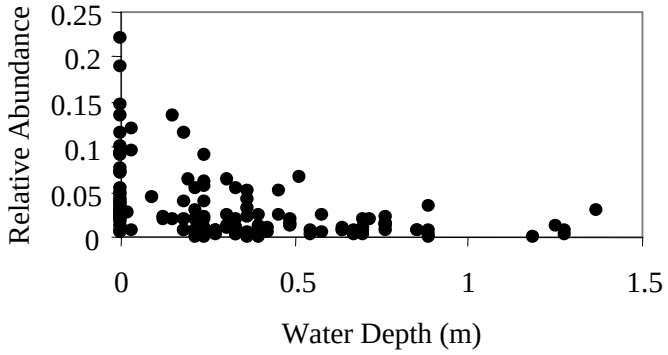


Figure 5.13. Relationships of *N. cryptotenella* to influential environmental parameters.





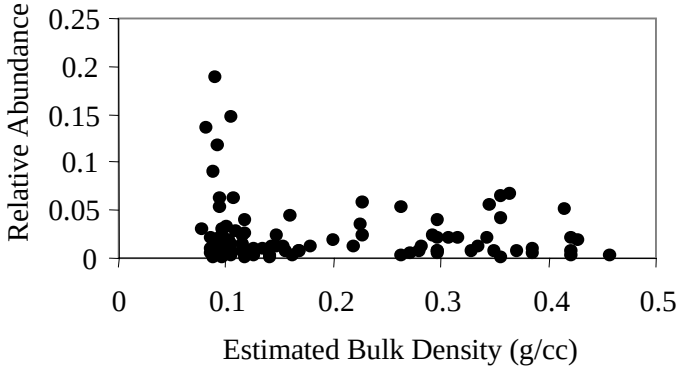
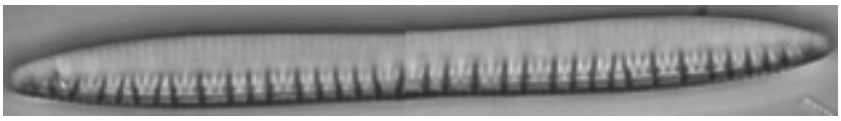
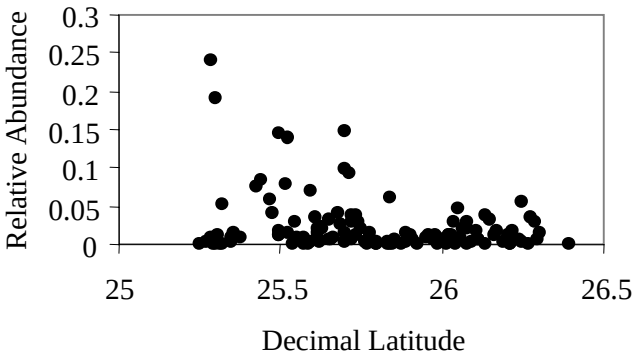
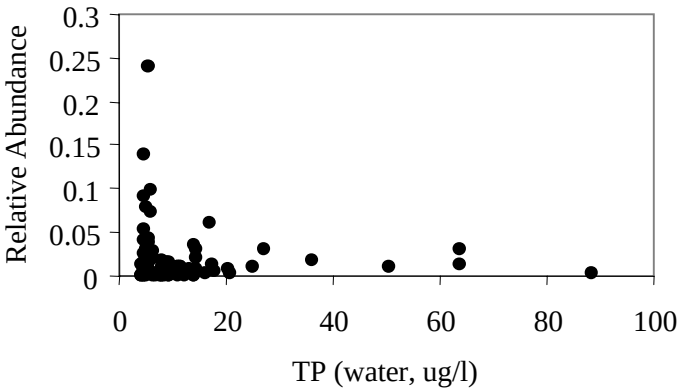
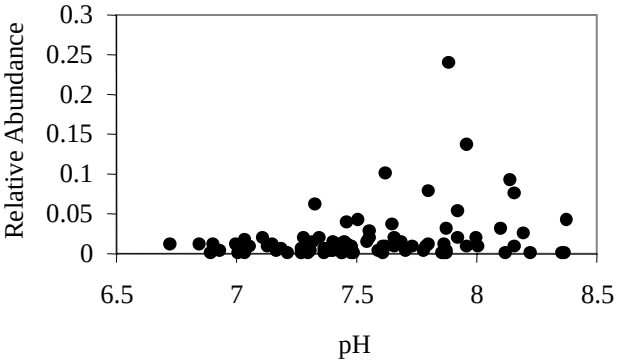


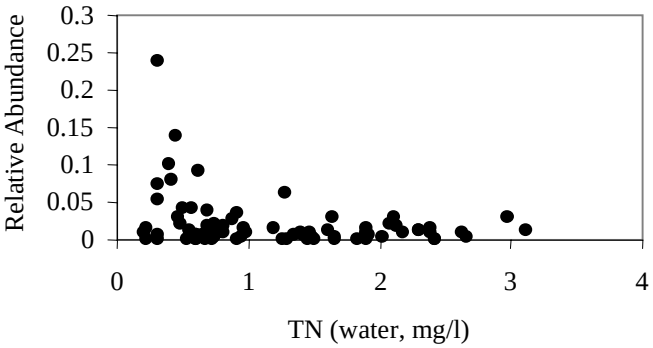
Figure 5.14. Relationships of *N. palea* to influential environmental parameters.

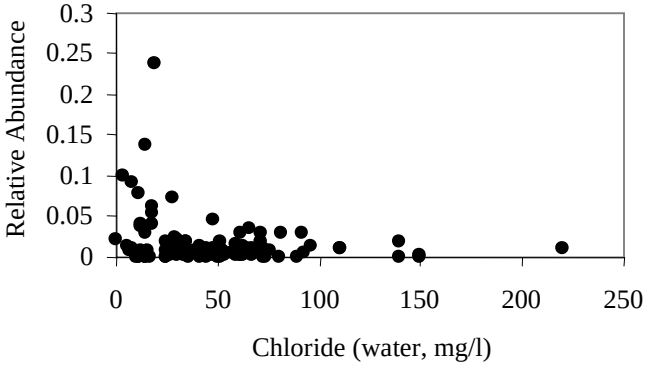


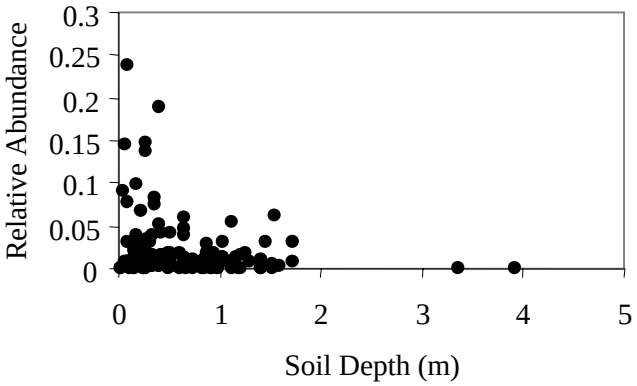












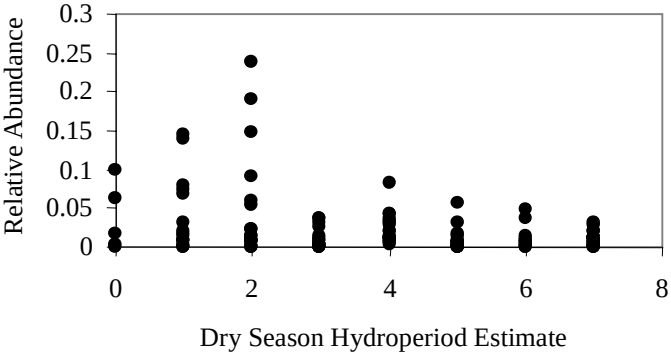


Figure 5.15. Relationships of *N. serpentiraphe* to influential environmental parameters.

6.0 LANDSCAPE PATTERNS

Understanding large-scale and landscape patterns is critical for managing the South Florida Everglades ecosystem to achieve restoration goals. Information from this Project can be used to describe the ecological conditions and patterns over this large 5,500 km^2 area. Historically, the South Florida Everglades ecosystem was one continuous marsh. Today dikes, levees, roadways, urban development and other landscape features alter water flow, habitat, nutrient loading and the corresponding ecological conditions. Some of the subareas created by these features are apparent, e.g., Loxahatchee National Wildlife Refuge, Water Conservation Areas 2 and 3, and Everglades National Park.

Alligator Alley (I-75) and Tamiami Trail (US Hwy 41) both bisect the South Florida ecosystem and create barriers to flow. During the Phase I Project, three subareas were identified based on these barriers and the patterns in water chemistry, soil constituents, and biotic mercury concentrations. These three subareas were north of Alligator Alley, between Alligator Alley and Tamiami Trail, and south of Tamiami Trail in Everglades National Park.

Seven subareas have been identified as being important for management in this Phase II Project. These seven subareas are: (1) Loxahatchee National Wildlife Refuge (Lox); (2) Water Conservation Area 2 (WCA2); (3) Water Conservation Area 3 North of Alligator Alley (WCA3-N); (4) the southeastern part of Water Conservation Area 3 (WCA3-SE); (5) the southwestern part of Water Conservation Area 3 (WCA3-SW); (6) Shark River Slough; and (7) Taylor Slough (Figure 1.1). The flow path and water quality patterns in Water Conservation Area 3 south of Alligator Alley are clearly demarcated into east and west patterns. In addition, the patterns in biotic mercury concentrations reflect these flow paths. The area south of Tamiami Trail is hydrologically divided with Shark River Slough being distinct from Taylor Slough. Because of these natural and artificial barriers to flow in the system, different landscape patterns develop throughout the system. These landscape patterns are discussed in this Chapter.

6.1 Water Regime

The South Florida Everglades is a hydrologically-driven ecosystem. In addition to precipitation, discharge through the structures and canal system affects the hydrologic regime.

6.1.1 Precipitation

Precipitation records for nine stations within and bordering the South Florida Everglades ecosystem were analyzed during Phase I to determine the relation of the Phase I years to the long term period of record (Figure 6.1). These records were extended through 1999 so the 1999 sampling year could be compared with both Phase I and the long-term period of record (Table 6.1). The total volume of precipitation during 1999 was similar to other years, but the distribution of the rainfall throughout the year was skewed even more than the norm. Typically, 80% of the precipitation in South Florida occurs during the summer wet season, from June through October. The 1999 dry season was quite dry with fires burning about 40% of the northern portion of WCA3. However, the 1999 wet season received precipitation volumes similar to 1995, which was a wet year.

6.1.2 Water Depth

The pattern in precipitation is reflected in the water depth distributions throughout the marsh in 1999. Water depth cumulative distributions for the Phase I and II seasons indicated that the 1999 dry season had the shallowest water depths for any of three years, while the 1999 wet season had some of the deepest water of any of the three years (Figure 6.2). The median water depths for the 1999 dry and wet seasons were 0.0 and 0.64 m, respectively. The range of hydrologic conditions captured during Phase I and II is relatively broad, and provides a solid baseline for determining whether future changes are due to alternative management practice or are within the expected range of hydrologic conditions (Figure 6.2).

The spatial variability in water depth that occurred within the different subareas is shown in Figures 6.3 to 6.5. The areas that were consistently wet during the dry season were in the central portion of WCA3 (Figure 6.3). Sampling occurred during April 1995 rather than May (Figure 6.5), so these water depths were not included in Figure 6.3 to maintain comparability. Lox and WCA2 had lower median water depths and greater variability in water depth than the central area of WCA3, but were wet during the dry season. WCA3-N, and Taylor Slough were essentially dry during May 1996 and 1999, with very shallow water depths (<0.02 m) in those areas that retained water. Shark River Slough did have areas that were dry during 1996 and 1999, and had a median water depth of about 0.05m.

There also were spatial differences in subarea water depths during the wet seasons in 1995, 1996, and 1999, but the entire marsh was wet (Figure 6.4). The greatest water depths again occurred in the central portion of WCA3 with significantly lower water depths in Shark River and Taylor Slough than in the areas north of Tamiami Trail. In addition, there was no significant difference in water depth distributions among the three years.

The temporal variability in water depths is apparent by comparing May with September water depths. The long-term temporal range in water depth (minimum, maximum) is indicated for selected gaging stations in four of the seven subareas in Figure 6.6. The longest period of record was 47 years for Station P33. The water depths at sampling stations in the immediate proximity of these gages are shown for both the dry and wet seasons in 1995, 1996 and 1999. While there is considerable temporal variability, the Phase I and II dry season water depths are within the long term range for water depths, in these subareas. During the wet season, however, some of the Phase I and II water depths were outside the maximum range previously recorded for the station. In general, considering both spatial and temporal variability, the Phase I and II hydrologic regime spans the historical range of water depths and should provide an adequate baseline for detecting future changes and trends in ecological condition associated with management actions.

The flow path through the marsh system is apparent by considering the spatial distribution of water depth throughout the system (Figure 6.5). Water is discharged from the Everglades Agricultural Area into the canals. Seepage water from the canals enters any of the marsh areas that border the canals (e.g., Loxahatchee National Wildlife Refuge) or through which the canals flow (e.g., WCA3-SE). The general flow path through the marsh is along the eastern side of the system from WCA2 through WCA3-SE and down Shark Slough to Florida Bay. This flow path, based on water depth, is corroborated in subsequent sections of this chapter by considering spatial patterns in conductivity, chloride and other constituents in the system.

The water depth intervals shown in Figure 6.7 correspond to the hydroperiod ponding depth classes predicted by the South Florida Water Management Model (SFWMM). The May and October average ponding depth classes predicted by the SFWMM for the period of record (POR) from 1965 through 1995 are shown in Figures 6.8 and 6.9. The May 1996 water depths are generally comparable to the average POR ponding depths, while May 1999 is significantly drier than the May average POR ponding depths. September 1995 had water depths that also

were similar to the average POR ponding depths for October (September depths were not available). September 1999 had ponding depths that were significantly greater than the October POR average ponding depths (Figure 6.9). Thus, 1999 represented both an exceptionally dry season and an above average wet season.

Because the sampling design was based on a systematic, probability sample survey, there was a relatively uniform distribution of sites throughout the system. A systematic distribution of points is particularly advantageous when using spatial statistical software such as SURFER and ARCVIEW. In addition, the probability samples permit estimates of the surface area associated with each sampling site. The measurements taken at sites were used to characterize conditions, including water depth, for the entire 5,500 km^2 area. Using the mean depth computed for the areas inundated during the dry seasons from 1995 to 1999 and the wet season in September 1996, water volumes were estimated for each season (i.e., volume = mean depth multiplied by surface area).

Examination of stage duration curves for gages located in southern WCA3 and northern Shark Slough indicated about 5 inches of water were ponded behind (i.e., north) Tamiami Trail during the 1999 dry season. A surface water volume to surface area curve for the ecosystem was developed using GIS techniques for the four driest sampling cycles. A fifth point to estimate the loss of ponding in the system was determined by subtracting 5 inches from the dry 1999 water levels (Figure 6.10). The curve illustrates the very large surface area to volume ratio characteristic of this ecosystem. It also indicates that the 5,500 km^2 ecosystem is covered with a surface water volume of about 2.9×10^9 m^3. Under extreme drought conditions, the surface water volume in the marsh declines to about 0.5×10^9 m^3. Elimination of ponding in the system would result in an additional dry area of about 400 km^2 of present slough habitat. The long and intermediate hydroperiod area of the marsh occupied about 4,200 km^2 with an associated volume of 1.5×10^9 m^3. To inundate the additional 1,300 km^2 of marsh required an equivalent volume of water even though the surface area was about one-third of the longer hydroperiod marsh.

Hydroperiod management to sustain ecological resources will require substantial quantities of water to maintain minimum habitat coverage during the dry seasons, while the short hydroperiod portion of the marsh beyond 4,200 km^2 will most likely remain dependent on the wet season rainfall. Due to the present system of levees and canals, ponding in the system occurred primarily in WCA3-SW, WCA3-SE and NE Shark Slough with smaller areas along the

southern reaches of Lox and WCA2 (Figure 6.11). The surface areas of inundation illustrated show the area without ponding <dry 1999 water level <4,200 km^2 <1,300 km^2 describing the long, intermediate, short, and extremely short hydropatterns, respectively. Drought prone areas were the northern tip of Lox, WCA3-N and Taylor Slough. A major loss of peat soils has occurred in WCA3-N. Water management to establish surface flow in extremely short hydroperiod marshes like WCA3-N will be a considerable challenge. Alternatively, maintenance of ponded slough habitat during drought conditions is critical because the most stable aquatic habitats with a rich flora and fauna occur in these areas.

This study provided a synoptic look at the water regime over the entire system during both dry and wet seasons. It spanned the range of hydrologic conditions that typically occur in the system and provided a sound baseline for evaluating future changes in hydropattern during restoration. It also provided a general surface area to volume relationship that can be used to quickly evaluate volume requirements for different inundation regimes.

6.2 Surface Water Quality

Landscape surface water chemistry patterns are presented in two formats using spatial plots of the entire study area and median plots for each of the seven subareas with 95% confidence intervals. Plots are shown for dry (April/May) and wet (September) sampling cycles in Phase I (1995-96) and Phase II (1999) to allow comparisons between seasons and over time. These data provide the basis for empirical models of the ecosystem. Emphasis is placed on comparability or differences among the patterns identified in each presentation. For example, conductivity is a tracer of flow through the marsh. Other constituent patterns will be compared with the conductivity patterns to see if these constituent concentrations also reflect the flow path. Some constituents with particulate and dissolved phases (e.g., nutrients) may show different patterns because of uptake and sedimentation. Source contributions can also be inferred from the patterns (e.g., contributions from the EAA for most constituents except mercury). The implications of these differences will be incorporated into an Ecological Risk Assessment (Chapter 8.0). Because of drought in some subareas there were indicators (e.g., fish, periphyton) that occasionally had three values or less measured in the subarea. These subareas were not represented in the figures because of the small sample size. However, the median values for these indicators and subareas are included in numeric form in Table 6.2.

6.2.1 pH

An ANOVA of the Phase I and II wet season data for the whole ecosystem showed a significant ($p = 0.001$) increase in the pH in 1999 (Table 6.3). The median pH of the Everglades ecosystem was consistently greater than 7 su in every subarea except Lox, which was significantly lower at about 6.5 su or less during all cycles (Figure 6.12). An increasing gradient in pH was evident during the 1995-96 wet cycles, ranging from a median in WCA2 of 7.13 to 7.68 su in Taylor Slough (Table 6.2). The wet season median gradient in 1999 was consistent at about 7.4 su from WCA2 to WCA3-SW, significantly increasing to medians of 7.8 su in Shark River and Taylor Slough (Figure 6.12). These down system increases in pH reflect the increase in marl versus peat in Shark River and Taylor Sloughs. Downstream pH gradients during the dry sampling cycles were less evident (Figure 6.13). However, all subareas south of Lox remained above pH 7. There was limited influence of low pH water from Lox on the downstream marsh in WCA2 due to the interception and transport of water out of the system by the Hillsboro Canal and the weak acidity in Lox. The acidity of Lox may result from the greater depth of peat, which is predominant in the subarea, and from Lox being a precipitation dominated, weakly buffered system. The peat depths in subareas to the south decline and marl increases resulting in more contact of surface water with the underlying bedrock that produces higher pH values.

6.2.2 Conductivity

Conductivity in the dry season was significantly higher than during the wet season due to the concentrating effect of evaporation and low flow through the system. A downstream gradient from WCA2 to Taylor Slough was apparent during all cycles. During the wet seasons the median concentrations ranged from 684 and 659 FS/cm in WCA2 to 294 and 254 FS/cm in the Taylor Slough for 1995-96 and 1999, respectively (Table 6.2). The concentrations during both phases of the study were significantly lower in WCA3-SW, and Shark River and Taylor Sloughs than the three subareas immediately upstream, where the greatest changes occurred (Figure 6.12). Lox was least impacted by agricultural runoff waters and consistently had median concentrations less than 301 FS/cm, which occurred during the extreme 1999 dry season (Figure 6.12). The Lox subarea is dominated by rainfall and water flow from the center toward the perimeter. However, the canals surrounding Lox change the water quality around the perimeter of the refuge. The spatial plots (Figure 6.14) of conductivity in the marsh define a predominant flow path of water

down the east side of the ecosystem. Maximum concentrations of over 1000 FS/cm were flowing into WCA2 from the Hillsboro Canal during each of the wet seasons. Concentrations above 400 FS/cm define a footprint across subareas WCA2, WCA3-N, and WCA3-SE. Large areas of intermediate conductivity (<400 FS/cm) predominated in WCA3-SW, Shark Slough and Taylor Slough. Lowest conductivities (<200 FS/cm) in the system consistently occurred near the center of Lox and WCA3-SW. A well defined gradient (Figure 6.14) occurred across the seven subareas during each wet season and the "wet" dry seasons sampled in 1995 and 1996. During the extreme dry down in 1999, however, when surface flow through the system was interrupted, the highest conductivity (median = 1417 FS/cm) occurred along the southern edge of WCA2 and WCA3-N. These high values are likely to have resulted from the remnant flow of water into the system from the EAA and shallow ground water drainage into the declining surface water pool. An ANOVA showed that there was no significant difference in conductivity measurements over the entire system between Phase I and Phase II wet seasons ($p = 0.97$) (Table 6.3).

6.2.3 Chloride

Chloride concentrations in surface water were measured during the 1999 surveys. The dry season concentrations were significantly higher in WCA2 and WCA3-N than in the wet season (Table 6.2). Dry season concentrations ranged from medians of 150 mg/L in WCA2 to 34 mg/L in WCA3-SW. Southern Lox was also relatively low with a median of 43 mg/L. Wet season concentrations described a strong gradient through the system following the flow path down the east side of the system from a median of 80 mg/L in WCA2 to11 in Taylor Slough (Figure 6.12). WCA3-SW, Shark Slough, and Taylor Slough had significantly lower medians of 12, 16 and 11 mg/L, respectively, than the immediate upstream subareas (Figure 6.12). The surface water chloride pattern indicates that the center of Loxhatchee, WCA3-SW and Taylor Slough were the subareas in the system during the 1999 wet season with concentrations less than 20 mg/L (Figure 6.15). During the wet season it was apparent that most of the chloride was entering the system from the Hillsboro and upper Miami canals in WCA2 and WCA3-N following the flow path down the east side of the system into Shark Slough (Figure 6.15).

6.2.4 Sulfate

Comparison of the sulfate data is affected by the minimum detection limit of 2 mg/L during Phase I. In Phase II, however, the minimum detection limit was 0.05 mg/L (Figure 6.16). Much of the southern 2/3 of the system had sulfate water concentrations at less than 2 mg/L. The footprint of sulfate across the marsh is particularly striking with median concentrations ranging from 23 to 44 mg/L (Table 6.2) in WCA2 and near the Miami Canal in northern WCA3-N (Figure 6.17). The median concentrations in the northern four subareas were not significantly different from Phase I to Phase II (Figure 6.16). The southernmost subareas (WCA3-SW, Shark Slough and Taylor Slough) had significantly lower medians (<1 mg/L) in 1999 than in 1995-96 due to a reduction in detection level. A significant decline in large amounts of available sulfate occurred across WCA2, WCA3-N and WCA3-SE and very low concentrations (<1 mg/L) occurred in the lower three subareas of the ecosystem. An ANOVA of the Phase I vs II wet season systemwide data (Table 6.3), however, indicated there was not a statistically significant difference between the two Phases ($p = 0.28$). Strong gradients were evident extending down the east side from WCA2 and WCA3-N to WCA3-SE during each cycle, with the lowest sulfate concentrations in WCA3-SW, and Shark River and Taylor Sloughs. WCA3-SW was relatively uncontaminated by sulfate during each sampling Cycle and represents a part of the system least affected by storm water runoff. The dry season sample in May 1999 had a median concentration of 56.5 mg/L in WCA3-N, which may have resulted from the extreme drought and associated wildfire that occurred in this area 2 weeks prior to sampling. The surface water sulfate gradient in the wet season 1999 did not impact most of WCA3-SW west of a north-south line from the intersection of I-75 and Miami Canal (Figure 6.17). All of the area east of this line (WCA3-SE) was impacted with excess sulfate ranging from 10 mg/L in the north to 1 mg/L in the south. Excess sulfate concentrations from 2 to <1 mg/L extended down Shark Slough (Figure 6.17). Background concentrations at less than 2 mg/L were consistently found in the center of Lox. However, there were sharp sulfate gradients from the center toward the surrounding canals, where sulfate concentrations reached 30 mg/L. The source of sulfate is associated with the agricultural runoff water entering the system (Orem et al. 1999, 2000), as is the case with conductivity and chloride. It has not, however, been conclusively determined whether the entrainment of connate seawater from underground cavities also contributes to higher constituent concentrations during pumping at S5, 6, 7, and 8.

6.2.5 Sulfide

Sulfide in surface water was measured in Phase II following development of the syringe sampling and preservation method. Therefore no comparison can be made with Phase I. Dry season surface water sulfide concentrations were significantly higher than wet season concentrations (Figures 6.16, 6.18). However, the foot print across the marsh with remaining surface water showed a dry season pattern in WCA3-SE and WCA3-SW that may have been influenced by the L-67 canal. A median concentration of 0.21 mg/L occurred in WCA2, but median concentrations were 0.06 mg/L or less in all other subareas (Table 6.2). September 1999 wet season median concentrations were 0.01 mg/L in all subareas except Taylor Slough, which was less than the 0.007 (detection level). Low level surface water sulfide patterns were more prevalent in the northern 2/3 of the ecosystem. Most of Shark and Taylor Slough subareas were below the detection level of 0.007 mg/L (Figure 6.18).

6.2.6 Total Organic Carbon

There was no significant wet season change ($p = 0.99$) in the systemwide TOC concentrations from Phase I to Phase II. Wet season concentrations ranged from median concentrations of about 30 mg/L in WCA2 to around 8 mg/L in Taylor Slough (Figure 6.16). Dry season concentrations were higher with a maximum concentration in WCA3-N during dry 1999 of 45.98 mg/L (Table 6.2) which may have been a result of the wildfire that burned this subarea 2 weeks before sampling. The TOC gradient emanated from the North New River Canal into WCA2 and the Miami Canal in WCA3-N and followed the flow path down the east side of the ecosystem during the wet seasons (Figure 6.19). Taylor Slough had the lowest TOC concentrations with less than 8.6 mg/L. WCA3-SW, Shark Slough and parts of Lox were less than 20 mg/L (Figure 6.16). The east-west gradient in TOC in WCA3 indicates a water quality footprint consistent with other parameters that follow the flow path through the system (Figure 6.19), however, TOC concentrations were relatively high everywhere in this system except in Taylor Slough. The high concentration of organic matter in the northern third of the ecosystem is apparently due to the runoff from the EAA. TOC can serve as a ligand, with available binding sites for many labile water quality parameters. Interactions with TOC are of primary importance in the bioavailability of metals, including mercury. The binding of total

methyl mercury on dissolved organic colloids in Everglades surface water has been demonstrated by Cai etial, 1999 & Cai, 1999).

6.2.7 Total Phosphorus

The highest wet season median concentrations of TP were 15.97 and 11.37 Fg/L in Phase I and II, respectively, and both occurred in subarea WCA3-N (Figure 6.20). Concentrations declined across WCA3-SE and WCA3-SW in both wet season samples, with medians less than 9 Fg/L in 1995-96 and 7 Fg/L in 1999 throughout the lower four subareas of the ecosystem (Table 6.3). An ANOVA found a significant decline ($p = 0.004$) in the total phosphorus concentrations in wet season Phase II compared to wet season Phase I. Dry season concentrations were elevated during both Phases with an extreme median concentration of 229.19 Fg/L in WCA3-N following a wildfire in May1999. The variance within subarea declined from Phase I to II. A gradient in phosphorus concentrations was evident in the system with high concentration inflows occurring in WCA2 and WCA3-N emanating from the North New River and Miami Canals (Figure 6.21). Inflow of water from Big Cypress National Preserve with total phosphorus concentrations in excess of 15 Fg/L into western WCA3-N and WCA3-SW was evident. The wet season gradient moved northward from Phase I to II, another indication that a significant decline in phosphorus input to the system had occurred by September 1999. Wet season concentrations indicated that over 2/3 of the system in 1999 had total phosphorus concentrations in water of 10 Fg/L or less. The excess concentrations continue to occur in WCA2 and WCA3-N from overflows from the North New River and Miami canals. The observed reductions in TP concentrations in the southern subareas would be expected to occur first if the overall ecosystem loading is being reduced in the northern inflows.

6.2.8 Total Nitrogen

Total nitrogen in water showed a significant wet season decline ($p = 0.000$) from Phase I to Phase II across the entire ecosystem (Table 6.3). A comparison of wet season median plots of total nitrogen in surface water showed a significant decline occurred in WCA3-SE, WCA3-SW, Shark Slough and Taylor Slough in 1999 while the upper three subareas showed no significant change (Figure 6.20). Dry season concentrations were higher than wet season concentrations in both phases. A gradient downstream occurred during both wet seasons with medians ranging

from 1.51 in WCA2 to 0.78 mg/L in Taylor Slough during 1995-96 (Table 6.2). The gradient in 1999 had medians from 1.22 mg/L in Lox decreasing downstream to 0.32 mg/L in Taylor Slough. The total nitrogen foot print in September 1996 followed the flow path through the system with WCA3-SW and Taylor Slough the least affected areas (Figure 6.22). The decline in total nitrogen concentrations in September 1999 showed that over 2/3 of the lower system was less than 1 mg/L. The high total nitrogen concentrations found in WCA3-N and northern WCA3-SW in dry 1999 may have resulted from the wildfire which preceded sampling by two weeks.

6.2.9 Total Mercury

There was a significant decline in wet season total mercury across the entire ecosystem from Phase I to Phase II ($p = 0.000$) (Table 6.3). The highest wet season median concentrations occurred in Lox (3.4 ng/L) and WCA2 (2.26 ng/L) and the lowest wet season median concentration occurred in WCA3-SW (1.01 ng/L) in 1999 (Table 6.2). Taylor Slough showed an increase in both wet seasons (Figure 6.23). Most of the inorganic mercury in water was strongly influenced by the amount of rain falling in the system as wet deposition. The easternmost subareas (Lox and WCA2) of the ecosystem are located in the area of maximum rainfall, which may explain the higher surface water concentrations found there. Dry season samples showed higher total mercury concentrations in water during dry down (Figure 6.24) and the associated concentration effects, which were most pronounced in Lox and WCA3-SW (Figure 6.24). The latter may have been the result of a wildfire in WCA3-N that preceded the sampling by two weeks. Total mercury concentrations in surface water were found to increase with decreasing average water depth ($r^2=0.895$) when all six sampling cycles were analyzed (Figure 6.25). Because wet atmospheric deposition is a major source of the total mercury concentrations in water during the wet season, the decline throughout most of the system in 1999 might indicate that local emission controls are having an effect.

6.2.10 Methyl Mercury

An ANOVA of the Phase I and Phase II wet season data for the whole ecosystem showed a significant ($p = 0.020$) decline in methyl mercury concentrations in water occurred in September 1999 (Table 6.3). Methyl mercury concentrations in water were consistently higher in

the three northern subareas (WCA1, WCA2 and WCA3-N) during both wet and dry seasons. However, the dry season data was much more variable (Figure 6.23). Dry season concentrations were generally twice the wet season concentrations, which had median concentrations of 0.4 ng/L in the northern three subareas, declining to the lowest median concentration of 0.06 ng/L in Taylor Slough (Table 6.2). Most of this decline occurred in subareas WCA3-N, WCA3-SE, WCA3-SW, and Shark Slough in the southern 2/3 of the system. The median wet season concentration in WCA2 was 0.74 ng/L, the only subarea with an increase in 1999. A declining gradient to the south was apparent in wet season data. Wet season concentrations greater than 0.4 ng/L, predominated in the northern half of the ecosystem, however, dry season concentrations may exceed this in all parts of the system that remain wet except for the southwest portion of WCA3-SW in 1999 and Taylor Slough during both wet seasons (Figure 6.26). A relationship of methyl mercury concentration to mean water depth showed an increase with decreasing depth ($r^2 = 0.71$) following analysis of all six sampling cycles (Figure 6.27). The occurrence of high levels of methyl mercury can be anywhere in the system during dry down. However, during the wet season, methyl mercury in water was closely associated with the agricultural runoff waters containing elevated levels of TOC, SO4, TP and other constituents entering the northern parts of the system. We have previously determined the generation of methyl mercury occurs primarily in the marsh and that significant quantities are not imported to the system with agricultural runoff waters (Stober et al. 1998).

6.3 Porewater

6.3.1 Sulfide

Porewater sulfide concentrations were determined in Phase II following development of an appropriate methodology. A north to south gradient was apparent (Figure 6.23) in September 1999, declining from median concentrations of 1.02 mg/L in subarea WCA2 to 0.05 mg/L in Taylor Slough (Table 6.2). The footprint of porewater sulfide (Figure 6.28) showed that it occurred in association with the flow path of the water through the system with highest concentrations in WCA2 and WCA3-SE. Under wet conditions the center of Lox, WCA3-SW, Shark Slough and Taylor Slough had porewater sulfide concentrations of 0.28 mg/L or less (Figure 6.23). During the dry season the least affected areas remained the same except for Taylor Slough, which was dry. The spatial pattern of porewater sulfide in the system was remarkably

repeatable under both dry and wet conditions (Figure 6.28). Sulfide can significantly affect the availability of total and methyl mercury in the system.

6.4 Floc

Floc was defined in the field as the slurry of particulate matter and water which was trapped on the top of the soil core sample in the process of soil sampling. The slurry was pored into an Imhoff cone to concentrate the particles, which became the floc sample. Floc was limited in Taylor Slough. This resulted from a shortage or complete lack of sufficient floc material due to the extreme drying of the area during the dry season and the very low productivity. For many Taylor Slough sites no samples could be collected. Any floc samples that could be obtained were analyzed for total and methyl mercury.

6.4.1 Ash Free Dry Weight

The percentage of organic matter in the five upstream subareas (Lox, WCA2, WCA3-N. WCA3-SE, WCA3-SW) ranged from medians of 94.8 % in Lox to a low of 84.58 % in WCA3-N and WCA3-SE during both seasons (Figure 6.29). A significant decline in percent organic matter occurred in the Shark Slough subarea with a median of 60.43 % (Table 6.2). Floc was not recorded for Taylor Slough. AFDW in floc in the upper 2/3 of the ecosystem was over 80 % and Lox and WCA3-SW were over 90% (Figure 6.30).

6.4.2 Mineral Content

The percentage mineral content in the upstream subareas had medians ranging from 5.2% in Loxahatcee to 15.4 % in WCA3-N and WCA3-SE. The five upstream subareas were consistently low in mineral content (Figure.6.29). The mineral content in Shark Slough increased to a median of 39.6 % (Table 6.2). Figure 6.31 shows that floc in approximately 50% of the area of Shark Slough is 40 % mineral.

6.4.3 Total Phosphorus

Median total phosphorus concentrations in floc ranged from 568 Fg/g in Lox to 214 Fg/g in Shark Slough (Figure 6.29). Both extremes occurred during the dry season, with wet season concentrations bracketed by these two extremes. A wet season concentration of 560.3 Fg/g

occurred in WCA3-N (Table 6.2) with an apparent decline to Shark Slough. All subareas showed a high variance in the data. The spatial plots (Figure 6.32) suggest a phosphorus gradient from north to south with concentrations greater than 600 Fg/g in southwest WCA1, WCA2 and the northwest half of WCA3-N. Intermediate concentrations between 400-600 Fg/g occurred mainly in WCA3-SW with lower concentrations to the south.

6.4.4 Total Mercury

Median total mercury concentrations in floc ranged from 323.6 to 72.7 Fg/kg and indicated a decline downstream from WCA1 and WCA2 to the remaining subareas downstream (Figure 6.33). A dry season median of 331.9 Fg/kg occurred in WCA3-SW (Table 6.2). Spatial plots indicate concentrations greater than 200 Fg/kg occurred in large areas of WCA1, WCA2 and WCA3-SW (Figure 6.34). There may be an association between AFDW and total mercury in floc.

6.4.5 Methyl Mercury

Median methyl mercury concentrations in floc showed high variance in all subareas except Shark Slough and Taylor Slough (Figure 6.33). Wet season median concentrations ranged from 10.1 to 0.48 Fg/kg in WCA2 and Taylor Slough, respectively, suggesting a north to south gradient (Table 6.2). Spatial plots indicated concentrations greater than 2 Fg/kg were prevalent over most of WCA1, WCA2, WCA3-N and WCA3-SW during the wet season (Figure 6.35). Dry season concentrations were similar, suggesting that the floc concentrations were greater than 2 Fg/kg in the northern half of the ecosystem.

6.5 Soil Patterns

Patterns of soil organic content, mineral content and depth (subsidence/accretion) reflect processes that are occurring in the marsh system, as well as potential diagnostic indicators of changes that have occurred or are occurring in the marsh. This section presents these patterns, their variance as a function of season, and gradients or hot spots that are apparent in these soil constituents. Soil chemistry patterns are displayed as spatial plots. Soil patterns integrate loading and provide a better perspective than water chemistry on processes and patterns that have occurred over long time scales. Water chemistry provides a snapshot of seasonal conditions,

while soil patterns provide a better indicator of long term trends. Patterns are compared among the soil constituents to see if distributions observed in the water constituent concentrations reflect the longer term patterns observed over space.

6.5.1 Soil Depth

An analysis of variance of soil thickness showed no significant differences in the measurements from Phases 1 and 2 (Table 6.3), however, subarea WCA3-SW was close with a p value of 0.057. If accretion is occurring, it will be measured over longer time intervals than the time scale of this study to date. Medians of soil depth (Figure 6.36) show that maximum depths of nearly 3 m occur in Lox, which is significantly greater than any other subarea. WCA2 has the next greatest soil thickness with a median of 1.3 m. WCA3-N has a median soil depth of 0.4 m, suggesting that when compared to the subareas immediately downstream (WCA3-SE and WCA3-SW) that remain flooded most of the time, soil subsidence may have occurred. WCA3-N has been dried by decades of water management practices in the system. Minimum soil depth medians of around 0.3 m occur in Shark Slough and Taylor Slough (Table 6.2) where the bedrock is closer to the ground surface. Spatial plots of soil thickness for Phase 1 and Phase 2 are shown in Figure 6.37.

6.5.2 Soil Subsidence/Accretion

This determination was made by taking the present soil thickness and subtracting the average of the minima and maxima for soil thickness from the 1946 Davis map, resulting in accretion as positive and subsidence as negative soil depths. A plot of median soil depths shows that accretion of over 1 ft has occurred in Lox in the last 50 years (Figure 6.38). However, subsidence has persisted in WCA2, WCA3-N, WCA3-SE, and parts of WCA3-SW and Shark Slough. The worst case is in WCA3-N, which lost up to 2.45 ft over the same time period (Table 6.4). Little change has occurred in Shark Slough, however, accretion between 0.35 and 1.55 ft was observed in Taylor Slough. Spatial plots of the minimum and maximum peat loss show the areas of predominant soil loss concentrated in WCA2, WCA3-N, WCA3-SE and Shark Slough (Figure 6.38).

6.5.3 Ash Free Dry Weight

An analysis of variance of Phase I wet compared with Phase II wet seasons found Phase II AFDW was significantly greater than Phase I ($p = 0.00$) (Table 6.3). Median plots indicate that most of the increase in Phase II occurred in WCA3-N, Shark Slough and Taylor Slough (Figure 6.36). Comparing the medians of the five northern subareas (WCA1, WCA2, WCA3-N, WCA3-SE and WCA3-SW), WCA3-N had medians of 62 to 78% compared to the other subareas with medians of 82 to 97 % (Table 6.2). The AFDW declined significantly in Shark Slough and Taylor Slough with medians ranging from 43 to 21 %, respectively. Taylor Slough was consistently lower. Spatial plots of AFDW show the subareas with greater than 80% occurred in WCA1, WCA2, WCA3-SE and WCA3-SW (Figure 6.39), most of the area north of Tamiami Trail.

6.5.4 Mineral Content

Soil mineral content was also measured in 1999, which mirrors AFDW. The median plot shows 3% in Lox, increasing to 74% in Taylor Slough, indicating a great range in soil types (Figure 6.36). The median mineral content of soil in WCA3-N was higher (21 to 23 %) than found in the other five northern subareas which had medians of 3 to 14 %. The medians for Shark Slough and Taylor Slough significantly increased to a range from 57.7 to 74.5 % (Table 6.2). Spatial plots showed that mineral concentrations were generally highest south of Tamiami Trail and across WCA3-N (Figure 6.40).

6.5.5 Average Corrected Redox

An ANOVA comparing the average soil redox in Phase I to Phase II wet seasons showed no significant change ($p = 0.782$) (Table 6.3). Wet season medians of less than 100 mV were found in WCA2 and WCA3-N in 1995-96 while in 1999 WCA2, WCA3-N, WCA3-SE and Shark Slough had medians greater than 100 mV (Figure 6.41, Table 6.2). An average Eh less than 100 mV indicates anoxic or reducing conditions are occurring in the soils. Spatial plots indicate that most of the area affected by low Eh was concentrated in WCA2, WCA3-N, and WCA3-SE(Figure 6.42). Subareas that had large areas of oxic soils (Eh > 100 mV) were Lox, WCA3-SE, WCA3-SW, Shark Slough and Taylor Slough in 1995-96 and Lox, WCA3-SW and Shark Slough in 1999. Oxic soils are the typical condition throughout most of the Everglades marsh. Anoxic soil conditions result when excess nutrients are introduced with stormwater

runoff into the system. Most other wetland ecosystems have anoxic or reducing soil conditions similar to those found in WCA2 on at least a seasonal basis (Mitch and Gosselink, 1986) posing one of the fundamental differences found in the Everglades ecosystem.

6.5.6 Total Phosphorus

An ANOVA comparing total phosphorus in soil in Phase I to Phase II wet seasons showed a significant (P = 0.000) decline occurred in Phase II throughout the ecosystem (Table 6.3). Median plots of wet season data by subarea show increasing medians from Lox to WCA3-N a significant decline in WCA3-SE followed by an increase in WCA3-SW and declining to minimum concentrations in Taylor Slough (Figure 6.41, Table 6.2). It is apparent that the magnitude of total phosphorus decline in subareas south of WCA3-N was greater in Phase II than occurred in the northern three subareas. This response is to be expected as the total phosphorus loading to the system declines. The downstream subareas indicate the initial response, followed by declines in the more impacted areas upstream. Spatial plots of the combined total phosphorus in soil for 1995-96 and 1999 show the spatial change over time (Figure 6.43). The size of the area with concentrations exceeding 400 mg/kg has decreased sharply with the most impacted areas above this level in WCA2 and WCA3-N. The sites where cattails occurred are indicated showing distribution in WCA2, WCA3-N, and WCA3-SE coincident with high soil phosphorus and marsh disturbance.

6.5.7 Total Sulfate

An ANOVA comparing log transformed total sulfate in soil in Phase I to Phase II wet seasons showed a significant (P = 0.000) increase occurred in Phase II throughout the northern subareas impacted with excess sulfate from the EAA (Table 6.3). Median plats of Phase I (1995-96) wet season data described a steep gradient from Lox at 430 mg/kg to 71 mg/kg in Taylor Slough (Table 6.2, Figure 6.44). Comparative median values for the Phase II (1999) wet season described a steep gradient from WCA2 at 1600 mg/kg to 26 mg/kg in Taylor Slough. Wet season sulfate concentrations were about four times higher in 1999 than in 1995-96 and the gradient did not include Lox with a median of 170mg/kg. The dry season medians in 1995-96 described gradient similar to the wet season ranging from 296 to 78.5 mg/kg, however, the 1999 dry season medians increased to 2950, 3100, and 2500 mg/kg in WCA2, WCA3-N and

WCA3-SE while Lox, WCA3-SW, Shark Slough and Taylor Slough remained at 120, 81, 160, and 71.5 mg/kg, respectively. High soil sulfate concentrations in these subareas are coincident with high concentrations of sulfate in water and sulfides in porewater. Spatial plots illustrate the dramatic increase from the baseline 1995-96 condition to that found in 1999 during both dry and wet conditions (Figure 6.45). It is important to note that the soil sulfate footprint across the marsh is mostly restricted to WCA2, WCA3-N, and WCA3-SE and the WCA3-SW and Taylor Slough remained mostly free of excess sulfate contamination in soil. The high sulfate values found in 1999 followed an extreme drought which dewatered much of WCA2 and WCA3-N and exposing the soil to air oxidizing soil sulfides back to sulfates.

6.5.8 Total Mercury

An ANOVA of total mercury in soil comparing Phase I to Phase II wet seasons found no significant change ($p = 0.203$) (Table 6.3). Highest median concentrations were found in Lox, WCA2 and WCA3-SW which ranged from 130 to 180 Fg/kg (Figure 6.41). Medians for WCA3-N were lower and ranged from 85 to 110 Fg/kg. Wet season total mercury concentrations in soil declined from medians of 180 and 170 Fg/kg in 1995-96 and 1999, respectively to medians of 34 and 43.5 Fg/kg in Taylor Slough, respectively (Table 6.2). Total mercury in soil was generally greater than 120 Fg/kg throughout Lox and WCA2, however, the reoccurring hotspot with maximum concentrations was the center of WCA3-SW which was apparent in both phases and seasons (Figure 6.42).

6.5.9 Methyl Mercury

An ANOVA of methyl mercury in soil comparing Phase I to Phase II wet seasons found a significant increase ($p = 0.00$) in Phase II primarily due to increases in Lox, WCA2 and WCA3-N (Table 6.3). The plot of medians by subarea, however, shows a consistent gradient in 1999 from 5.03 and 4.79 Fg/kg during the dry and wet seasons, respectively, in Lox to 0.29 and 0.13 Fg/kg during the dry and wet seasons, respectively in Taylor Slough (Figure 6.41). The gradient was similar in 1995-96, however, median concentrations in Lox were 1.96 and 1.13 Fg/kg in the dry and wet seasons, respectively, declining to Taylor Slough concentrations of 0.22 and 0.1 Fg/kg during dry and wet seasons, respectively (Table 6.2). The variance was greater in 1999 than in 1995-96. Spatial plots show the highest concentrations of methyl mercury

in soil in Lox, WCA2 and WCA3-N were along the borders of these subareas with the Everglades Agricultural Area (Figure 6.47). These are areas were dry during the 1999 dry season.

6.6 Periphyton Mercury

Heavy growths of periphyton can serve as methylation sites within the marsh, but only under the right conditions (Cleckner et al. 1998). The periphyton mercury concentration, both for total and methyl mercury, are compared with nutrient distributions both in water and soil. In addition, changes in species composition are compared with the nutrient gradients and concentrations.

6.6.1 Average Total Mercury

Due to the inconsistent coverage of periphyton among seasons and years all sample types were averaged together to improve the data coverage. An ANOVA comparing Phase I with Phase II wet seasons showed a significant decline in Phase II ($p = 0.000$) (Table 6.3). The median plot of Phase I wet season data showed a gradient occurred throughout the system from a median of 352.6 Fg/kg in Lox to a median of 42.8 Fg/kg in Taylor Slough (Figure 6.48, Table 6.2). Wet season 1999 concentrations showed a less pronounced gradient with a median of 45.5 Fg/kg in WCA2 to 15.2 Fg/kg in Taylor Slough. Spatial plots demonstrate a tendency for higher total mercury in periphyton in the northern half of the system (Figure 6.49), however, the fact that periphyton did not occur in large enough quantities to sample at every station, especially north of Alligator Alley, reduced the consistency of the coverage. The significant decline in total mercury in periphyton from Phase I to Phase II may be a response to a reduction in atmospheric deposition, since this community is very closely associated with the initial uptake of mercury from atmospheric deposition.

6.6.2 Average Methyl Mercury

An ANOVA of methyl mercury in periphyton showed there was no difference in methyl mercury in averaged periphyton concentrations from Phase I to Phase II (Table 6.3). The median plots suggest that higher concentrations were found in Lox and WCA3-SW, with medians of 4.6 and 2.75 Fg/kg, respectively, in both dry and wet seasons (Figure 6.48, Table 6.2). Taylor

Slough had the lowest concentrations during each Cycle with medians ranging from 0.13 to 0.65 Fg/kg. The periphyton coverage was less consistent in 1999 than in 1995-96. However, the spatial plots show a tendency for larger spatial coverage of higher concentrations in WCA3-SW and Lox than most other subareas (Figure 6.50). This suggests that methyl mercury in periphyton may be an important factor in the availability of methyl mercury in the food chain in WCA3-SW.

6.7 Macrophyte Mercury

Concentrations in plant leaf tissue were measured at every site where cattail and sawgrass occurred during the May 1999 sampling cycle. Cattail coverage was limited to sites on the edge of Lox, WCA2, WCA3-N and WCA3-SE. Sawgrass occurred at all other stations.

6.7.1 Cattail Total Mercury

Median mercury concentrations in cattail ranged from 0.78 to1.57 Fg/kg in leaves (Figure 6.48). One subarea had a median of 6.35 Fg/kg, but there were only three sites in this subarea (Table 6.2). Concentrations in cattail tissue were very low and are of little importance as an indicator. A spatial plot of the sites where cattail occurred is shown in Figure 6.51.

6.7.2 Sawgrass Total Mercury

Median mercury concentrations in sawgrass ranged from 3.97 to 13.21 Fg/kg in leaves (Figure 6.48, Table 6.2). The highest concentration was found in WCA3-N, however, most sawgrass samples from this subarea were rapidly growing new leaves following a wildfire, which may have resulted in increased uptake of mercury in the tissue. A spatial plot describes the area of concentrations greater than 10 Fg/kg occurred mostly throughout WCA3-N the area of the wildfire in May 1999 (Figure 6.45). Macrophyte mercury concentrations in leaf tissue were found to be low. Translocation, or flux of mercury through the plant, was not considered. Lindberg et al (1999) has demonstrated that evasion of Hg^0 above a cattail marsh can be a significant pathway for mercury flux from soil to air. It is possible the macrophytes are having a greater effect on mercury distributions within the system than indicated based on tissue concentrations.

6.8 Mosquitofish, Food Webs, and Bioaccumulation

Mosquitofish mercury concentrations are displayed using median plots and spatial maps and comparisons are made among seasons, particularly the May and September seasons in 1999. The 1995-96 period was one of high water during the first year, with relatively high water during the second year. The May to September 1999 period permits us to examine how rapidly mosquitofish recolonize and accumulate mercury. Similar patterns in the distribution of mercury spatially may indicate that the factors controlling mercury methylation, uptake and bioaccumulation remain relatively constant over both time and space, but may be displaced in space by hydropattern.

6.8.1 Mosquitofish Total Mercury

An ANOVA of total mercury in mosquitofish was done comparing Phase I with Phase II wet season fish (Table 6.3). No differences were found ($p = 0.693$) on a systemwide comparison. However, when individual subareas were tested, WCA3-SW showed a significant decline ($p = 0.00$) as did Shark Slough ($p = 0.008$). Wet season comparisons of mosquitofish tissue concentrations of total mercury showed the highest median concentrations occurred in WCA3-SW and Shark Slough in both Phase I and II (Figure 6.52). Low median concentrations (56.2 and 57.1 Fg/kg) were recorded for WCA2 during both dry seasons (Table 6.2). Spatial plots clearly show the mercury hot spot in fish is located in the northern part of WCA3-SW trailing downstream through Shark Slough (Figure 6.53). The findings are similar for 1999, however, the concentrations are lower throughout these subareas. Mercury concentrations in fish are consistently low throughout Lox, WCA2 and WCA3-N, where methyl mercury in water is very high. Mercury concentrations in fish are also low in Taylor Slough but methyl mercury in the water is very low in this subarea. These differences between methyl mercury concentrations in water and fish mercury concentrations are reflected in the bioaccumulation factor or BAF.

6.8.2 Bioaccumulation

A bioaccumulation factor or BAF is the methyl mercury concentration in the biotic species or assemblage (e.g., mosquitofish or periphyton) divided by the methyl mercury concentration in water. Fish tissue is typically expressed as total mercury because methyl mercury constitutes 95 to 99% of the total mercury in fish (Bloom et.al. 1992).

An ANOVA comparing the Phase I and Phase II wet seasons showed a significant ($p = 0.04$) increase occurred in the bioaccumulation factor (BAF) in Phase II (Table 6.3). The higher methyl mercury concentrations in the water column in Phase I and the lower total mercury concentrations in the fish in Phase II could explain this change in the BAF. Median plots of the BAF show a gradient in the ratio from WCA3-N through Shark Slough during both wet seasons (Figure 6.48). During the 1995-96 wet season the median BAF remained below 350,000 in Lox, WCA2, WCA3-N and WCA3-SE but increased to around 800,000 in WCA3-SW, Shark Slough and Taylor Slough (Table 6.2). A similar gradient was observed in September 1999 with medians less than 465,000 in Lox, WCA2 and WCA3-N and a significant downstream increase to a median BAF of around 1×10^6 in the four downstream subareas. The wet season spatial plots show the BAF less than 600,000 in Lox, WCA2, WCA3-N and the northern part of WCA3-SE, while the BAF in WCA3-SW, Shark Slough and Taylor Slough rose above 600,000 (Figure 6.54) clearly showing the interface between the more impacted areas to the north and less impacted areas to the south.

6.8.3 Food Webs

Niche Breadth and Trophic Position

The gut contents of 2,784 mosquitofish collected from 259 sites (Figure 6.55) were quantified into 5 categories for analysis of trophic position and niche breadth (Table 6.4). Overall, midge larvae, pupae, and adults accounted for the primary diet item (34.5%), while an assortment of spiders, ants, and other surface prey accounted for a similarly high proportion of food (30.1%). Detritus was also an important food item, and accounted for 25.1% of the diet. Cladocera, mites, and other invertebrates too small to enumerate by mass (notably rotifers) comprised less than 10% of the diet by mass. Overall, the trophic position of mosquitofish was approximately 2.2, on a scale ranging from an herbivore as 1 and a piscivore consuming carnivorous fishes as 5. However, the niche breadth exceeded the average trophic position (2.3), indicating a large variance in diet among samples. Trophic score was highly correlated with the frequency of detritus/plant material in the diet, and to a lessor extent on adult dipterans and other prey items (Table 6.5). Adult diptera and other prey items, typically ants, are the individual prey items with the largest biomass in the mosquitofish diet.

Mosquitofish trophic position varied regionally and temporally, but most of the variation was among samples collected at smaller spatial scales within sampling cycles (Figure 6.56). While significant patterns were noted, the statistical model including both regional and temporal variation explained only 13.5% of the total variance in trophic position (Figure 6.57; regions: $F_{7,242} = 3.379$, $P = 0.002$; cycle: $F_{2,242} = 9.675$, $P < 0.001$; interaction ns). Tukey HSD pairwise comparisons indicated that fish from Taylor Slough had a higher trophic score than those from Shark Slough (df = 1,242 P=0.026), WCA3-SE (df = 1,242 P=0.012), and WCA3-SW (df = 1,242 P=0.080). Big Cypress was only sampled for gut contents in mosquitofish in Cycle 3, but averaged a higher trophic score than Shark Slough (df = 1,242 P=0.020), WCA3-SE (df = 1,242 P=0.010), and WCA3-SW (df = 1,242 P=0.046). The estimates of trophic score were least in Cycle 3 (Cycle 3 < 4 by 0.240, df = 1,242, P= 0.001; Cycle 3 < 4 by 0.181, df 1, 242, P = 0.001), and did not differ between cycles 4 and 5. However, most of the variance in trophic position was found among samples within study regions.

Niche breadth is a measure of the range of diet items observed within a sample. The observed average niche breadth of 2.3 (Table 6.6) indicates that the trophic scores for each sample derived from foods covering a wide range of trophic positions. While generally broad, there were no regional patterns in niche breadth, but there was some variation among sampling cycles (regions ns; cycle: $F_{2,234} = 15.907$, $P < 0.001$; interaction ns). Niche breadth was similar in cycles 3 and 5 (both in September) but was higher in Cycle 4 (Cycle 3 < 4 by 0.841, df = 1,234, $P < 0.001$ and Cycle 5 < 4 by 0.712, df = 1,234, $P < 0.001$).

The relative mix of plant matter/detritus and animal prey in their gut contents determined variation in the trophic score of mosquitofish. Though mosquitofish consume a variety of animal prey, all the animal types had similar trophic scores (2) such that choosing amongst them had little effect on the Adam's formula. The same can be said for their anticipated effects on mercury bioaccumulation. Thus, spatial and temporal patterns in the frequency of plant/detritus matter in mosquitofish guts are the primary determinant of variation in trophic score. Plant matter was more common in the gut contents of fishes collected in September 1996 than in either 1999 sampling (Figure 6.58). As expected, most of the variance in the frequency of plant matter in the diet of mosquitofish was within spatial regions similar to variation in trophic score.

Since over 85% of the variance in trophic position was found within study regions and sample times, we tested for correlations between trophic score and various environmental

parameters using backwards stepwise regression. Conductivity was the only environmental parameter that explained a significant amount of variation in these analyses. When added to a statistical model of trophic score that included sampling region and cycle, conductivity explained an additional 3.3% ($R^2 = 0.168$; region $F_{7,239} = 2.543$, $P = 0.003$; Cycle $F_{2,239} = 2.379$, $P < 0.001$; conductivity $F_{1,239} = 1.112$, $P = 0.002$). This relationship was explored through quadratic regression analysis of each prey category with conductivity and conductivity squared, using a backwards-stepping procedure. The dietary percentage of adult diptera, cladocera, other animal prey, and detritus/plant matter revealed a significant relationship with site conductivity, though none explained more than 4.8% of the total variance (Figure 6.59; Table 6.6). The relative abundance of animal prey based on counts of individuals were analyzed because weight estimates were near the minimum resolution for small numbers of small species like mites and cladocera. In spite of this, several prey types had substantial numbers of samples where they were absent, at times close to 50%. In such cases, the data may not be well modeled with the normal distribution and least-squares regression. These analyses were repeated with logistic regression which models binomial data and estimated the odds of a diet item being present or absent, relative to conductivity when it was collected. The results are illustrated in Figure 6.60, but are not reported in detail because they were consistent with the more common least-squares regression analyses.

The hypothesis that trophic position could be used to explain mercury concentration in mosquitofish was not supported. This hypothesis was tested using backwards stepping regression of mosquitofish mercury concentration and concentrations of soil, floc, and periphyton methyl mercury, as well as conductivity and trophic score. Only periphyton methyl mercury and conductivity were retained in this model (Figure 6.61). The trophic score was then replaced with the percentage of total weight comprised of each of our food categories, and niche breadth. In this case, periphyton methyl mercury, conductivity, and percentage of cladocera in the diet were retained in a regression model that explained approximately 34% of the variance in mosquitofish mercury (Table 6.7). Percentage of cladocera in the diet explained less than 1% of the variance in mosquitofish mercury, while periphyton methyl mercury was responsible for 28% of the explained variance.

Analyses reported in previous sections of this report note that mercury bioaccumulation is greater in WCA3-SW and Shark Slough in Everglades National Park than in WCA2 and

WCA3N. We tested for evidence that this effect was influenced by trophic score or environmental factors with a backwards stepping multiple regression of bioaccumulation (mosquitofish total mercury – periphyton methyl mercury) on estimated mosquitofish trophic score, geographic location north and south of the Tamiami Canal (we limited the northern sites to those between the Tamiami Canal and I-75), hydroperiod, and water total phosphorus. Consistent with the earlier analysis, we found that bioaccumulation was greater south of the Tamiami Canal and that it was correlated with trophic score (Figure 6.62). There was no significant correlation of water total phosphorus on this measure of bioaccumulation. Also, the trophic score correlation was small and negative, indicating that higher trophic scores were accumulating less mercury than those with lower scores.

Alternative Hypothesis

No link between trophic score or gut content data, in general, was found from estimates of mercury concentration in the tissues of mosquitofish collected simultaneously with those analyzed for gut contents. Interestingly, a relationship was found between periphyton methyl mercury and mosquitofish mercury concentration. Either the gut content data failed to adequately represent the diets of mosquitofish in the sample areas or the correlation between periphyton mercury and mosquitofish mercury was not a causal (trophic) one.

Gut content data provided a good estimate of trophic position when compared to independent estimates made from stable isotopes. Loftus (2000) found a significant correlation between δ^{15} N and trophic score (Pearson's $r = 0.681$, $P = 0.002$) estimated from gut content data using Everglades fishes with trophic classes ranging from 1 to 5. He observed a similar correlation between trophic score and tissue mercury concentration ($n = 28$, $r = 0.684$, $P < 0.001$). While mercury concentration did increase with increasing trophic class, Loftus (2000) noted that there was not a significant difference between trophic classes 1 and 2 or 2 and 3, though 2 did differ significantly from 4 and 5. In other words, while the presence of bioaccumulation is clear, the statistical power in his sample (and probably in general) was not so great as to reveal mercury concentration effects for shifts of 1 trophic level. The range of mosquitofish trophic scores we estimated, unfortunately, bridge this scale from 1.5 to 3.5. Thus, significant mercury bioaccumulation effects in mosquitofish trophic shifts will only be detected at the extreme of the species' trophic range.

Mosquitofish are clearly omnivores with highly varied diets (Harrington and Harrington 1961; Hurlbert and Mulla 1981; Crivelli and Boy 1987; Linden and Cech 1990; Daniels and Felley 1992; Nesbeit and Meffe 1993; Cabral et al. 1998). Experimental studies indicate that mosquitofish switch their prey choice relative to food availability (Bence and Murdoch 1986) and intraspecific competition (Taylor and Trexler, in press). Mosquitofish do consume algae from Everglades periphyton mats, though the mat structure can limit their ability to access it (Geddes and Trexler, in review). Thus, much of the diet variability in mosquitofish is among animal prey types with relatively little difference in mercury concentration (switching from midge larvae to cladocera). There is probably a seasonal shift in the relative amount of algae in their diet related to its abundance in the environment and availability for consumption (periphyton mat structure presumably becomes more complex as the growing season progresses). Finally, the data reported here suggest that there are spatial and temporal changes in the relative role of cladocera and midge larvae, and adult diptera, spiders, and ants in mosquitofish diets. The latter prey items indicate surface feeding while the former are water column or benthic dwellers. The shift in relative use of detritus/plant material and animal prey is likely to affect mercury concentration via bioaccumulation.

Loftus (2000) provided experimental evidence that spatial patterns of environmental mercury may influence mosquitofish mercury concentration more than diet variation. He raised neonate mosquitofish in cages placed in three paired short- and long-hydroperiod marshes in the Everglades National Park to test the hypothesis that hydroperiod influenced the rate of mercury uptake. The neonates were obtained from lab-reared females and were very low in mercury at the outset of the experiment. The diet of mosquitofish showed small differences between the two hydroperiods; fish from long-hydroperiod marshes consistently had lower trophic scores (more plant matter) than those from short-hydroperiod ones. At two of the paired sites, the long-hydroperiod fishes displayed less mercury, consistent with the prediction from bioaccumulation. However, at the third pair of sites the mercury concentrations were greater in the long-hydroperiod fish. The pattern of mercury in cage-reared mosquitofish matched the pattern from free-ranging specimens collected during the experiment. Stober et al.'s unpublished data on mercury in periphyton indicated that the anomalous pair of sites were located in a mercury hot spot of unknown origins. Thus, the major variation in the experimental results could best be explained by environmental mercury unrelated to hydroperiod or mosquitofish diet.

Conductivity explained more variation in mosquitofish mercury than did trophic position. Conductivity is correlated with flow path and nutrient level in the water column and is probably a surrogate for the effects of nutrient level on biogeochemistry of an area. While these nutrient effects could act to change the food web, nutrients could also act to change the availability of mercury in a local area and expedite its transfer to mosquitofish without changing the food web *per se*. While mosquitofish do eat more detritus/algae/plant matter in high nutrient sites, it doesn't explain much variation. This pattern is also the inverse of that predicted by increased mercury in mosquitofish inhabiting sites with high conductivity. Mosquitofish also eat more surface prey in high conductivity circumstances. The mercury effect could be related to surface film contamination equally as well as detritus.

There were no strong correlations explaining large fractions of the variance in mosquitofish mercury. The most convincing statistical model explained less than 50% of the mercury as a function of periphyton mercury concentration and conductivity. This lack of clear results probably results from multiple sources of variation in the data. In particular, individual fish make idiosyncratic foraging choices that influence their individual mercury contamination and yielding large niche breadth within a sample of fish. Also, there are unexplained but marked spatial patterns in mercury availability across the Everglades. These patterns appear to propagate through the food web locally and are reflected in mosquitofish living there. While mosquitofish diet and Everglades food webs vary seasonally and spatially, these patterns appear to yield small effects on mosquitofish contamination, at least compared to the effects of environmental availability.

The trophic cascade or "top-down" versus "bottom-up" approaches postulated for eutrophication (Carpenter et al. 1993; Harris 1994) might have relevance for mercury contamination. In the northern areas of the ecosystem (e.g., WCA2, WCA3-N), methyl mercury concentrations in water and soil are high, but mosquitofish mercury concentrations are low. The methyl mercury might not be biologically available because it is bound by organic and sulfide ligands. The periphyton mat is reduced and there may be more foraging on macrophyte detritus. In Shark Slough, methyl mercury concentrations are low, but mosquitofish mercury concentrations are high. In this area, the methyl mercury might be readily available for biological uptake, accumulation and magnification through the food web. The interaction of

local environmental conditions with food web and trophic dynamics, therefore, might explain the spatial patterns and variability observed in mosquito fish mercury concentrations.

6.9 Mercury Mass Estimates

Mass estimates of total mercury in precipitation, surface water, floc, soil, periphyton, and mosquitofish were calculated for each synoptic sample. Mass estimates were also made for methyl mercury in surface water, floc, soil, and periphyton. These estimates were developed to provide a relative perspective of instantaneous masses among constituents and not to develop a mass balance or budget. The models used to calculate Hg mass estimates are shown in Table 6.8. The thickness of the floc layer was difficult to accurately measure in the field. The floc layer thickness typically varied from about 0.01 to 0.1 (i.e., 1 - 10%) of the water depth. Floc mercury mass estimates, therefore, were estimated as a range. Periphyton densities were assumed to range from 171 g/m^2 dry weight in the ENP to 452 g/m^2 dry weight in WCA-3; based on ash free dry weight measurements collected by J. Trexler (personal communication). The density of fish was assumed to be 3.5 $fish/m^2$ during dry seasons and 14.5 $fish/m^2$ during wet seasons based on data gathered by J. Trexler (personal communication).

Mass estimates of total mercury in precipitation were also calculated for the wet and dry seasons corresponding to the sampling cycles. The mass estimate was calculated by multiplying total precipitation for the season by the area of the study area and the average of total mercury in precipitation measurements for the season. The wet season was assumed to be June through October, and the dry season was assumed to be November through May. The precipitation data used for the 1995 and 1996 calculations came from 5 National Oceanic and Atmospheric Administration (NOAA) weather stations located in the study area; Belle Glade Experiment Station, Devils Garden, Homestead Experiment Station, Royal Palm Ranger Station, and Tamiami Trail. Measurements of total mercury in precipitation for 1995 and 1996 were available for the 4 stations monitored for FAMS. Measurements of precipitation and total mercury in precipitation for 1999 came from 3 stations in the National Atmospheric Deposition Program, Mercury Deposition Network; FL04, FL11, and FL34.

The mass estimates for total mercury by media and cycle are compiled in Table 6.9. The system wide estimates for water range from 2.3 to 3.4 kg during the dry cycles and from 5.2 to

9.0 kg in the wet cycles. Higher loading during the wet season is consistent with the pattern of atmospheric deposition. Wet deposition of Hg during the wet season accounts for 80% of the annual total atmospheric deposition of Hg in the Everglades system.

Floc was collected only during 1999. The total mercury mass estimates for floc for 1999 indicate that this is a variable sink for mercury. The masses for the dry season and the wet season differ by an order of magnitude. This sink is also dependent on the amount of water in the system.

System wide estimates of soil total mercury were relatively consistent in the first four cycles ranging from 10,561 to 11,896 kg. In 1999 however, soil total mercury estimates were less than 10,000 kg. The soil represents the largest Hg sink in the system.

Total mercury mass estimates for periphyton were based on total mercury measurements in all types of periphyton. Periphyton total mercury mass estimates were variable, ranging from 22.7 to 227.5 kg during the wet seasons, and from 30.7 to 90.9 kg in the dry seasons.

Total mercury mass estimates in mosquitofish were extremely low, ranging from 0.06 to 0.44 kg during the dry cycles and 0.57 to 0.83 kg during the wet cycles. The low estimates obtained may be partly due to low biomass estimates used to represent the standing stock.

System wide mass estimates of methyl mercury for water, floc soil, and periphyton, by cycle are presented in Table 6.10. Methyl mercury mass estimates in water ranged from 0.58 to 1.6 kg during the dry cycles to 0.92 to 1.8 kg during the wet cycles. The consistency in these estimates indicates that the amount of methyl mercury is likely controlled by internal processes in the marsh rather than outside influences external to the marsh (e.g., atmospheric deposition).

System wide mass estimates of methyl mercury in soil ranged from 68 to 120 kg during the dry cycles and 39 to 131 kg during the wet cycles. It is interesting to note that 1999 methyl mercury in soil mass estimates were an order of magnitude greater than the 1995 and 1996 estimates, whereas total mercury in soil estimates for 1999 were an order of magnitude lower than the previous years.

Mass estimates of methyl mercury across the system for periphyton ranged from 1.3 to 5.7 kg during the dry cycles and 1.2 to 2.1 kg during the wet cycles. The greatest mass estimate was for cycle 0. The lowest mass estimate was for Cycle 5.

Areal mass estimates were also calculated for subareas of the Everglades for each cycle. The subareas were LOX, WCA2, WCA3-N, WCA3-SE, WCA3-SW, SRS, and TS. Figures 6.63 and 6.64 are plots of areal mass estimates of total mercury and methyl mercury in water and soil.

As expected, areal mass estimates of total mercury in water tended to be higher during the wet cycles. Areal mass estimates of total mercury in soil were consistent between cycles, with no seasonal pattern apparent. For the soil, there was a strong north to south gradient with greater loads in the southern subareas. This pattern corresponded to the general pattern of water flow in the system.

The patterns of areal mass estimates of methyl mercury in water and soil were fairly consistent between cycles. Methyl mercury in water tended to decrease from north to south. Methyl mercury mass estimates in water were also more variable in the WCAs than in SRS and TS. Methyl mercury mass estimates in soil were highest in LOX and WCA3-N. During 1999 (cycles 4 and 5) the areal mass of methyl mercury in soil were greater than in 1995 and 1996 in most of the subareas. Areal masses of methyl mercury in soil were very similar for all cycles in WCA3-SE.

6.10 Landscape Summary

Table 6.3 is a summary of ten water quality parameters, one porewater parameter, five constituents of floc, nine soil constituents and five biological tissues and a BAF index. The table shows the constituent, the median high and low, whether there was a gradient and the direction from high to low, the subareas included in the gradient and the significance of change from Phase I (1995-96) values to Phase II (1999) values. A complete or partial gradient was indicated in all parameters with the exception of total mercury in water, soil subsidence, and total mercury in cattails. Most gradients changed from high to low extending from north to south with the exception of pH, mineral content in floc and soil, redox in soil, total mercury in mosquitofish and the bioaccumulation factor. Total mercury in water and subsidence did not show a gradient across space either due to higher concentrations on both ends of the system or change in the middle of the system. Surface and porewater quality constituents were strongly influenced by

agricultural runoff from the EAA and showed greatest change from subareas WCA2 to Taylor Slough with decreasing extent. The exceptions were pH, total nitrogen, and total mercury which showed gradients from Lox to Taylor Slough. Floc constituents showed greatest change from subarea WCA3-SW to Shark Slough for AFDW and mineral content and total phosphorus changed most in subareas WCA3-SE to Shark Slough. Total and methyl mercury in floc changed across the entire ecosystem from north to south. Most natural soil constituents changed across the entire ecosystem from Lox to Taylor Slough. Soil redox and total phosphorus gradients changed most from WCA2 to Taylor Slough while the change in soil total mercury was most pronounced from WCA3-SW to Taylor Slough. Methyl mercury in soil changed from the edge of the EAA in all bordering subareas to Taylor Slough. Average periphyton total mercury changed across the entire system while methyl mercury changed most from WCA3-SW to Taylor Slough. Cattail total mercury concentrations were measured only where this species is most abundant (WCA2 - WCA3-SE). The change in sawgrass total mercury was most apparent from WCA3-N to Taylor Slough. Mosquitofish changed most from subarea WCA2 to WCA3-SW. The bioaccumulation factor changed across the entire ecosystem from south to north.

Significant surface water declines in phase I and II wet season concentrations were found in total phosphorus, total nitrogen, total mercury, and methyl mercury. Total phosphorus in soil also declined significantly, however, methyl mercury increased. Wet season mean total mercury in periphyton decreased significantly from Phase I to II and the bioaccumulation factor increased. Collectively these observations suggest that total mercury and total phosphorus in this ecosystem are declining and that collective actions to control local atmospheric mercury emissions and deposition and runoff of phosphorus by best management practices in the EAA are beginning to achieve the desired responses. However, only continued monitoring can verify these responses.

Table 6.1 Precipitation summaries for the 9 stations used to establish the long-term norm and baseline precipitation conditions.

STATIONS									
	S5A	**BELLE GLADE**	**DEVILS GARDEN**	**S6**	**S39**	**S8**	**S9**	**TAMIAMI TRAIL**	**ROYAL PALM**
LONG TERM AVERAGE PRECIPITATION (cm)									
	142.5	137.3	132.2	140.8	126.7	131.4	119.1	125.7	127.5
NUMBER OF YEARS									
	38	66	58	36	32	27	35	57	47
ACTUAL PRECIPITATION (cm)									
1992	151.1	146.8	141.0	109.9	M	133.6	122.3	88.6	121.3
1993	128.0	133.3	140.9	91.7	99.4	141.9	108.4	146.0	114.1
1994	217.5	195.5	144.3	193.0	114.2	167.9	181.0	171.1	111.6
1995	144.8	146.9	163.5	137.6	138.6	140.5	137.3	112.9	141.6
1996	159.8	129.8	119.0	126.6	91.6	126.6	103.8	127.4	96.8
1999	116.3	110.7	141.4	M	156.7	151.2	M	146.5	M
PERCENT OF LONG TERM AVERAGE PRECIPITATION									
1992	106%	107%	107%	78%	M	102%	103%	73%	95%
1993	90%	97%	107%	65%	78%	108%	91%	121%	89%
1994	153%	142%	109%	137%	90%	128%	152%	142%	87%
1995	102%	107%	124%	98%	109%	107%	115%	94%	111%
1996	112%	95%	90%	90%	72%	96%	87%	106%	76%
1999	82%	81%	107%	M	124%	115%	M	116%	M

M = missing data

Table 6.2. Surface water, floc, soil, and tissue medians and 95% CI by Everglades ecosystem subarea for Phase I (1995-1996) wet and dry seasons and Phase II (1999) wet and dry seasons).

			AREA						
			MEDIAN (95%CI)						
Parameter	Phase	Season	LOX	WCA2	WCA3-N	WCA3-SE	WCA3-SW	SHARK SLOUGH	TAYLOR SLOUGH
pH, su	1	D	6.26(6.44,6.08)	7.37(7.5,7.24)	7.25(7.33,7.16)	7.33(7.38,7.28)	7.18(7.28,7.08)	7.49(7.64,7.34)	7.28(7.35,7.2)
	1	W	6.54(6.84,6.24)	7.13(7.25,7)	7.1(7.17,7.02)	7.34(7.42,7.26)	7.31(7.41,7.21)	7.63(7.73,7.53)	7.68(7.78,7.58)
	2	D	6.18(6.64,5.72)	7.88(8.3,7.46)	7.27(7.27,7.27)	7.3(7.44,7.16)	7.1(7.22,6.98)	7.35(7.52,7.18)	
	2	W	6.59(6.8,6.38)	7.4(7.53,7.27)	7.39(7.46,7.32)	7.42(7.5,7.34)	7.36(7.48,7.23)	7.79(7.94,7.63)	7.88(8.03,7.73)
CONDUCTIVITY, uS	1	D	161.5(212.56,110.44)	935(1042.61,827.39)	699(880.43,517.57)	638(662.54,613.46)	450(490.96,409.04)	600(646.46,553.54)	535(600.84,469.16)
	1	W	69(145.08,-7.08)	684(784.17,583.83)	462.5(544.3,380.7)	553.5(598.88,508.12)	316(351.75,280.25)	392(433.15,350.85)	294(328.63,259.37)
	2	D	301(412.28,189.72)	875(1068.57,681.43)	1417(1417,1417)	621(665.97,576.03)	400.5(453.93,347.07)	676.5(772.64,580.36)	
	2	W	117.95(267.09,-31.19)	659(830.14,487.86)	638(791.24,484.76)	465(517.84,412.16)	243(292.72,193.28)	265.5(291.83,239.17)	254(286.03,221.97)
SURFACE WATER CHLORIDE, mg/L	1	D							
	1	W							
	2	D	43(59.06,26.94)	150(186.35,113.65)	145(172.83,117.17)	68(73.24,62.76)	34(45.38,22.62)	62.5(80.17,44.83)	
	2	W	17.5(36.7,-1.7)	80(99.65,60.35)	60(69.6,50.4)	47(55.13,38.87)	12(15.87,8.13)	16.5(20.74,12.26)	11(16.94,5.06)
SURFACE WATER SULFATE, mg/L	1	D	2(2.5,1.5)	44(54.47,33.53)	13(24.98,1.02)	5.4(9.79,1.01)	2(2.59,1.41)	2(2.39,1.61)	2(2.88,1.12)
	1	W	2(2.96,1.04)	27(34.89,19.11)	8.65(12.06,5.24)	5.7(8.62,2.78)	2(2.36,1.64)	2(2.52,1.48)	2(2.28,1.72)
	2	D	0.43(0.91,-0.05)	23(35.41,10.59)	56.5(67.07,45.93)	2.8(3.59,2.01)	0.17(0.31,0.03)	1.04(4.46,-2.38)	
	2	W	1.48(4.49,-1.53)	25(34.17,15.83)	26(41.93,10.07)	0.73(0.96,0.49)	7.4(9.68,5.12)	0.72(1.08,0.36)	0.26(0.45,0.07)
SURFACE WATER SULFIDE, mg/L	1	D	0.07(0.14,0)	0.4(0.71,0.09)	0.04(0.09,-0.02)	0.03(0.04,0.01)	0.02(0.04,-0.01)	0.01(0.02,0.01)	0.01(0.01,0.01)
	1	W	0.02(0.03,0)	0.01(0.03,-0.01)	0.01(0.02,0)	0.01(0.02,0)	0.01(0.01,0.01)	0.01(0.01,0.01)	0.01(0.01,0.01)
	2	D	0.06(0.07,0.04)	0.21(0.66,-0.25)	0.05(0.06,0.04)	0.05(0.12,-0.01)	0.04(0.05,0.03)	0.04(0.06,0.02)	
	2	W	0.01(0.01,0.01)	0.01(0.01,0.01)	0.01(0.01,0.01)	0.01(0.01,0.01)	0.01(0.01,0.01)	0.01(0.01,0)	0(0,0)
SURFACE WATER TOC, mg/L	1	D	25.79(31.32,20.26)	38.61(43.24,33.98)	29.51(36.06,22.95)	26.54(28.55,24.53)	22.65(25.26,20.04)	26.65(29.23,24.06)	14.77(18.41,11.12)
	1	W	17.9(20.76,15.04)	31.72(37.28,26.16)	23.65(25.42,21.87)	19.23(20.64,17.81)	14.41(15.85,12.96)	14.65(16.24,13.06)	8.7(9.36,8.04)
	2	D	28.23(36.01,20.45)	37.14(41.12,33.16)	45.98(58.29,33.67)	26.2(30.47,21.93)	22.62(25.91,19.33)	28.85(34.53,23.16)	
	2	W	22.47(27.59,17.34)	27.75(29.61,25.89)	27.47(32.69,22.25)	18.51(19.15,17.87)	13.46(15.11,11.81)	11.17(12.34,10)	8.64(9.52,7.76)
SURFACE WATER TOTAL P, ug/L	1	D	20.85(26.2,15.5)	24.1(32.72,15.48)	19.48(26.83,12.13)	17.9(25.57,10.23)	24.1(28.31,19.89)	18.79(25.78,11.79)	12.62(17.31,7.93)
	1	W	8.89(10.2,7.58)	13.12(17.14,9.1)	15.97(21.46,10.47)	8.53(12.92,4.13)	5.74(6.92,4.56)	6.2(7.86,4.54)	7.75(9.65,5.85)
	2	D	43.09(76.71,9.47)	22.09(37.77,6.41)	229.19(456.04,2.33)	11.19(13.03,9.35)	20.27(32.3,8.25)	15.96(19.26,12.66)	
	2	W	9.14(9.98,8.31)	9.51(10.58,8.44)	11.37(14.42,8.33)	6.21(6.95,5.46)	5.69(6.39,4.99)	4.89(5.15,4.62)	5.25(5.6,4.9)
SURFACE WATER TOTAL N, mg/L	1	D	1.87(2.29,1.45)	2.29(2.49,2.09)	2.11(2.59,1.62)	1.77(1.96,1.58)	1.75(1.93,1.57)	1.94(2.2,1.68)	1.3(1.75,0.85)
	1	W	1.2(1.35,1.05)	1.51(1.72,1.3)	1.25(1.36,1.14)	1.11(1.19,1.03)	0.94(1.01,0.86)	1.17(1.23,1.11)	0.78(0.9,0.66)
	2	D	1.5(1.92,1.08)	1.89(2.16,1.63)	6.22(10.84,1.6)	1.5(1.69,1.3)	2.02(2.47,1.58)	2.21(2.54,1.89)	
	2	W	1.22(1.45,0.99)	1.12(1.38,0.86)	0.93(1.13,0.74)	0.7(0.75,0.65)	0.52(0.58,0.46)	0.6(0.67,0.52)	0.32(0.36,0.27)
SURFACE WATER TOTAL MERCURY, ng/L	1	D	2.93(4,1.86)	1.69(2.72,0.66)	0.97(1.81,0.12)	1.84(2.35,1.33)	1.92(2.39,1.45)	2.26(2.57,1.95)	2.49(3.23,1.74)
	1	W	3.4(4.07,2.73)	2.35(2.72,1.98)	2.17(2.46,1.88)	2.01(2.27,1.74)	1.92(2.07,1.77)	1.66(1.78,1.54)	2.16(2.42,1.9)
	2	D	6.38(9.96,2.8)	3.14(3.85,2.43)	2.6(3.38,1.81)	2.35(3.46,1.24)	3.53(4.31,2.75)	2.93(3.76,2.1)	
	2	W	2.24(2.59,1.89)	2.26(2.63,1.89)	1.27(1.53,1.01)	1.18(1.45,0.91)	1.01(1.26,0.76)	1.18(1.37,0.98)	1.83(2.22,1.43)
SURFACE WATER METHYLMERCURY, ng/L	1	D	0.92(1.39,0.46)	0.85(1.31,0.4)	0.39(0.73,0.05)	0.56(0.72,0.4)	0.76(0.94,0.59)	0.42(0.54,0.31)	0.28(0.45,0.11)
	1	W	0.4(0.51,0.29)	0.43(0.54,0.33)	0.4(0.55,0.25)	0.31(0.36,0.27)	0.28(0.37,0.19)	0.2(0.24,0.17)	0.08(0.11,0.06)
	2	D	1.02(1.54,0.5)	0.97(1.38,0.55)	0.57(0.91,0.22)	0.7(0.93,0.47)	0.54(0.92,0.17)	0.67(1.41,-0.07)	
	2	W	0.4(0.79,0.01)	0.74(1.12,0.35)	0.24(0.37,0.12)	0.22(0.31,0.14)	0.19(0.26,0.12)	0.12(0.14,0.09)	0.06(0.09,0.04)
POREWATER SULFIDE, mg/L	1	D							
	1	W							
	2	D	0.12(0.23,0.01)	4.95(7.08,2.82)	0.27(0.52,0.02)	0.64(1.03,0.26)	0.09(0.12,0.06)	0.14(0.21,0.07)	
	2	W	0.11(0.23,-0.01)	1.02(1.99,0.04)	0.21(0.23,0.19)	0.27(0.59,-0.04)	0.08(0.11,0.04)	0.07(0.09,0.06)	0.05(0.06,0.03)
FLOC AFDW, %	1	D							
	1	W							
	2	D	94.8(95.85,93.75)	86.65(93.48,79.82)	84.58(84.58,84.58)	87.04(95.92,78.16)	90.4(92.72,88.08)	65.61(74.64,56.59)	
	2	W	91.55(93.19,89.92)	86.38(87.35,85.41)	86.94(89.55,84.33)	84.58(92.67,76.5)	91.86(93.16,90.56)	60.43(71.98,48.88)	
FLOC MINERAL CONTENT, %	1	D							
	1	W							
	2	D	5.2(6.25,4.15)	11.9(22.25,1.56)	15.42(15.42,15.42)	12.96(21.84,4.08)	9.6(11.92,7.28)	34.39(43.41,25.36)	
	2	W	8.45(10.08,6.81)	14.8(17.14,12.46)	13.06(15.67,10.45)	15.42(23.5,7.33)	7.93(9.22,6.64)	39.57(51.12,28.02)	
FLOC TOTAL P, ug/kg	1	D							
	1	W							
	2	D	568.34(693.84,442.85)	263.42(307.92,218.92)	842(842,842)	455.09(547.83,362.36)	371.14(465.68,276.6)	213.52(320.22,106.81)	
	2	W	431.5(575.99,287)	454.26(661.08,247.44)	560.25(772.5,348)	317.38(391.99,242.77)	453.23(502.46,404.01)	239.62(319.86,159.38)	

Table 6.2. Continued.

			AREA						
			MEDIAN (95%CI)						
Parameter	Phase	Season	LOX	WCA2	WCA3-N	WCA3-SE	WCA3-SW	SHARK SLOUGH	TAYLOR SLOUGH
FLOC TOTAL MERCURY, ug/kg	1	D							
	1	W							
	2	D	175.02(406.58,-56.54)	323.57(349.55,297.59)	156.71(156.71,156.71)	200.26(245.81,154.71)	331.93(448.43,215.42)	99.55(156.7,42.4)	
	2	W	233.84(300.6,167.08)	313.43(405.01,221.85)	136.8(159.29,114.31)	103.56(127.9,79.22)	157.14(195.22,119.06)	85.79(113.24,58.34)	72.71(111.63,33.79)
FLOC METHYLMERCURY, ug/kg	1	D							
	1	W							
	2	D	4.98(8.1,1.86)	0.2(1.46,-1.06)	9.19(9.19,9.19)	0.2(0.2,0.2)	0.2(0.2,0.2)	1.31(2.37,0.25)	
	2	W	2.99(9.87,-3.89)	10.13(16.63,3.63)	7.32(13,1.63)	2.78(5.76,-0.2)	3.38(6.3,0.46)	0.74(1.08,0.4)	0.48(0.85,0.11)
SOIL DEPTH, m	1	D	2.65(2.89,2.41)	1.34(1.5,1.19)	0.46(0.57,0.34)	0.94(1.08,0.81)	0.82(0.97,0.67)	0.3(0.36,0.25)	0.27(0.4,0.15)
	1	W	2.65(2.92,2.38)	1.2(1.41,1)	0.37(0.47,0.26)	0.82(1.04,0.61)	0.81(0.96,0.66)	0.37(0.47,0.26)	0.24(0.34,0.15)
	2	D	2.93(3.17,2.69)	1.13(1.31,0.95)	0.44(0.6,0.29)	0.88(1.09,0.68)	0.73(0.91,0.55)	0.29(0.39,0.19)	0.35(0.47,0.24)
	2	W	3.08(3.63,2.52)	1.4(1.6,1.2)	0.4(0.48,0.32)	0.94(1.12,0.77)	1.08(1.36,0.81)	0.3(0.37,0.23)	0.26(0.38,0.14)
SOIL AFDW,%	1	D	93.49(94.39,92.59)	87.06(88.03,86.09)	65.67(83.03,48.31)	82.52(85.15,79.89)	88.01(91.51,84.51)	34.18(45.39,22.97)	21.03(27.54,14.51)
	1	W	94.08(95.14,93.02)	87.42(89.36,85.47)	62.34(76.9,47.78)	84.31(90,78.61)	89.41(93.77,85.04)	43.7(52.89,34.51)	16.93(22.66,11.2)
	2	D	97.13(98.26,96)	90.73(94.72,86.74)	78.13(101.61,54.65)	85.11(94.49,75.73)	90.59(92.81,88.36)	34.05(44.45,23.65)	25.49(33.31,17.67)
	2	W	93.98(95.46,92.49)	88.71(90.61,86.81)	76.38(86.71,66.05)	86.22(89.46,82.98)	89.86(92.14,87.58)	42.3(52.63,31.97)	35.33(40.56,30.1)
SOIL MINERAL CONTENT, %	1	D							
	1	W							
	2	D	3(3.55,2.45)	9.27(13.26,5.28)	21.87(45.35,-1.61)	14.89(24.27,5.51)	9.41(11.64,7.19)	65.95(76.35,55.55)	74.51(82.33,66.69)
	2	W	6.02(7.51,4.54)	11.29(13.19,9.39)	23.62(33.95,13.29)	13.78(17.02,10.54)	10.14(12.89,7.38)	57.7(68.03,47.37)	64.67(69.9,59.44)
SOILSUBSIDENCE/ ACCRETION, m	1	D	0.36(0.58,0.15)	-0.27(-0.07,-0.48)	-0.67(-0.51,-0.83)	-0.26(-0.13,-0.39)	-0.26(-0.12,-0.4)	0.04(0.14,-0.05)	0.47(0.81,0.13)
	1	W	0.35(0.67,0.03)	-0.20(-0.07,-0.33)	-0.75(-0.53,-0.97)	-0.15(0,-.30)	-0.04(0.11,-0.20)	-0.01(0.11,-0.14)	0.11(0.20,0.01)
	2	D							
	2	W							
SOIL REDOX, mV	1	D	184.93(216.9,152.96)	55.14(87.53,22.75)	131.24(153.99,108.49)	106.7(136.15,52.41)	114.13(188.82,70.80)	82.8(116.76,48.84)	167.3(206.29,128.31)
	1	W	150.64(176,125.28)	35.65(58.46,12.84)	87.1(106.54,67.66)	119.09(141.40,76.40)	143.69(164.11,132.89)	135.14(146.71,123.57)	146.67(160.26,133.08)
	2	D	195.8(227.16,164.44)	45.8(61.1,30.5)	172.4(212.91,131.89)	82.93(113.76,14.80)	138.73(158.30,119.26)	115.8(139.99,91.61)	(,)
	2	W	227.4(334.43,120.37)	83.9(309.40,48.80)	81(106.41,55.59)	48.33(162.05,-65.38)	151.7(163.65,139.75)	81(97.08,64.92)	114.2(133.85,94.55)
SOIL TOTAL P, mg/kg	1	D	314.19(336.29,292.09)	420.94(482.58,359.3)	494.8(594.98,394.62)	341.43(380.34,302.52)	421.8(462.08,381.52)	311.75(368.19,255.31)	254.4(344.33,164.46)
	1	W	265.85(291.07,240.63)	334.5(391.48,277.52)	395.03(460.56,329.5)	306.65(329.45,283.85)	389.29(418.14,360.44)	342.22(400.33,284.11)	169.3(238.25,100.35)
	2	D	198.49(245.51,151.47)	341.79(493.8,189.78)	288.08(387.29,188.87)	204.14(238.87,169.4)	249.35(306.9,191.8)	187.25(218.58,155.92)	148.83(239.25,58.41)
	2	W	234.16(278.02,190.3)	354.68(416.47,292.89)	361.95(454.34,269.56)	174.91(232.82,117)	268.44(301.38,235.5)	164.04(199.82,128.26)	107.41(148.75,66.07)
SOIL SULFATE, mg/kg	1	D	265(166.45,363.55)	290(-23.26,603.2)	170(-28,368)	250(195.5,304.5)	296(212.6,379.4)	96(58.90,133.1)	78.5(49.75,107.2)
	1	W	430(234.6,625.4)	350(266.1,433.9)	165(97.78,232.2)	230(132.3,327.7)	135(46.65,223.4)	130(93.12,166.9)	71(41.53,100.5)
	2	D	120(26.57,213.4)	2950(1768,4131)	3100(1458,4742)	2500(943.4,4056)	81(6.46,155.5)	160(-205.8,525.8)	71.5(-2.96,146.0)
	2	W	170(-199.6,539.6)	1600(650.8,2549)	1200(649.9,1750)	990(47.10,1933)	230(71.61,388.4)	215(26.10,403.9)	26(13.19,38.81)
SOIL TOTAL MERCURY, ug/kg	1	D	160(177.6,142.4)	165(175.56,154.44)	94(121.87,66.13)	130(145.15,114.85)	170(182.6,157.4)	85(106.59,63.41)	49(67.74,30.26)
	1	W	170(180.3,159.7)	175(187.58,162.42)	87.5(103.71,71.29)	120(134.46,105.54)	180(199.79,160.21)	100(119.11,80.89)	34(43.76,24.24)
	2	D	140(166.19,113.81)	130(144.77,115.23)	85(116.43,53.57)	97(112.28,81.72)	160(180.61,139.39)	69.5(92.88,46.12)	45.5(60.96,30.04)
	2	W	145(163.18,126.82)	150(176.19,123.81)	110(127.46,92.54)	130(142.19,117.81)	170(196.4,143.6)	100(125.95,74.05)	43.5(72.58,14.42)
SOIL METHYLMERCURY, ug/kg	1	D	1.96(3.31,0.6)	0.47(0.67,0.26)	0.88(1.28,0.47)	0.39(0.59,0.19)	0.43(0.58,0.27)	0.32(0.47,0.17)	0.22(0.35,0.09)
	1	W	1.13(1.6,0.66)	0.35(0.45,0.25)	0.52(0.85,0.18)	0.43(0.58,0.28)	0.58(0.7,0.46)	0.26(0.36,0.16)	0.1(0.15,0.05)
	2	D	5.03(6.98,3.07)	0.86(2.72,-1)	1.64(2.6,0.67)	0.42(0.67,0.16)	0.72(1.39,0.05)	0.22(0.32,0.12)	0.29(0.65,-0.07)
	2	W	4.79(8.63,0.94)	0.86(2.39,-0.67)	3.63(4.69,2.57)	0.33(0.52,0.14)	0.54(0.76,0.31)	0.16(0.27,0.04)	0.13(0.17,0.08)
PERIPHYTON AVG TOTAL MERCURY, ug/kg	1	D	81.87(109.85,53.88)	54.83(60.64,49.01)	62.52(71.06,53.98)	54.35(66.25,42.44)	65.63(76.91,54.34)	45.21(49.44,40.98)	33.38(43.95,22.81)
	1	W	352.6(555.73,149.46)	247.54(326.72,168.35)	209.67(261.69,157.65)	157.14(193.32,120.96)	189.75(229.96,149.54)	99.97(124.63,75.31)	42.81(54.71,30.91)
	2	D		60.33(83.97,36.69)	9.5(9.5,9.5)	36.23(70.45,2.02)	28.62(46.71,10.52)	32.41(48.71,16.11)	28.05(40.39,15.72)
	2	W	18.97(18.97,18.97)	45.51(62.52,28.5)	25.11(31.51,18.7)	27.86(38.99,16.73)	35.01(59.56,10.46)	31.8(40.07,23.53)	15.24(20.18,10.29)
PERIPHYTON AVG METHYLMERCURY, ug/kg	1	D	4.61(6.83,2.39)	3.26(4.48,2.04)	2.32(3.48,1.16)	2.75(3.61,1.89)	4.61(6.5,2.72)	1.82(2.19,1.44)	0.65(0.96,0.33)
	1	W	5.55(7.33,3.77)	2.43(3.84,1.02)	1.03(2,0.06)	1.17(1.65,0.69)	2.42(3.28,1.56)	1.28(1.62,0.94)	0.13(0.24,0.02)
	2	D	0.2(0.2,0.2)	1.97(2.95,0.98)	4.47(4.47,4.47)	0.57(1.15,-0.02)	3.4(4.83,1.97)	1.3(1.95,0.65)	0.2(0.62,-0.22)
	2	W	2.55(2.55,2.55)	2.38(3.59,1.17)	1.42(2.32,0.51)	2.01(2.82,1.2)	2.19(2.89,1.49)	0.92(1.5,0.34)	0.2(0.27,0.13)
CATTAIL TOTAL MERCURY, ug/kg	1	D							
	1	W							
	2	D	6.35(9.02,3.68)	0.78(2.92,-1.36)	0.8(0.91,0.69)	1.57(2.43,0.71)		1.84(1.84,1.84)	
	2	W							
SAWGRASS TOTAL MERCURY, ug/kg	1	D							
	1	W							
	2	D	3.97(5.85,2.09)	7.45(8.52,6.38)	13.21(15.5,10.93)	6.58(7.44,5.72)	8.3(9.42,7.17)	4.41(5.37,3.46)	7.34(8.75,5.93)
	2	W							
GAMBUSIA TOTAL MERCURY, ug/kg	1	D	135.12(176.83,93.41)	56.19(108.65,3.73)	99.58(136.08,63.09)	155.88(190.1,121.65)	272.33(312.64,232.02)	233.66(264.84,202.48)	155.65(333.87,-22.57)
	1	W	108.04(151.67,64.41)	87.69(114.91,60.48)	156.85(187.8,125.9)	95.28(151.68,38.88)	234.85(283.01,186.68)	182.22(223.36,141.08)	67.24(95.9,38.58)
	2	D	55.44(98.46,12.43)	57.07(91.12,23.01)		56.65(88.26,25.04)	107.67(132.33,83.02)	244.86(295.18,194.54)	
	2	W	94.63(134.42,54.83)	133.81(179.86,87.77)	114.13(168.32,59.95)	104.48(176.32,32.65)	172.48(203.74,141.23)	145.73(173.81,117.65)	80.65(109.42,51.88)
BAF	1	D	143821.84(175282.23,112361.45)	119342.11(188391.7,50292.51)	242318.19(362617.79,122018.59)	273563.07(407186.34,139939.79)	359773.64(432668.01,286879.26)	475302.33(588975.99,361628.66)	711623.38(955219.36,468027.39)
	1	W	294852.46(367878.21,221826.71)	229970.27(315443.26,144497.28)	231951.17(358101.97,105800.37)	333056.77(524153.94,141959.59)	760018.48(941205.07,578831.9)	902763.16(1007938.78,797587.54)	826666.67(1153609.73,499723.6)
	2	D	121720.99(269901.76,-26459.78)	60984.88(98364.63,23605.12)		75761.28(159044.94,-7522.38)	197931.16(298185.39,97676.93)	366142.29(858304.9,-126020.31)	
	2	W	272080.17(366079.86,178080.48)	187219.05(289216.58,85221.52)	465848.4(570334,361362.79)	912682.29(1224660.75,600703.83)	1260578.66(1957370.07,563787.25)	985138.89(1166410.96,803666.83)	943233.77(1465892.12,420575.42)

Table 6.3. Comparisons of Phase I and II median wet season parameters measured on a landscape scale by subareas in the flow path. Subareas are designated by Lox=1, WCA2=2, WCA3-N=3, WCA3-SE=4, WCA3-SW= 5, Shark Slough=6 and Taylor Slough=7.

CONSTITUENT (Units)	Median		Gradient	Direction (High=>Low	Subareas Included	Phase	Significant Difference	p-value
	High	Low						
Surface Water								
pH (su)	7.88	6.54	Yes	S => N	1 - 7	I < II	Yes	0.001
Conductivity (uS/cm)	684	243	Yes	N => S	2 - 7	I = II	No	0.97
Chloride (mg/L)	80	11	Yes	N => S	2 - 7	II Only		
Sulfate (mg/L)	27	0.26	Yes	N => S	2 - 7	I = II	No	0.28
Sulfide (mg/L)	0.01	0.00	Yes	N => S	2 - 7	II Only		
Total Organic Carbon (mg/L)	31.7	8.64	Yes	N => S	2 - 7	I = II	No	0.99
Total Phosphorus (ug/L)	15.97	4.89	Yes	N => S	3 - 7	I > II	Yes	0.004
Total Nitrogen (mg/L)	1.5	0.32	Yes	N => S	1 - 7	I > II	Yes	0.000
Total Mercury (ng/L)	3.4	1.01	No	N & S	1 - 7	I > II	Yes	0.000
Methyl Mercury (ng/L)	0.74	0.08	Yes	N => S	2 - 7	I > II	Yes	0.020
Porewater								
Sulfide (mg/L)	1.02	0.05	Yes	N => S	2 - 7	II Only		

Table 6.3. Continued.

CONSTITUENT (Units)	Median		Gradient	Direction (High=>Low	Subareas Included	Phase	Significant Difference	p-value
	High	Low						
Floc								
AFDW (%)	91.86	60.43	Yes	N => S	5 - 6	II Only		
Mineral Content (%)	39.57	7.93	Yes	S => N	5 - 6	II Only		
Total Phosphorus (ug/g)	560.2	239.6	Yes	N => S	3 - 6	II Only		
Total Mercury (ug/kg)	313.4	72.7	Yes	N => S	1 - 7	II Only		
Methyl Mercury (ug/kg)	10.13	0.48	Yes	N => S	1 - 7	II Only		
Soil								
Depth (ft)	3.08	0.24	Yes	N => S	1 - 7	I = II	No	0.089
Subsidence/Accretion (m)	1.2	-2.45	No	N & S	1 - 7	I Only		
AFDW (%)	94	16.9	Yes	N => S	1 - 7	I < II	Yes	0.00
Mineral Content (%)	64.7	6.02	Yes	S => N	1 - 7		Yes	
Redox (mV)	35.65	227.4	Yes	S => N	2 - 7	I = II	No	0.782
Total Phosphorus (mg/kg)	395	107.4	Yes	N => S	2 - 7	I > II	Yes	0.000
Total Sulfate	3100	71	Yes	N => S	2 - 7	1 < 11	Yes	0.000

Table 6.3. Continued.

CONSTITUENT (Units)	Median		Gradient	Direction (High=>Low	Subareas Included	Phase	Significant Difference	p-value
	High	Low						
Total Mercury (ug/kg)	180	34	Yes	N => S	5 - 7	I = II	No	0.203
Methyl Mercury (ug/kg)	4.79	0.1	Yes	N => S	1 - 7	I < II	Yes	0.00
Tissue								
Mean Periphyton Total Mercury (ug/kg)	352.6	15.24	Yes	N => S	2 - 7	I > II	Yes	0.000
Mean Periphyton Methyl Mercury (ug/kg)	5.55	0.13	Yes	N => S	5 - 7	I = II	No	0.64
Cattail Total Mercury (ug/kg)	6.35	0.8	No		2 - 4	II Only		
Sawgrass Total Mercury (ug/kg)	13.2	3.97	Yes	N => S	3 - 7	II Only		
Mosquitofish Total Mercury (ug/kg)	?	?	Yes	S => N	2 - 5	I = II	No	0.44
Bioaccumulation Factor	1.260k	187k	Yes	S => N	1 - 7	I < II	Yes	0.04

Table 6.4. Summary of mosquitofish gut contents are reported by sampling period. N indicates sample size. The average proportion of each food category relative to total contents (wet weight) is reported for the 6 food categories. Summary indicates the total number of specimens or study sites examined, or the average proportion or trophic value for the entire data set.

	1996	1999			
	September	March	May	September	Summary
N individual fish	1,195	61	343	1,185	2,784
N sites	102	5	32	120	259
Cladocera	0.025	0.013	0.148	0.100	0.075
Mites	0.048	0.012	0.020	0.013	0.028
Adult diptera	0.323	0.507	0.264	0.261	0.290
Midge larvae and pupae	0.092	0.022	0.112	0.010	0.055
Detritus/plant matter	0.337	0.231	0.191	0.194	0.251
Other prey items	0.175	0.215	0.265	0.423	0.301
Niche Breadth	2.114	2.014	3.131	2.248	2.303
Trophic Position	2.086	2.206	2.262	2.255	2.188

Table 6.5. Matrix of Pearson correlation coefficients between food categories and trophic score. The asterisks indicate correlations significant at the P=0.05 level from Bonferoni corrected tables.

	Cladocera	Mites	Adult Diptera	Midge larvae	Detritus/plant matter	Other animal prey
Mites	-0.057					
Adult Diptera	-0.253*	-0.028				
Midge larvae	0.018	-0.018	-0.153			
Detritus/plant matter	-0.205*	-0.083	-0.458*	-0.108		
Other animal prey	-0.173	-0.164	-0.247*	-0.228*	-0.426*	
Trophic score	0.195*	0.195*	0.447*	0.104	-0.994*	0.401*

Table 6.6. Regression analyses of the relationship of relative abundance of dietary components to conductivity where mosquitofish were collected. Two analyses yielded significant non-linear relationship, which is indicated by row with $Cond^2$ to indicate second parameter in the model. All results were validated with logistic regression.

	Coefficient	Standard error	t	P	R^2
Cladocera	-0.041	0.020	-2.025	0.044	0.023
Mites	NS				
Adult diptera	Cond 0.070 $Cond^2$ -0.001	0.026 0.001	2.659 -3.270	0.008 0.001	0.047
Midge larvae	NS				
Other animal prey	Cond -0.047 $Cond^2$ 0.001	0.023 0.001	-2.028 2.611	0.045 0.010	0.033
Detritus/plant matter	0.001	0.001	2.492	0.013	0.048

Table 6.7. Analysis of mercury concentration in the tissues of mosquitofish. The full model explains 33.8% of the total variation in tissue mercury concentration. Total sample size in this analysis was 152. CD is the coefficient of determination for each factor in the model. Note that these sum to a larger total than explained by the full model because of multicollinearity in the model parameters.

	Coefficient	Standard Error	t	P	CD
Periphyton meHG	0.350	0.042	8.345	<0.001	28.2
Conductivity	-0.001	0.001	-4.084	<0.001	6.9
Cladocera	-0.532	-0.305	-1.747	0.083	0.9

Table 6.8. Mercury mass estimate models.

Water: $Mass_w = \dfrac{k \cdot A \cdot \sum_{i=1}^{n} \frac{Z_i \cdot d_i}{\pi_i}}{\sum_{i=1}^{n} \frac{1}{\pi_i}}$

Soil: $Mass_s = \dfrac{k \cdot A \cdot \sum_{i=1}^{n} \frac{Z_i \cdot s_i \cdot 0.1}{\pi_i}}{\sum_{i=1}^{n} \frac{1}{\pi_i}}$

Floc: $Mass_{fc} = \dfrac{k \cdot A \cdot \sum_{i=1}^{n} \frac{Z_i \cdot f_i \cdot d_i \cdot P}{\pi_i}}{\sum_{i=1}^{n} \frac{1}{\pi_i}}$

Periphyton: $Mass_p = \dfrac{k \cdot A \cdot \sum_{i=1}^{n} \frac{Z_i \cdot M_i}{\pi_i}}{\sum_{i=1}^{n} \frac{1}{\pi_i}}$

Fish: $Mass_f = \dfrac{k \cdot A \cdot \sum_{i=1}^{n} \frac{Z_i \cdot N_i}{\pi_i}}{\sum_{i=1}^{n} \frac{1}{\pi_i}}$

A=area of study region, km^2
Z=concentration Hg at a sample site
d=water depth at a sample site, m
π=sampling design inclusion probability
k=constant used to convert to appropriate units
f=floc bulk density at sample site, g/cc
p=floc thickness as a proportion of water depth, 0.01 to 0.1
s=soil bulk density at a sample site, g/cc
0.1soil depth, m
M=density of periphyton, g/m^2 (Trexler personal communication)
N=fish/m^2*Average fish weight for fish, g/fish (Trexler personal communication

Table 6.9 Everglades ecosystem total mercury mass estimates (kg).

	Cycle 0 (1995 Dry)	Cycle 1 (1995 Wet)	Cycle 2 (1996 Dry)	Cycle 3 (1996 Wet)	Cycle 4 (1999 Dry)	Cycle 5 (1999 Wet)
Input						
Precipitation	37.6	115.7	36.7	79.4	38.0	108.5
Sinks						
Water	2.944	9.038	3.435	5.550	2.288	5.191
Soil	10912.488	11895.558	11652.993	10561.007	8134.795	9848.237
Floc	0.000	0.000	0.000	0.000	166.572	1163.391
Periphyton	78.536	0.000	90.910	227.492	30.663	22.753
Fish	0.442	0.832	0.244	0.571	0.061	0.697
TOTAL	**10,994**	**11,905**	**11,748**	**10,795**	**8,334**	**11,040**

Table 6.10. Everglades ecosystem methyl mercury mass estimates (kg).

Sinks	Cycle 0 (1995 Dry)	Cycle 1 (1995 Wet)	Cycle 2 (1996 Dry)	Cycle 3 (1996 Wet)	Cycle 4 (1999 Dry)	Cycle 5 (1999 Wet)
Water	1.659	1.845	1.107	0.929	0.584	0.962
Soil	63.999	58.457	75.387	39.177	120.073	131.389
Floc	0.000	0.000	0.000	0.000	1.696	16.395
Periphyton	5.708	1.991	2.779	2.108	1.317	1.202
Fish	0.442	0.832	0.244	0.571	0.061	0.697
TOTAL	**72**	**63**	**80**	**43**	**123**	**151**

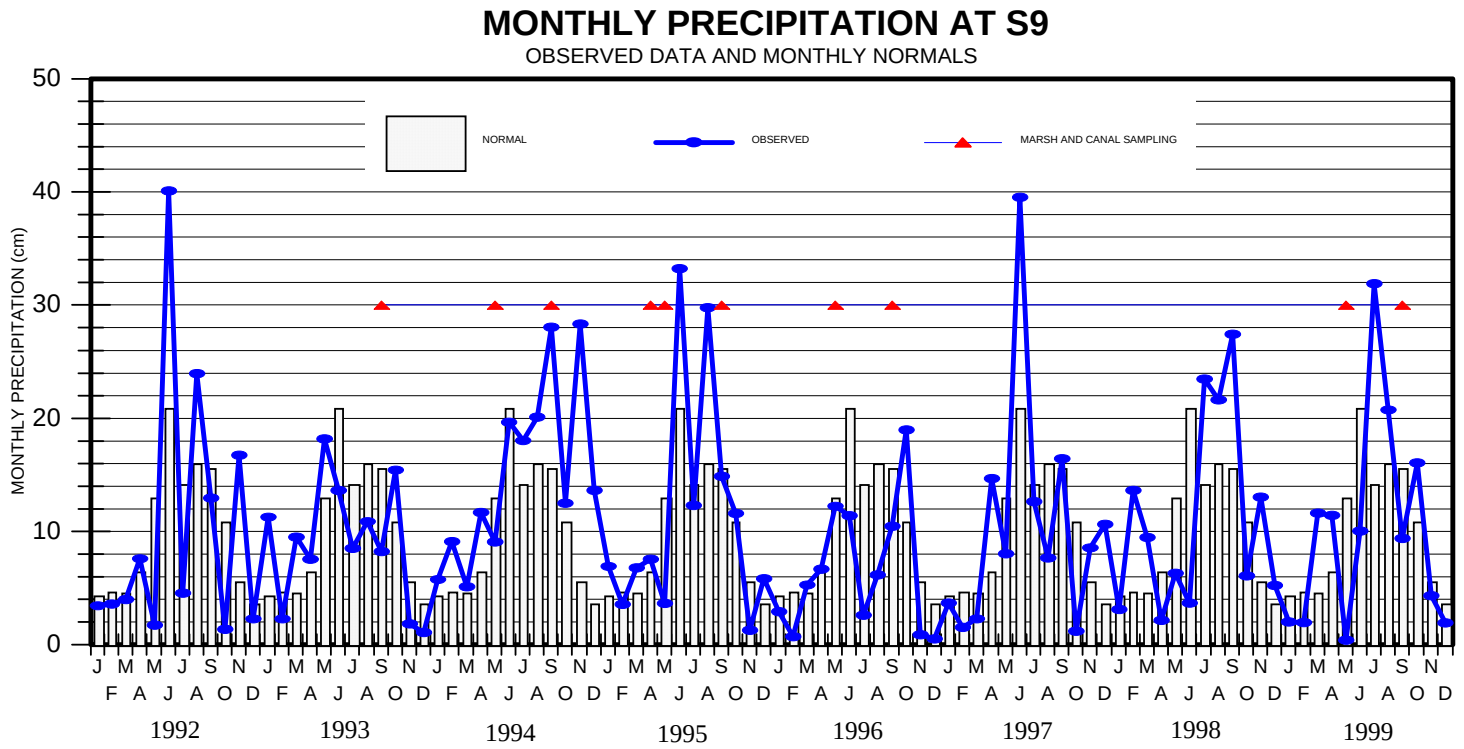


Figure 6.1. Comparison of monthly precipitation during the study period to normal monthly precipitation over the period of record at precipitation Station S9, with sampling periods indicated.

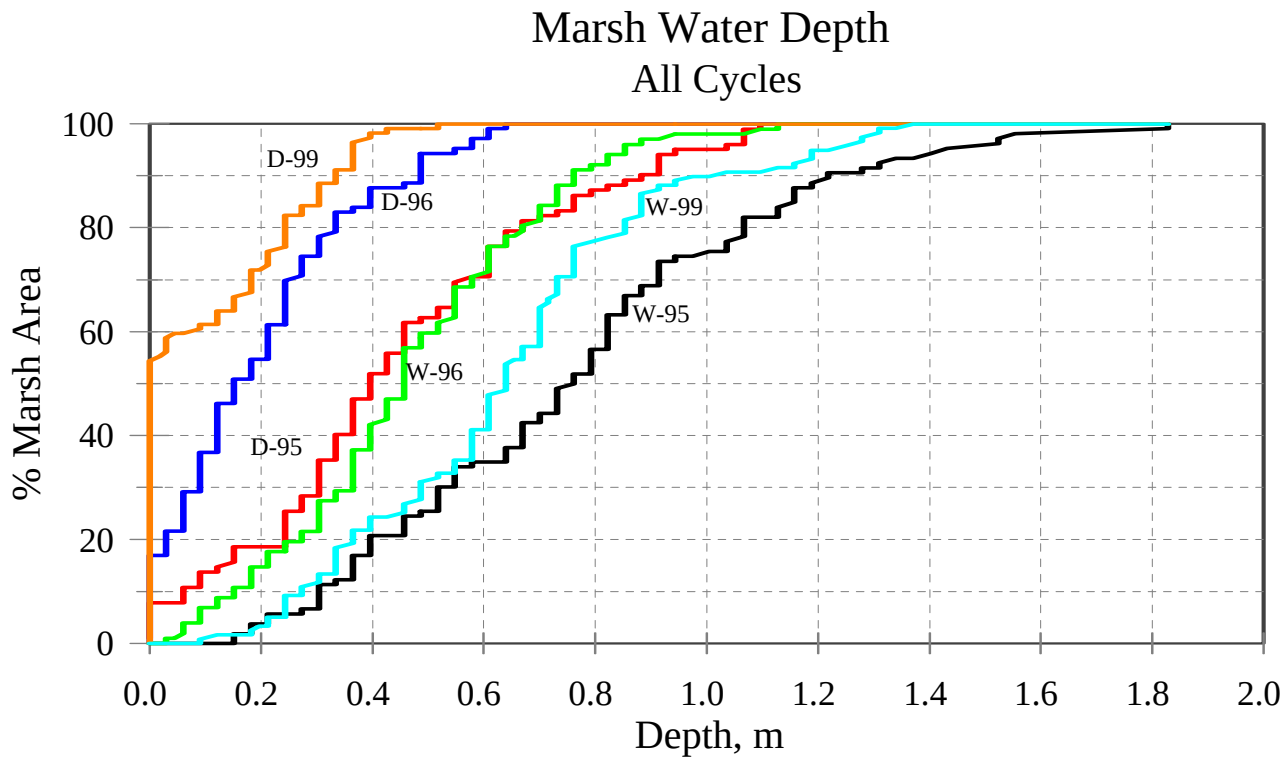


Figure 6.2. Cumulative distributions of water depths during sampling.

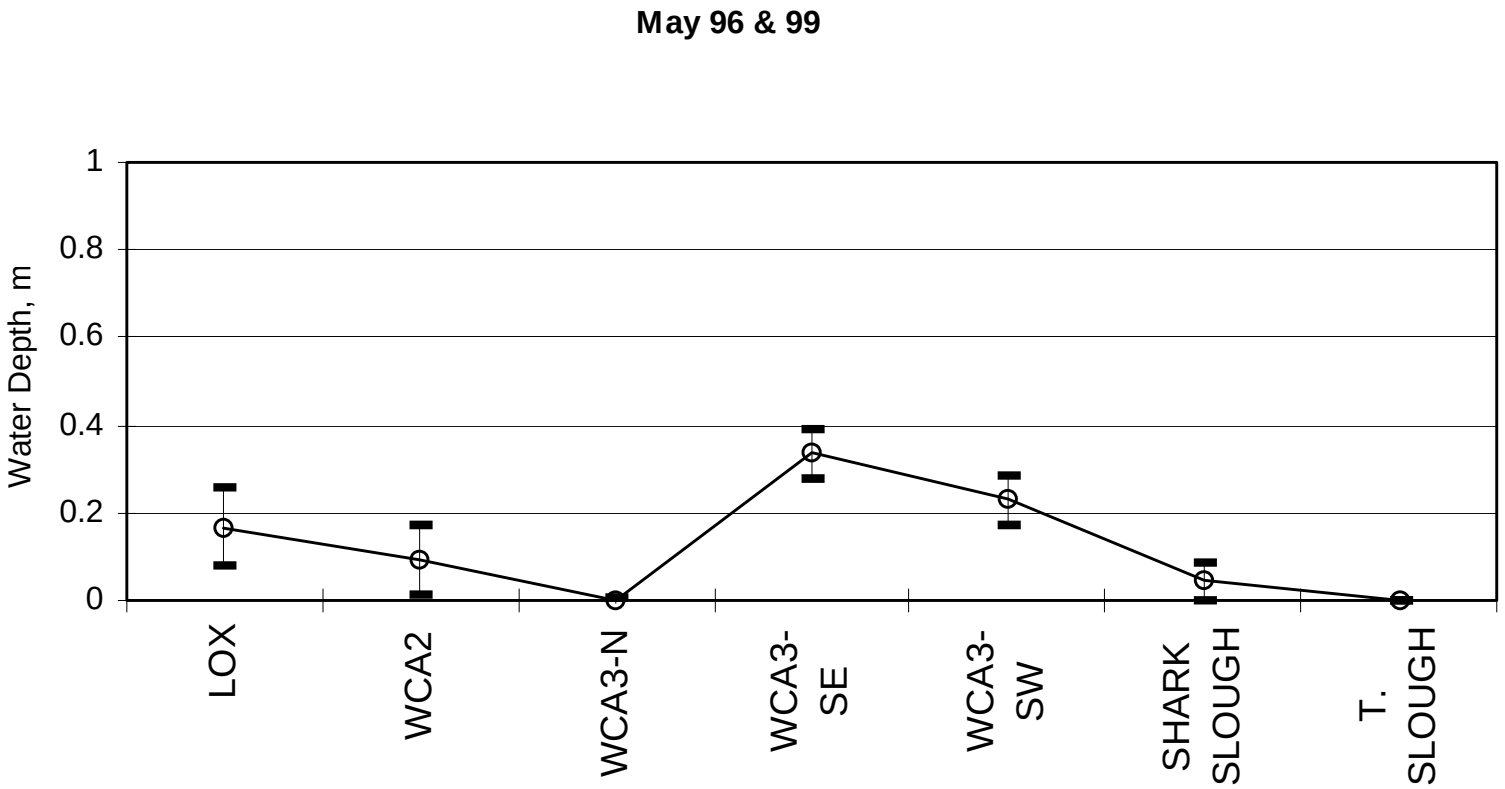


Figure 6.3. Median water depth measured in subareas during May 1996 and 1999 with 95% confidence interval.

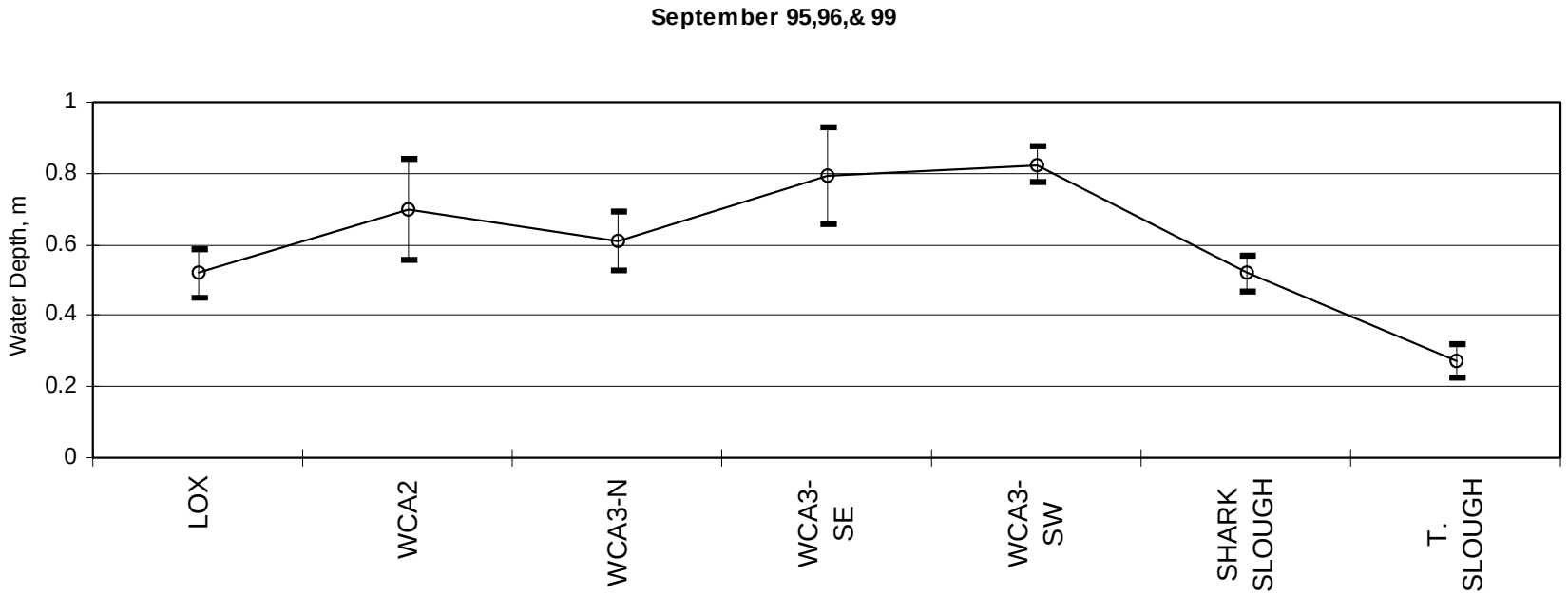


Figure 6.4. Median water depth measured in subareas during September 1995, 1996 and 1999 with 95% confidence interval.

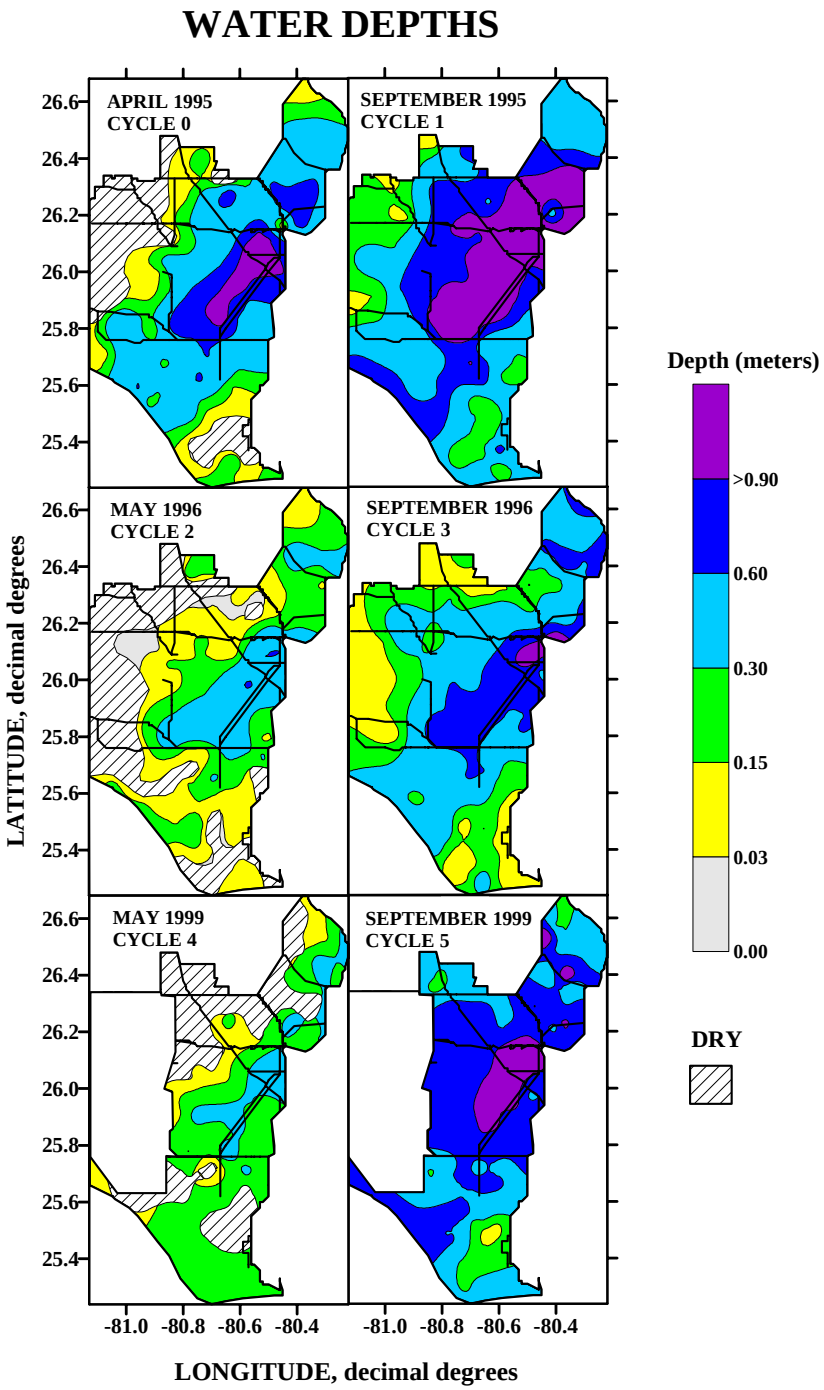


Figure 6.5. Surface plots of water depth measured during sampling.

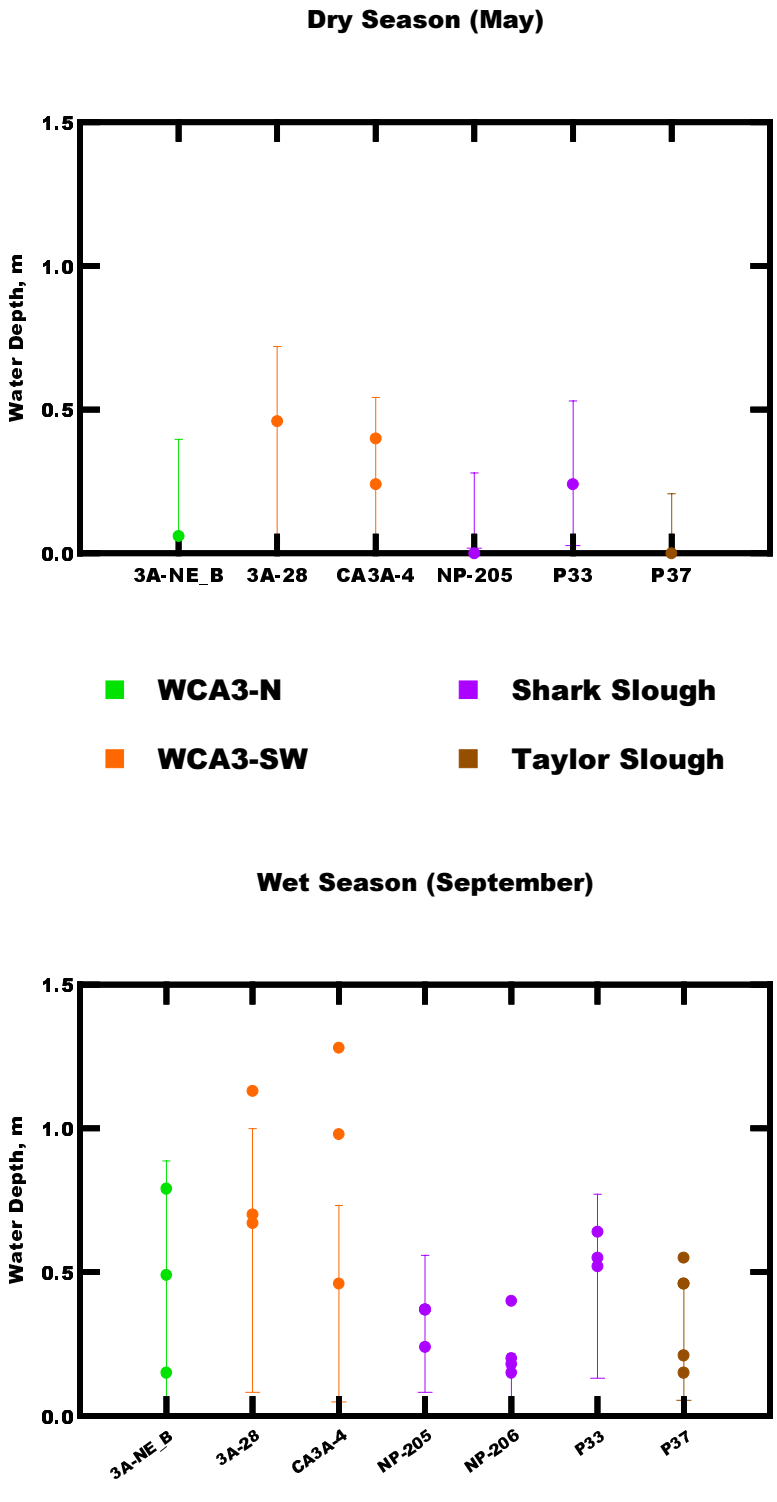


Figure 6.6. Comparison of historical ranges of water depths at SFWMD stage stations to water depths measured during sampling.

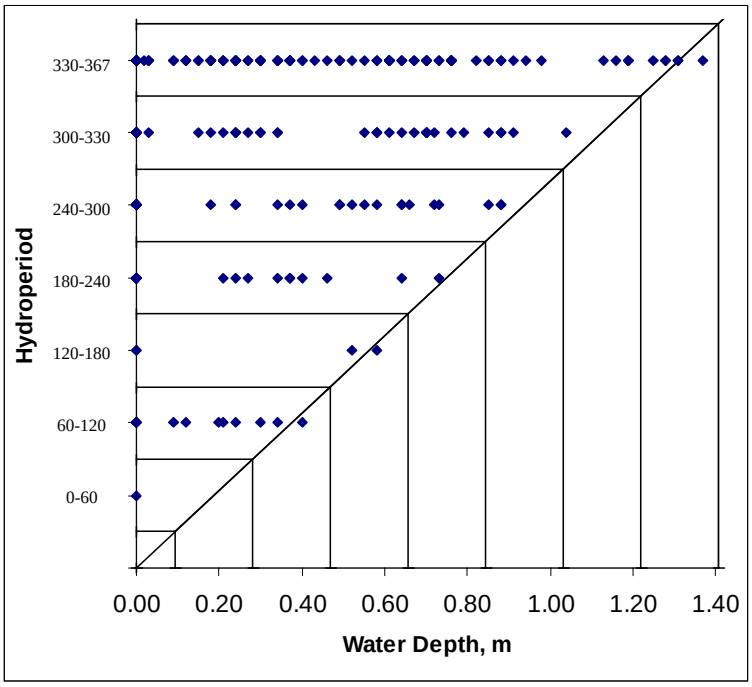
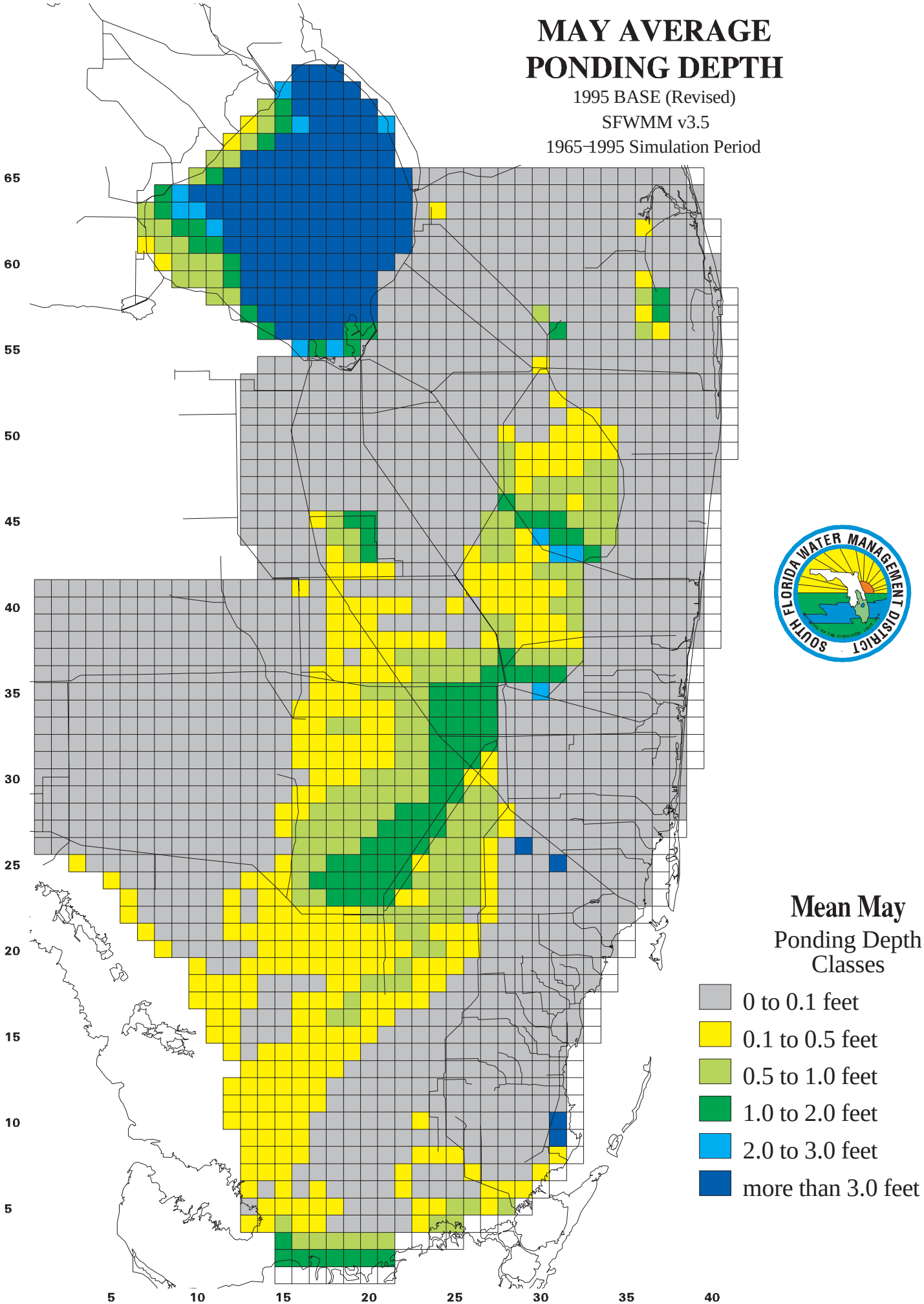


Figure 6.7. Water depths measured during phase 2 associated with SFWMM hydroperiod ponding classes.

MAY AVERAGE PONDING DEPTH

1995 BASE (Revised)
SFWMM v3.5
1965-1995 Simulation Period



SOUTH FLORIDA WATER MANAGEMENT DISTRICT

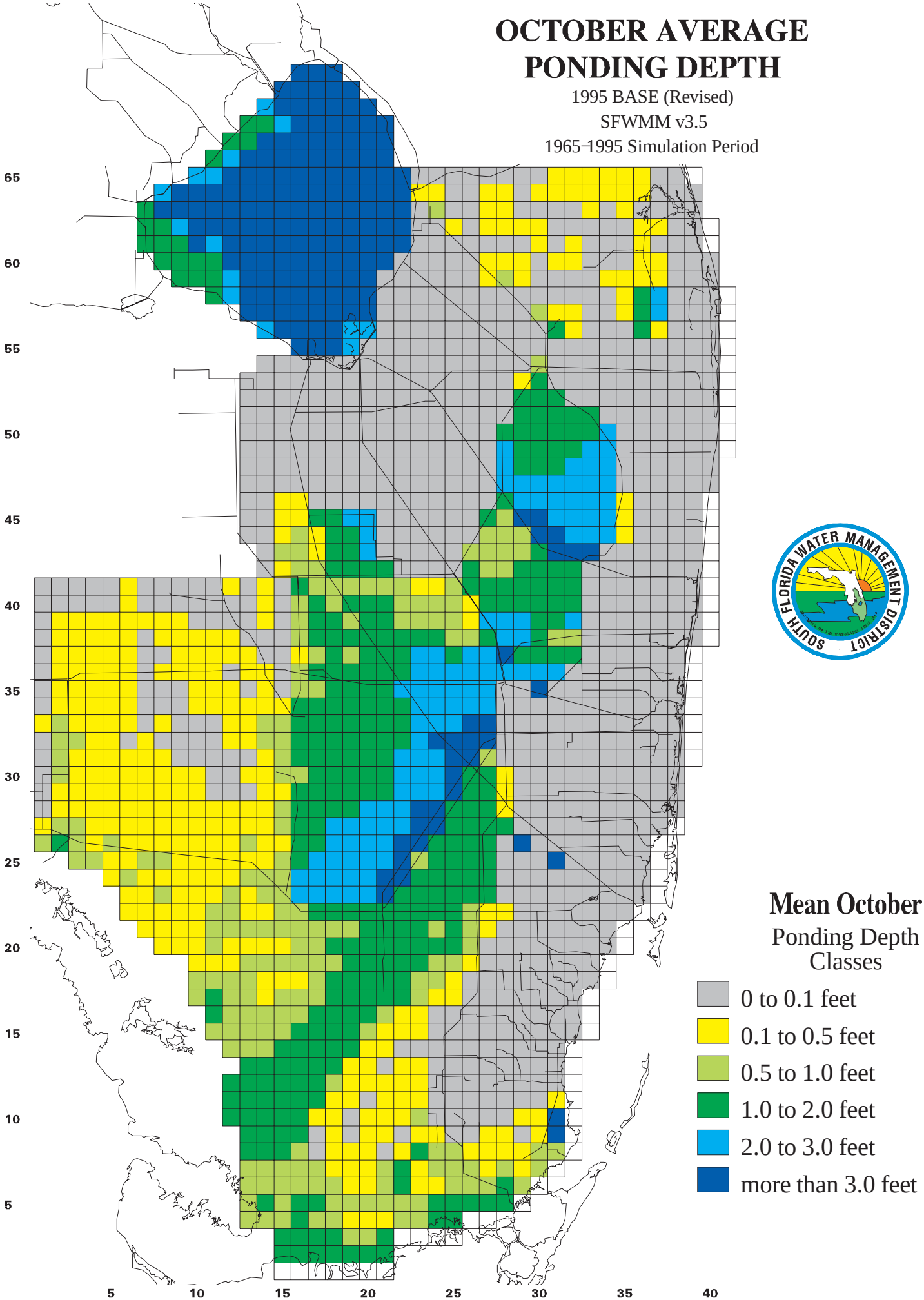
Mean May
Ponding Depth
Classes

- 0 to 0.1 feet
- 0.1 to 0.5 feet
- 0.5 to 1.0 feet
- 1.0 to 2.0 feet
- 2.0 to 3.0 feet
- more than 3.0 feet

27 Apr 98 09:20:33 Monday

OCTOBER AVERAGE PONDING DEPTH

1995 BASE (Revised)
SFWMM v3.5
1965-1995 Simulation Period



SOUTH FLORIDA WATER MANAGEMENT DISTRICT

Mean October
Ponding Depth
Classes

- 0 to 0.1 feet
- 0.1 to 0.5 feet
- 0.5 to 1.0 feet
- 1.0 to 2.0 feet
- 2.0 to 3.0 feet
- more than 3.0 feet

27 Apr 98 09:20:33 Monday

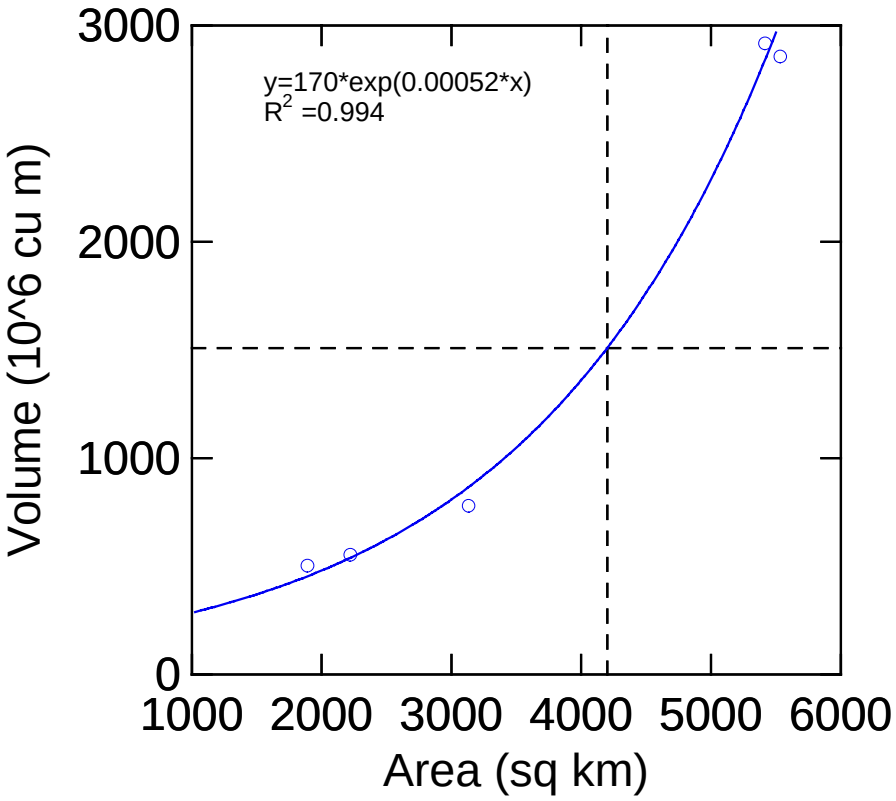


Figure 6.10. Surface water volume to surface area of inundation from the Everglades ecosystem.

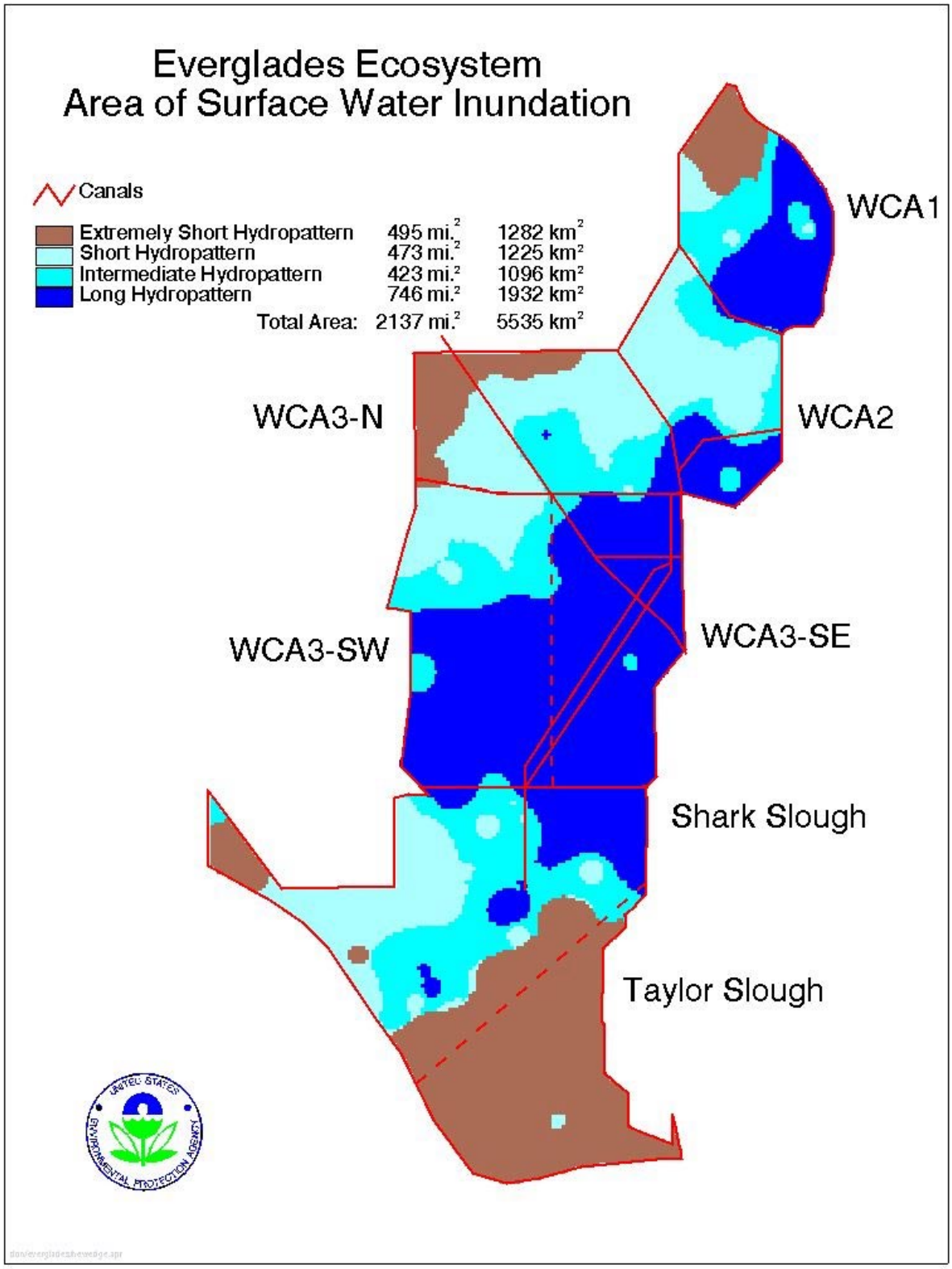


Figure 6.11. Everglades ecosystem area of surface water inundation.

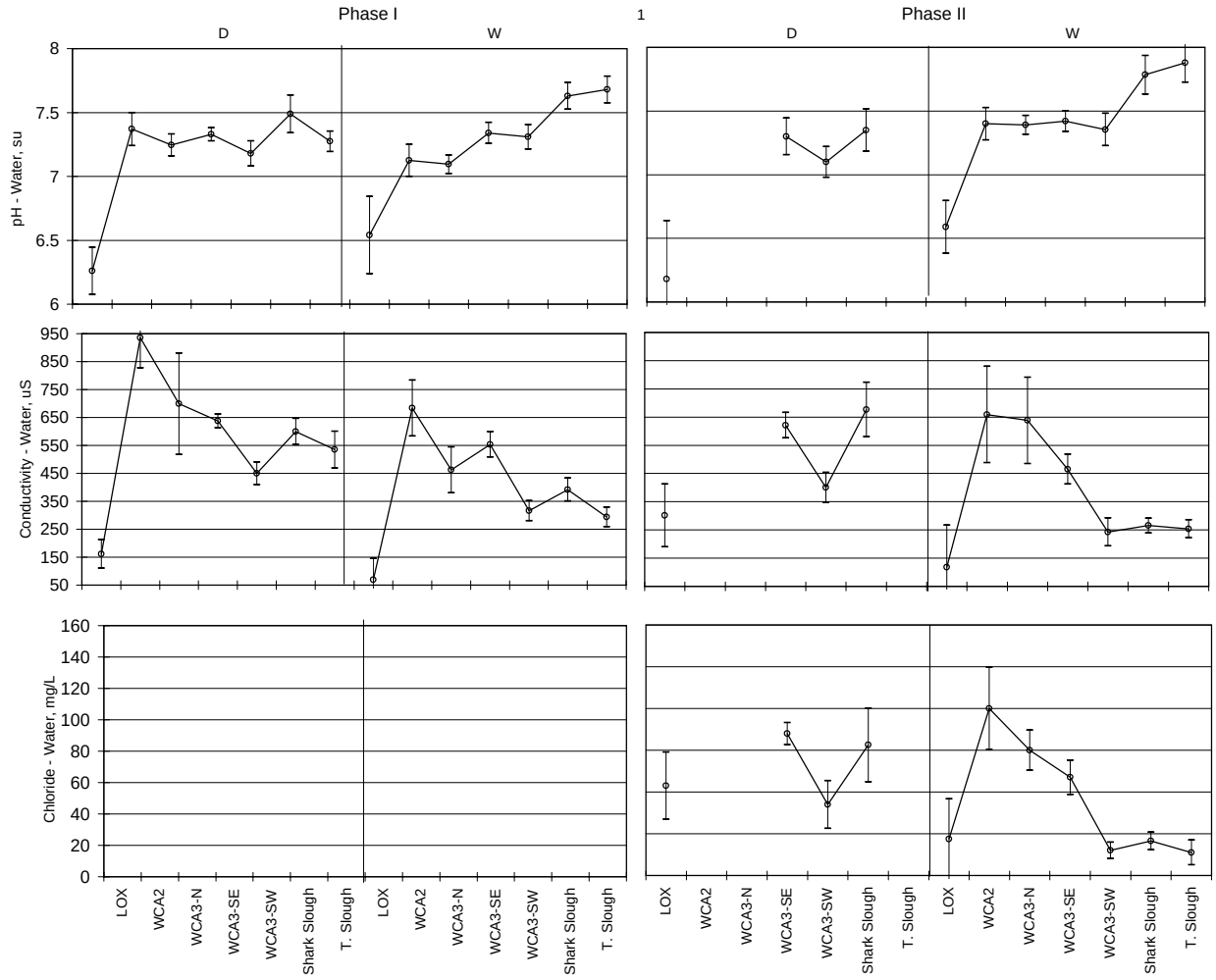


Figure 6.12. Median pH, conductivity, and chloride measurements (with 95% confidence interval) in each of the subareas during wet and dry seasons of phase 1 and phase 2.

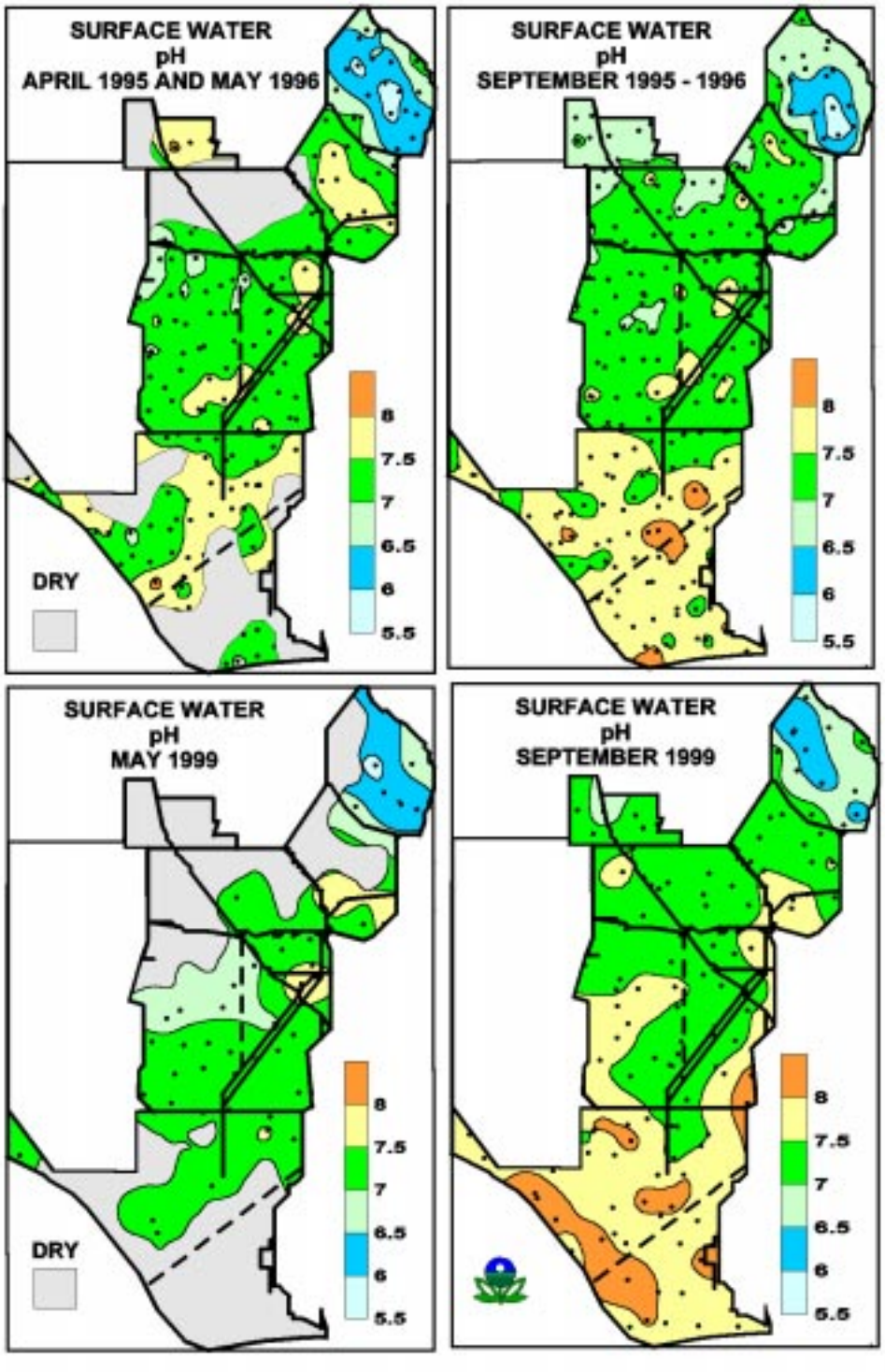


Figure 6.13. Surface plots of pH measured during wet and dry seasons of phase 1 and phase 2.

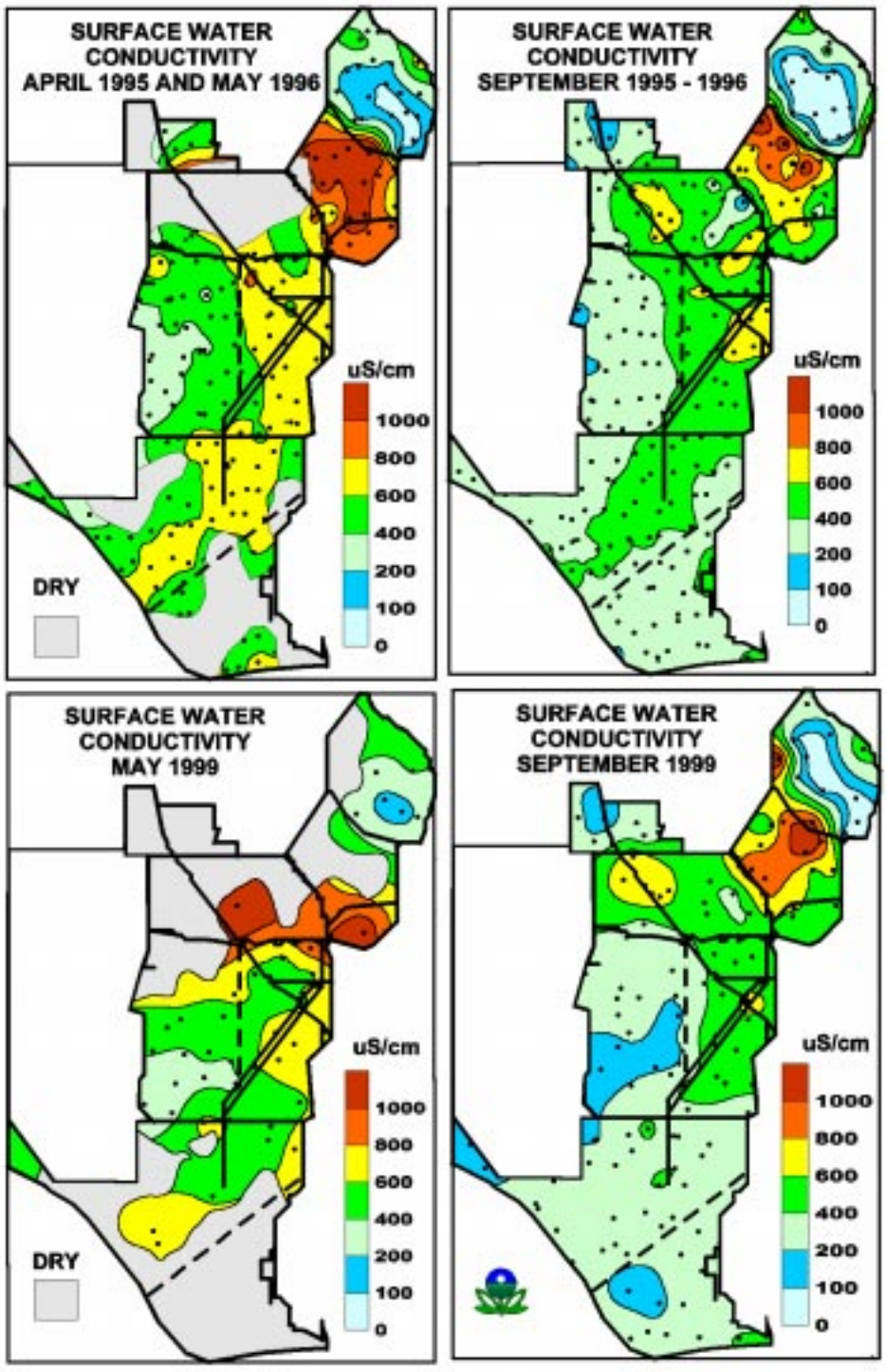


Figure 6.14. Surface plots of conductivity measured during wet and dry seasons of phase 1 and phase 2.

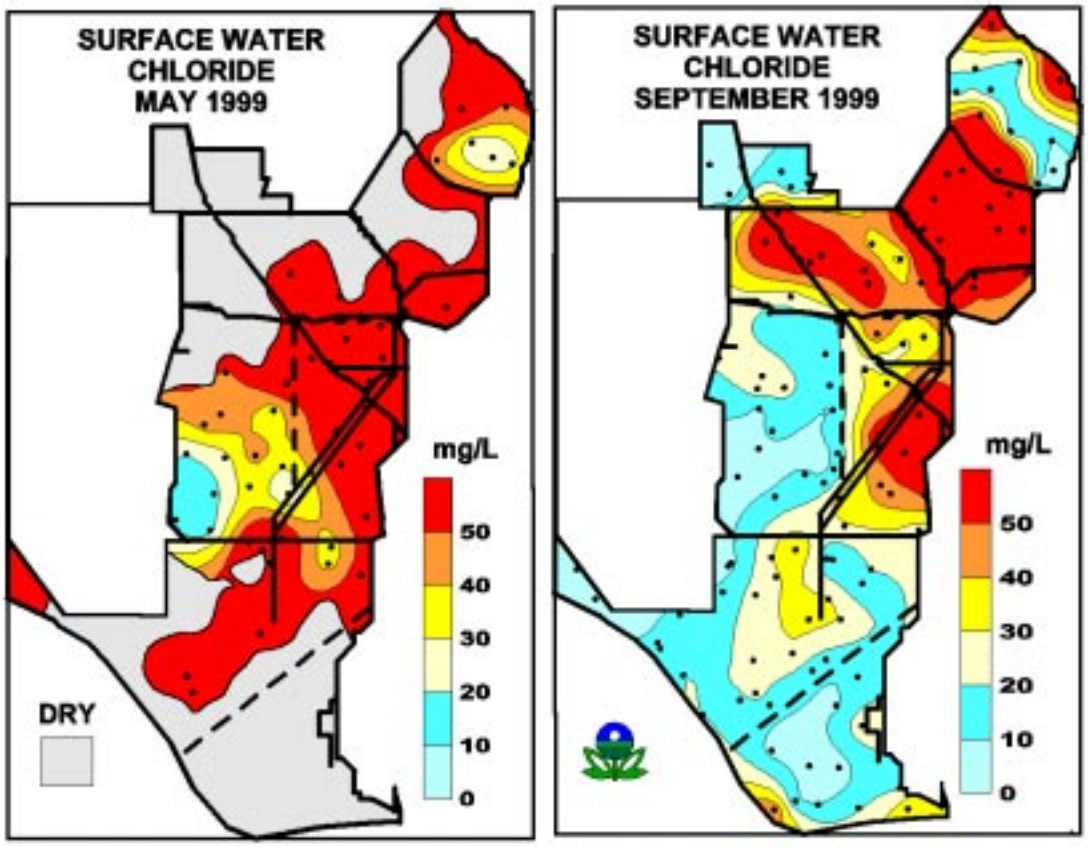


Figure 6.15. Surface plots of chloride measured during phase 2 wet and dry seasons.

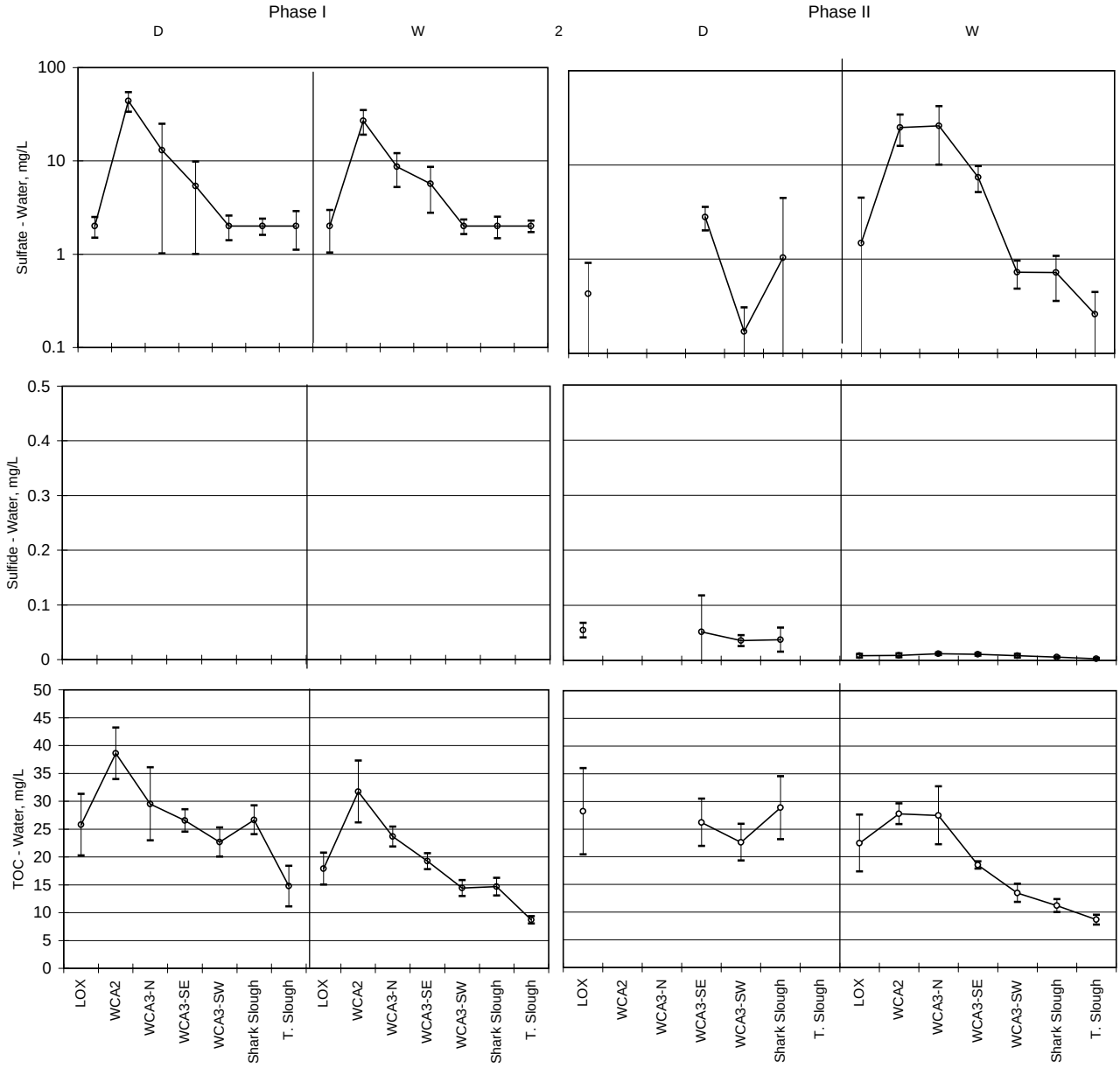


Figure 6.16. Median sulfate, sulfide, and TOC measured in surface water (with 95% confidence intervals) in the subareas during wet and dry seasons of phase 1 and phase 2.

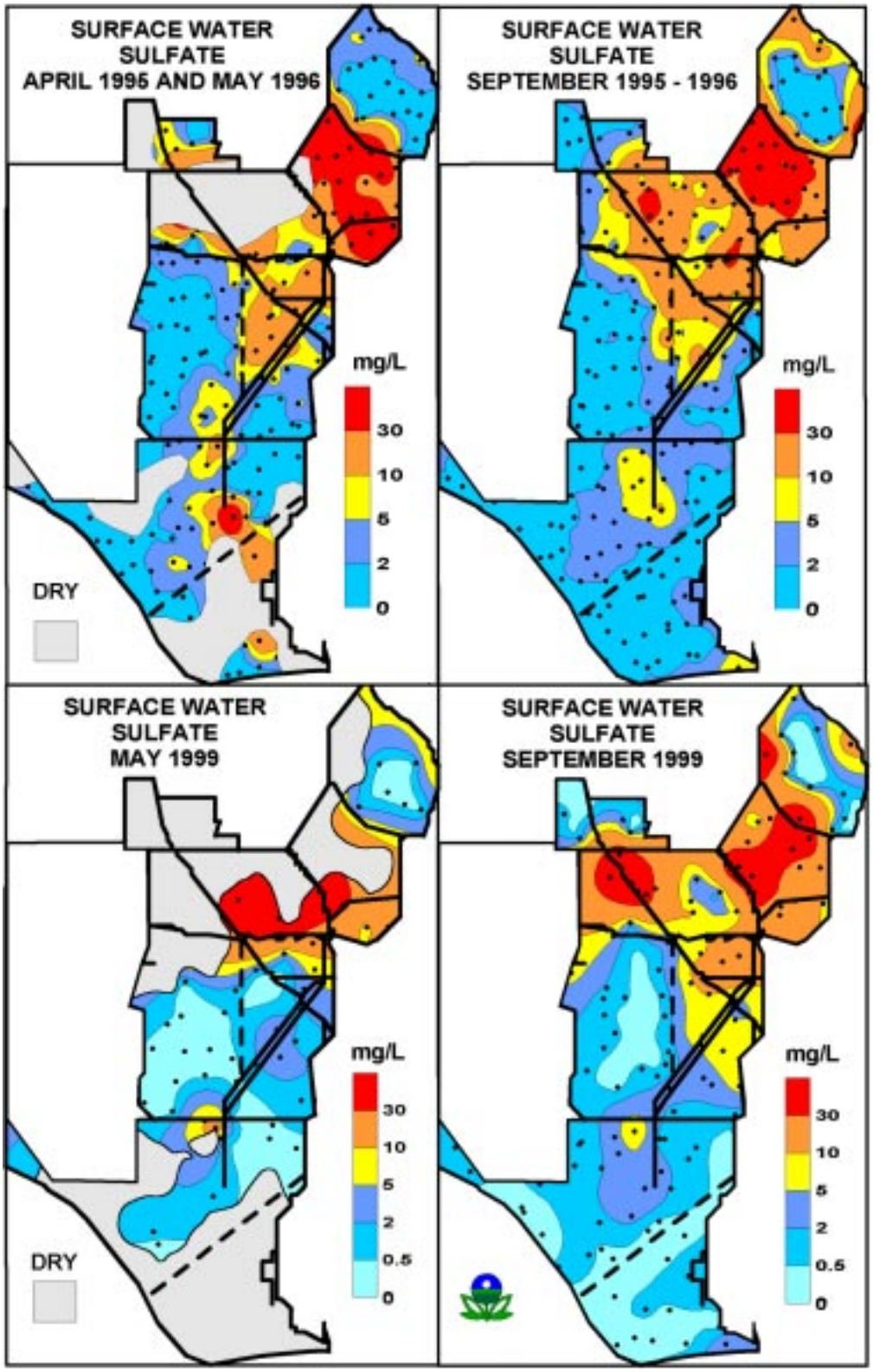


Figure 6.17. Surface plots of sulfate measured during wet and dry seasons of phases 1 and 2.

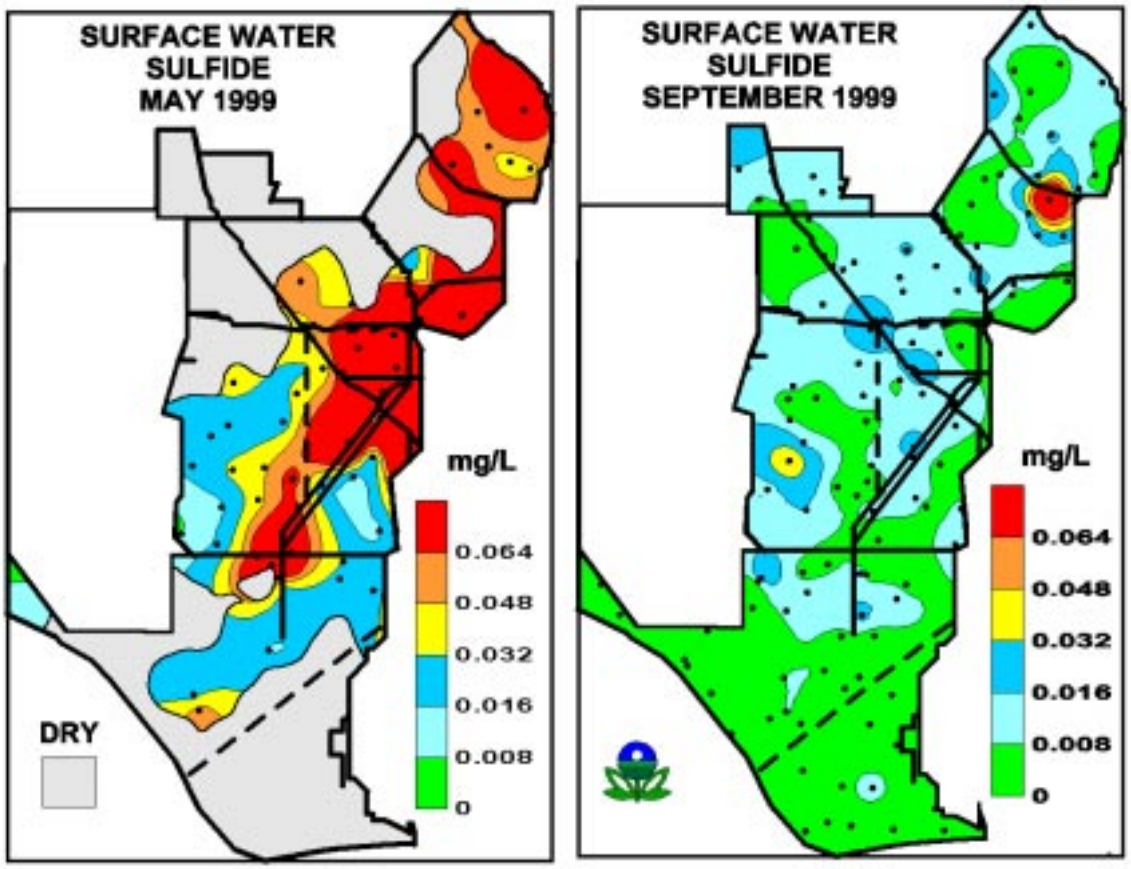


Figure 6.18. Surface plots of sulfide measured in surface water during wet and dry seasons in phase 2.

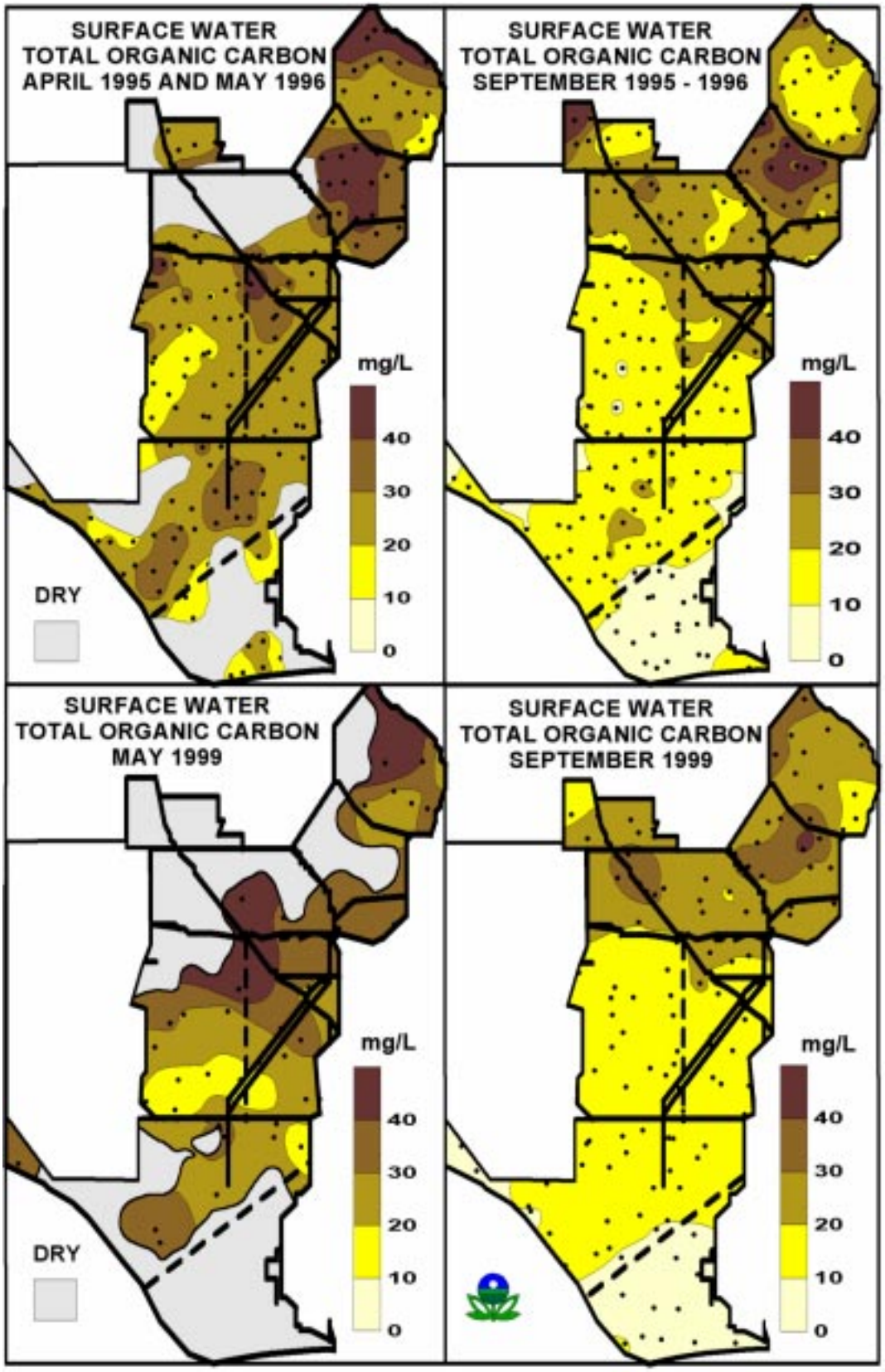


Figure 6.19. Surface plots of TOC measured in surface water during wet and dry seasons in phases 1 and 2.

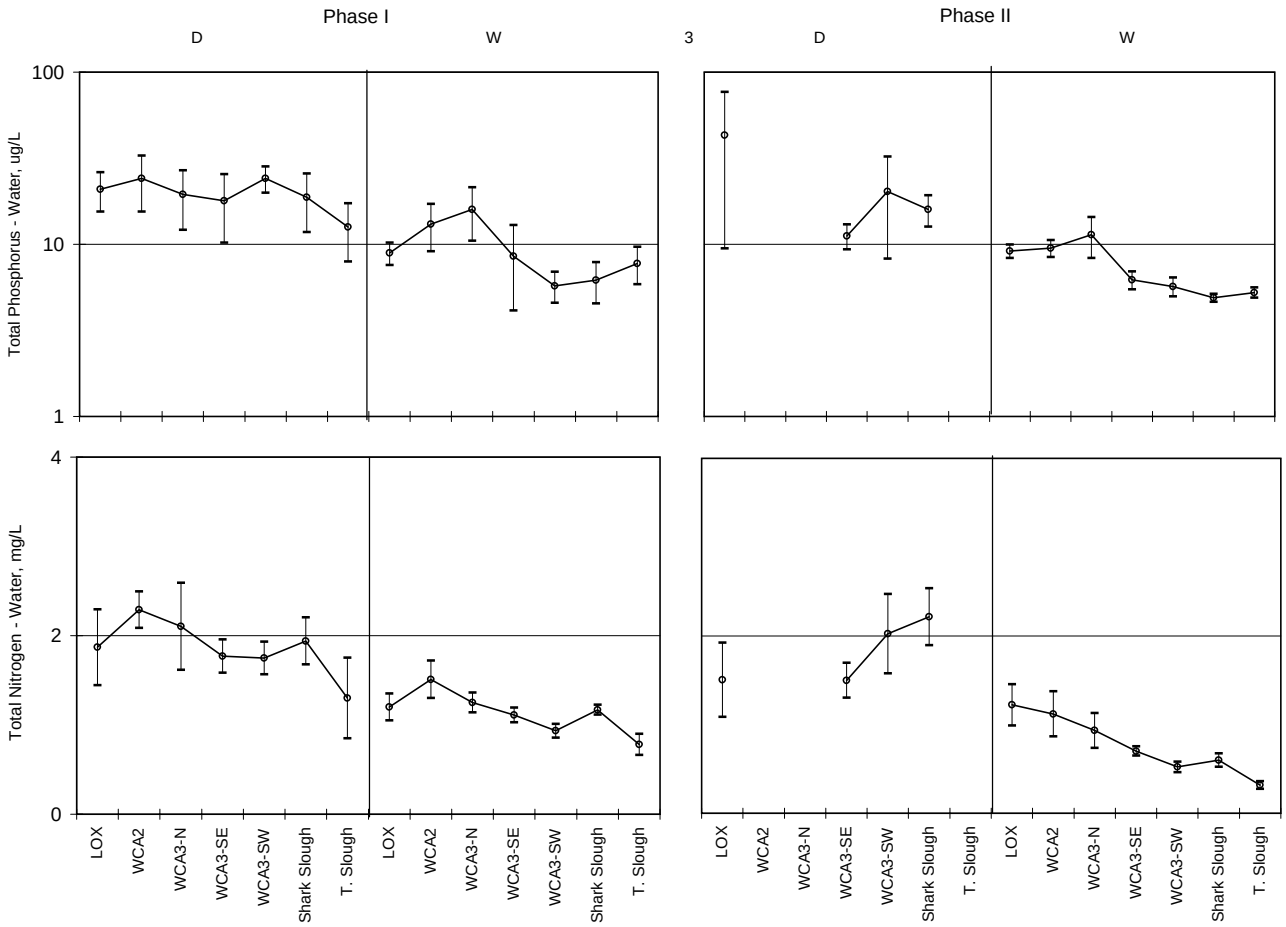


Figure 6.20. Median TP and total nitrogen measured in surface water in the subareas during wet and dry seasons of phases 1 and 2.

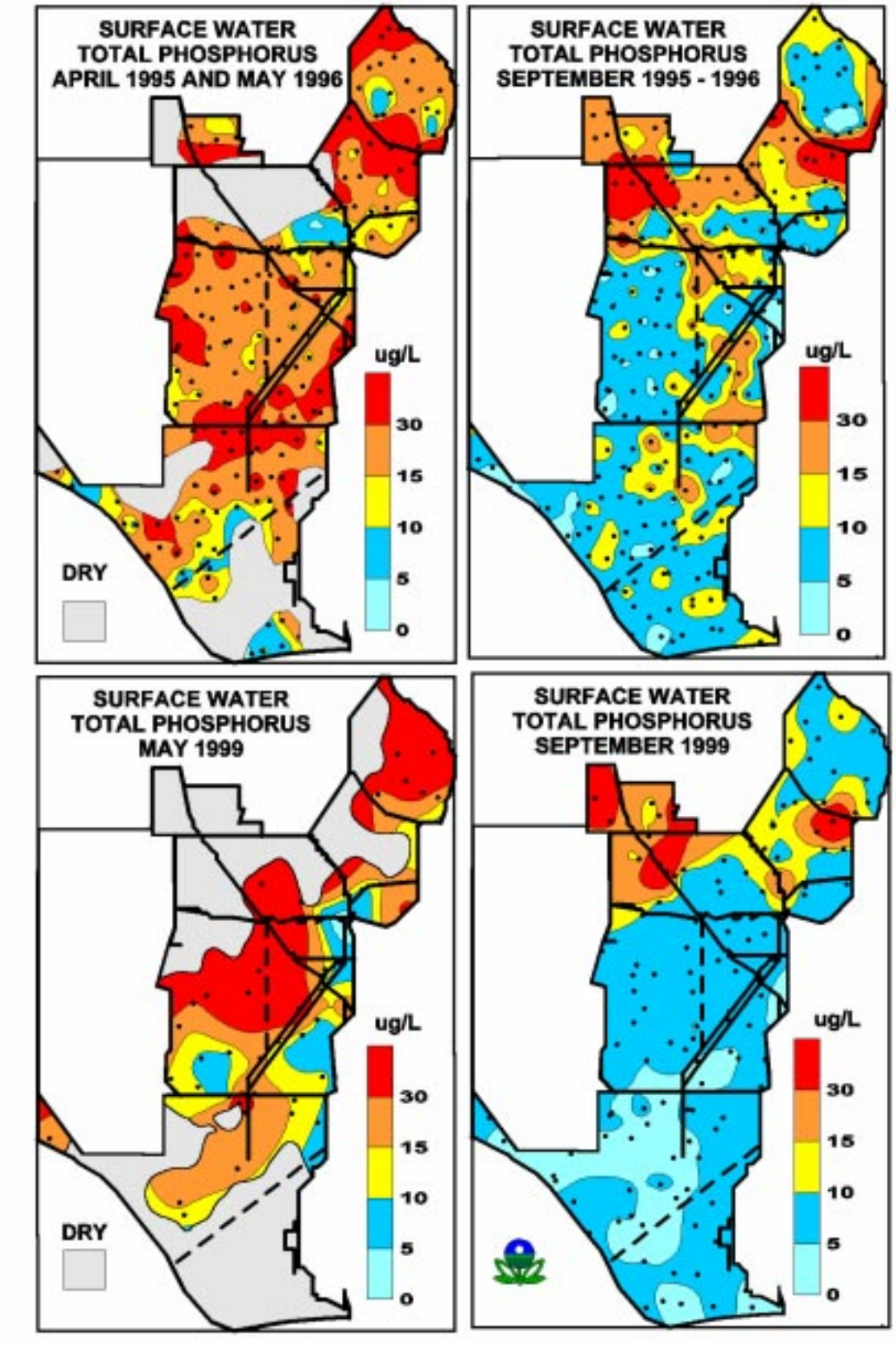


Figure 6.21. Surface plots of TP measured in surface water during wet and dry seasons of phases 1 and 2.

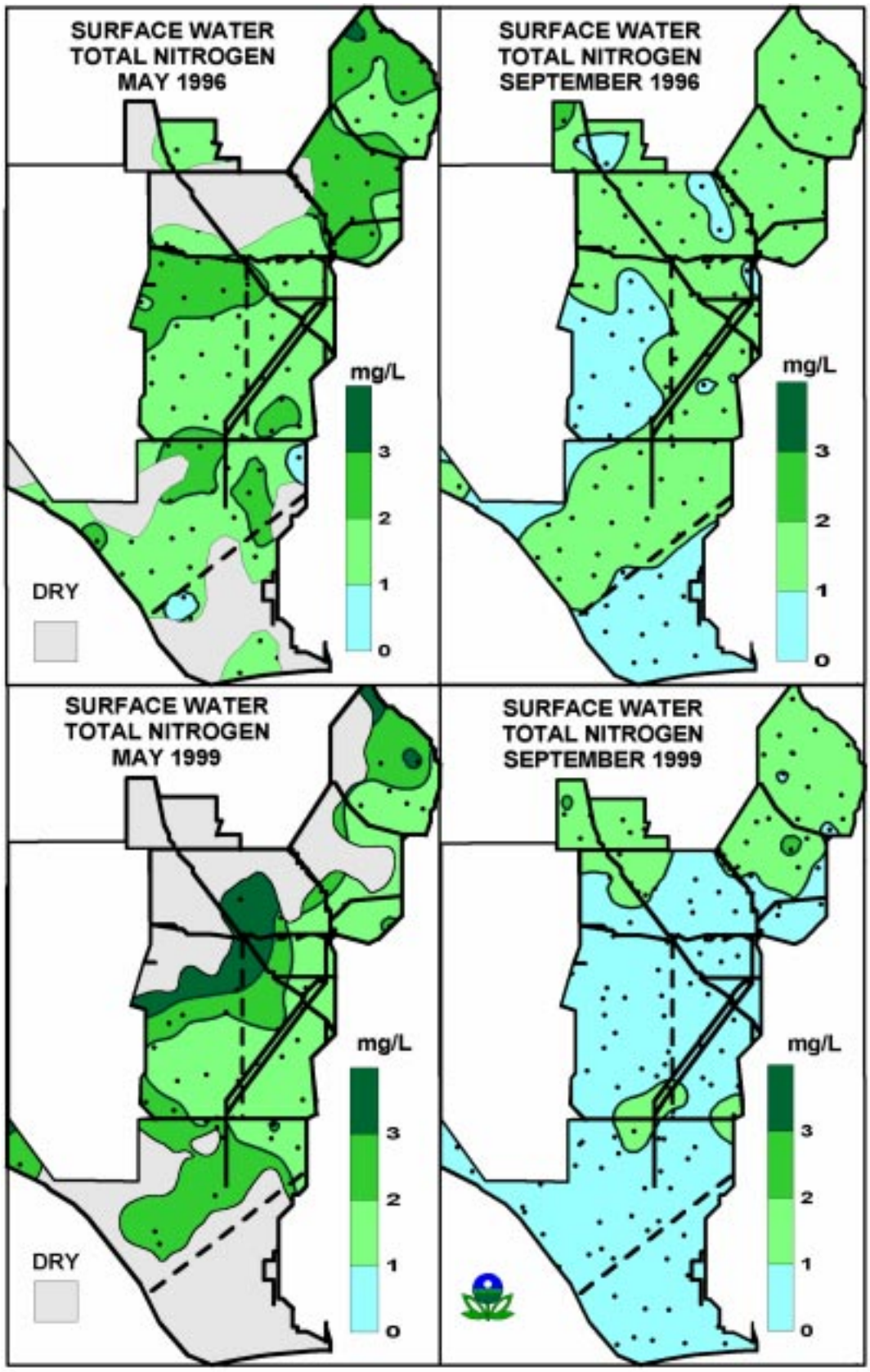


Figure 6.22. Surface plots of total nitrogen measured in surface water during wet and dry seasons in phases 1 and 2.

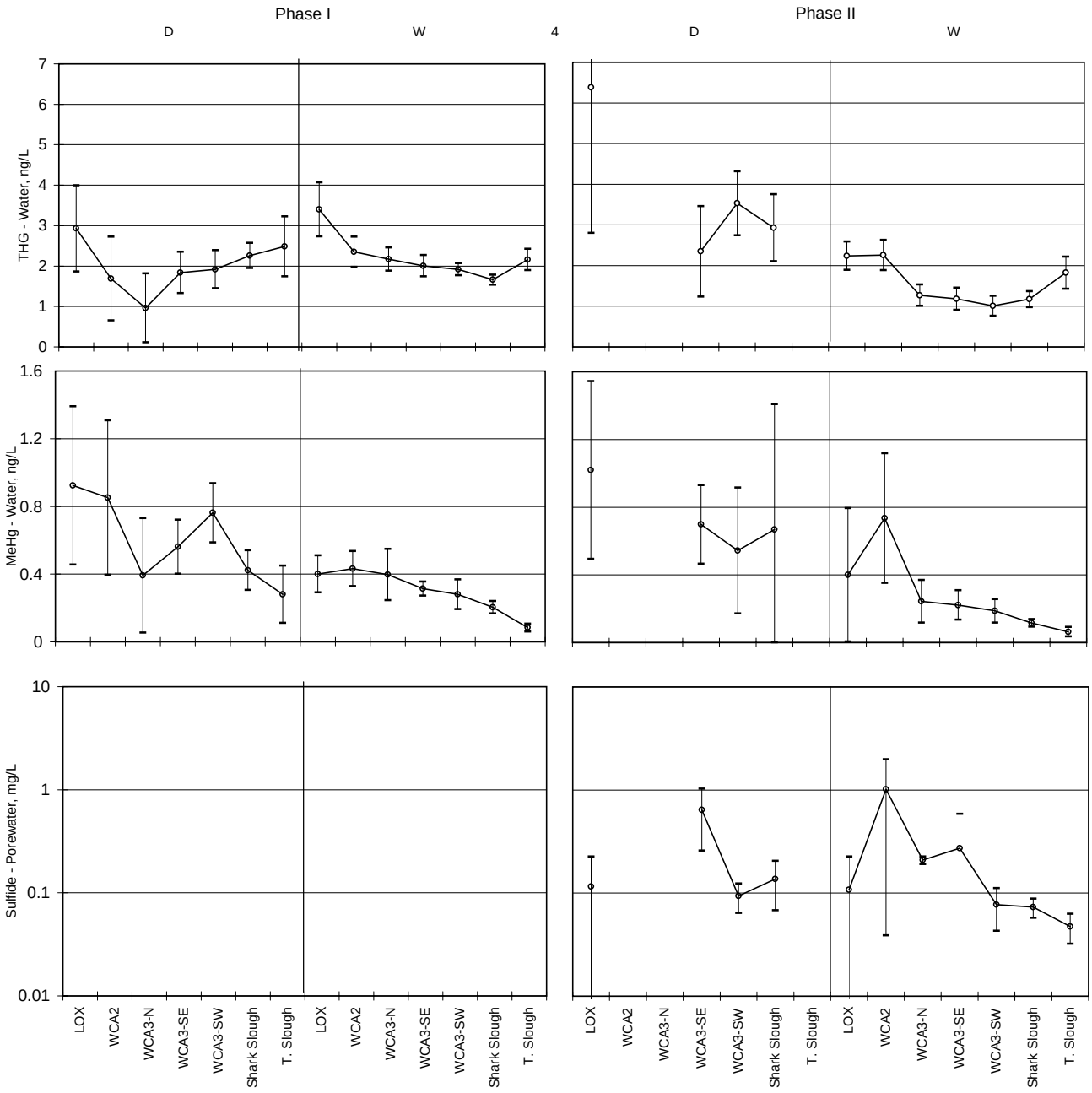


Figure 6.23. Median total mercury and methyl mercury in surface water, and sulfide in pore water (with 95% confidence intervals) measured in the subareas during wet and dry seasons of phases 1 and 2.

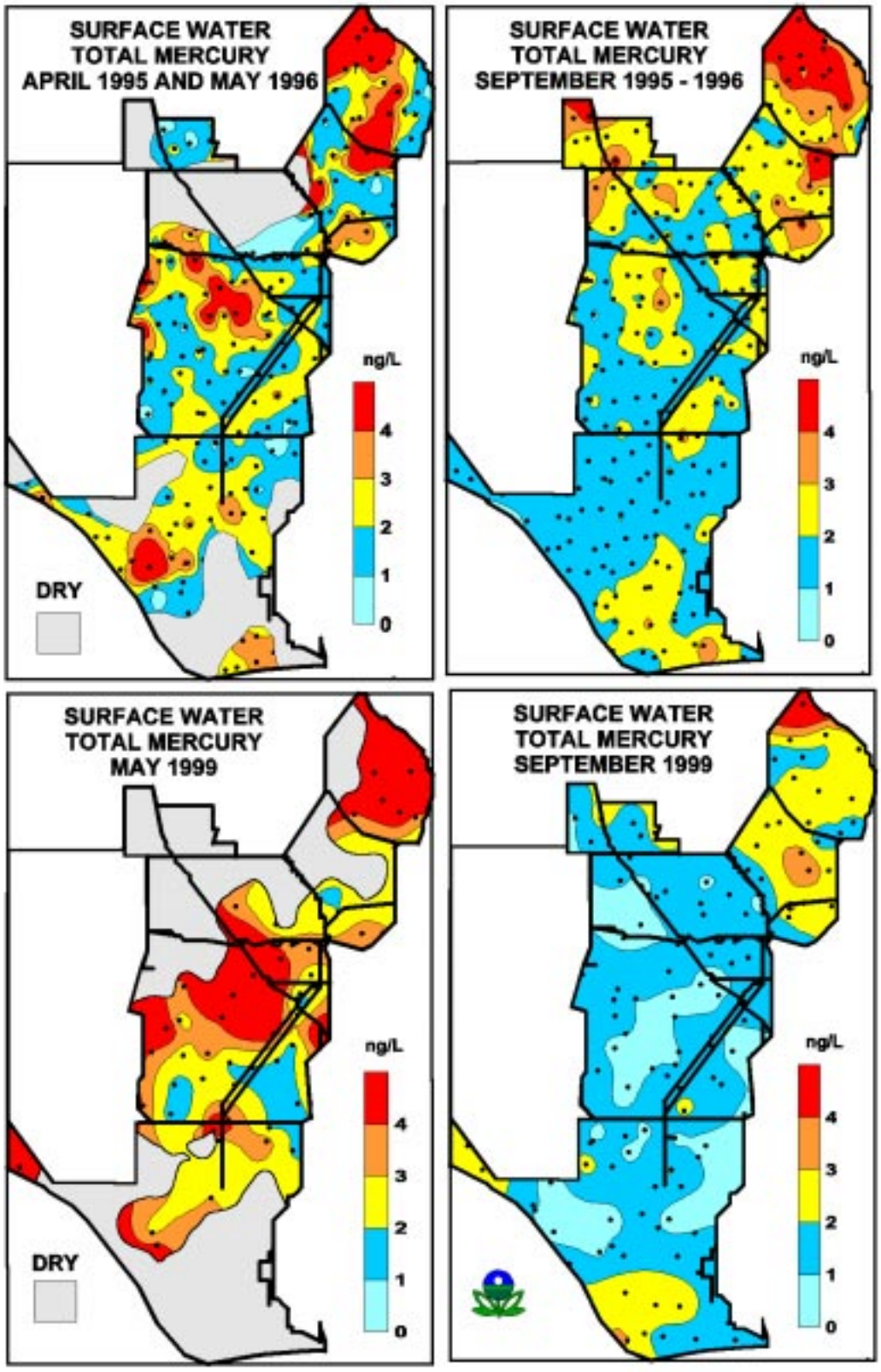


Figure 6.24. Surface plots of total mercury measured in surface water during wet and dry seasons of phases 1 and 2.

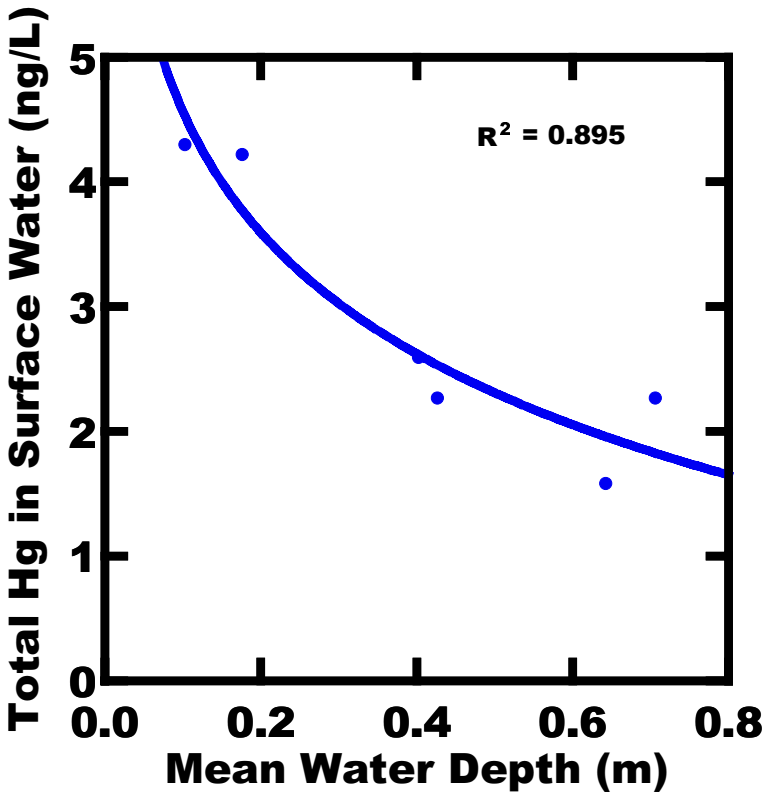


Figure 6.25. Relationship between mean total mercury in surface water and mean water depth for each of the sampling cycles (phase 1 and 2)

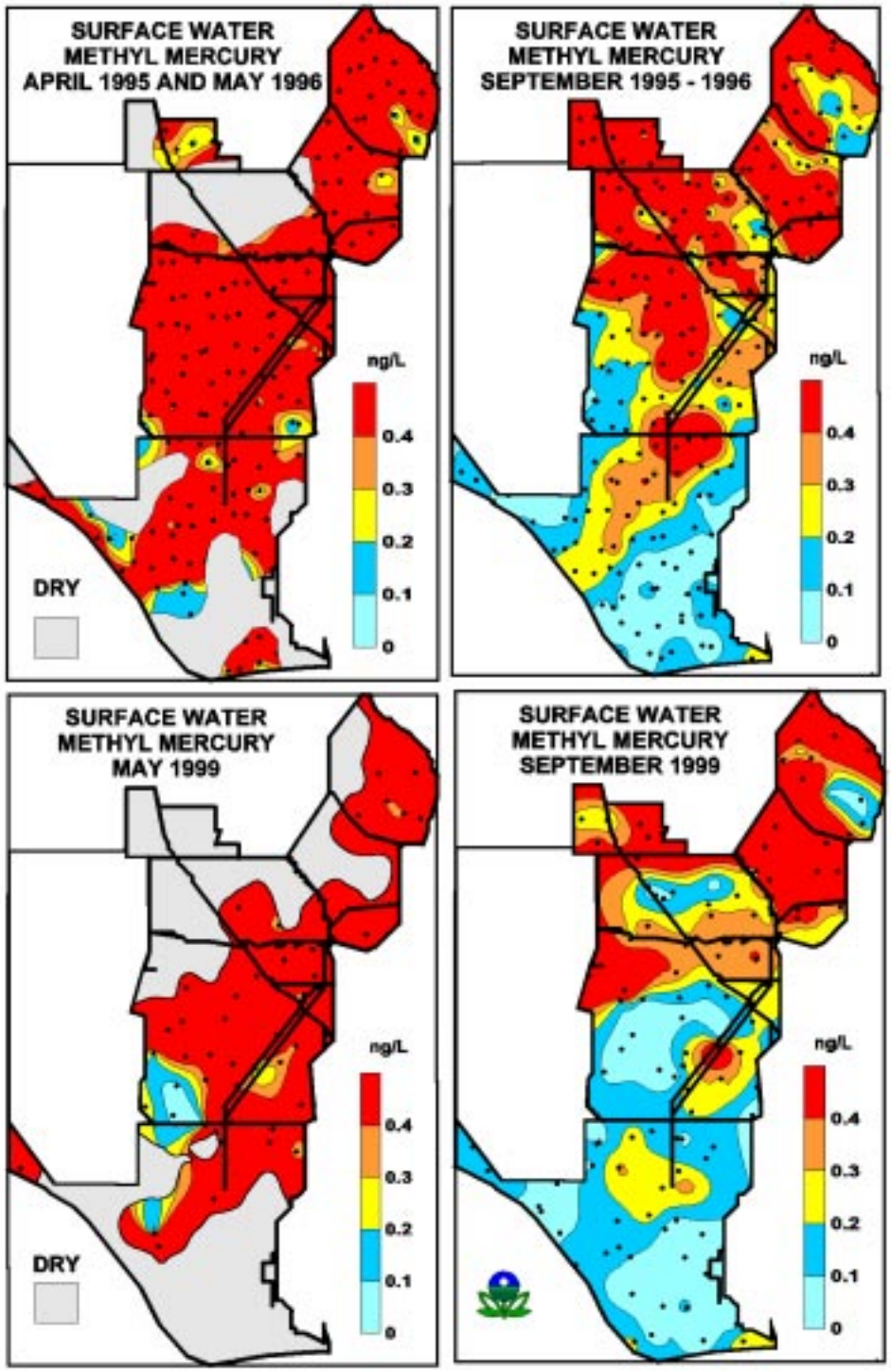


Figure 6.26. Surface plots of methyl mercury measured in surface water during wet and dry seasons of phases 1 and 2.

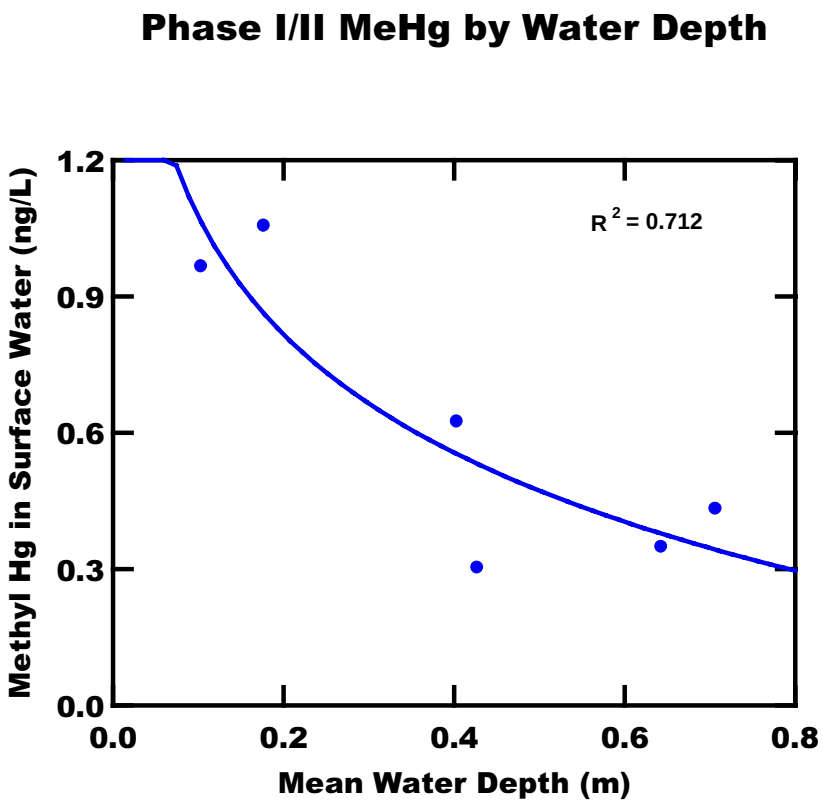


Figure 6.27. Relationship between mean methyl mercury in surface water and mean water depth for each of the sampling cycles (phase 1 and 2).

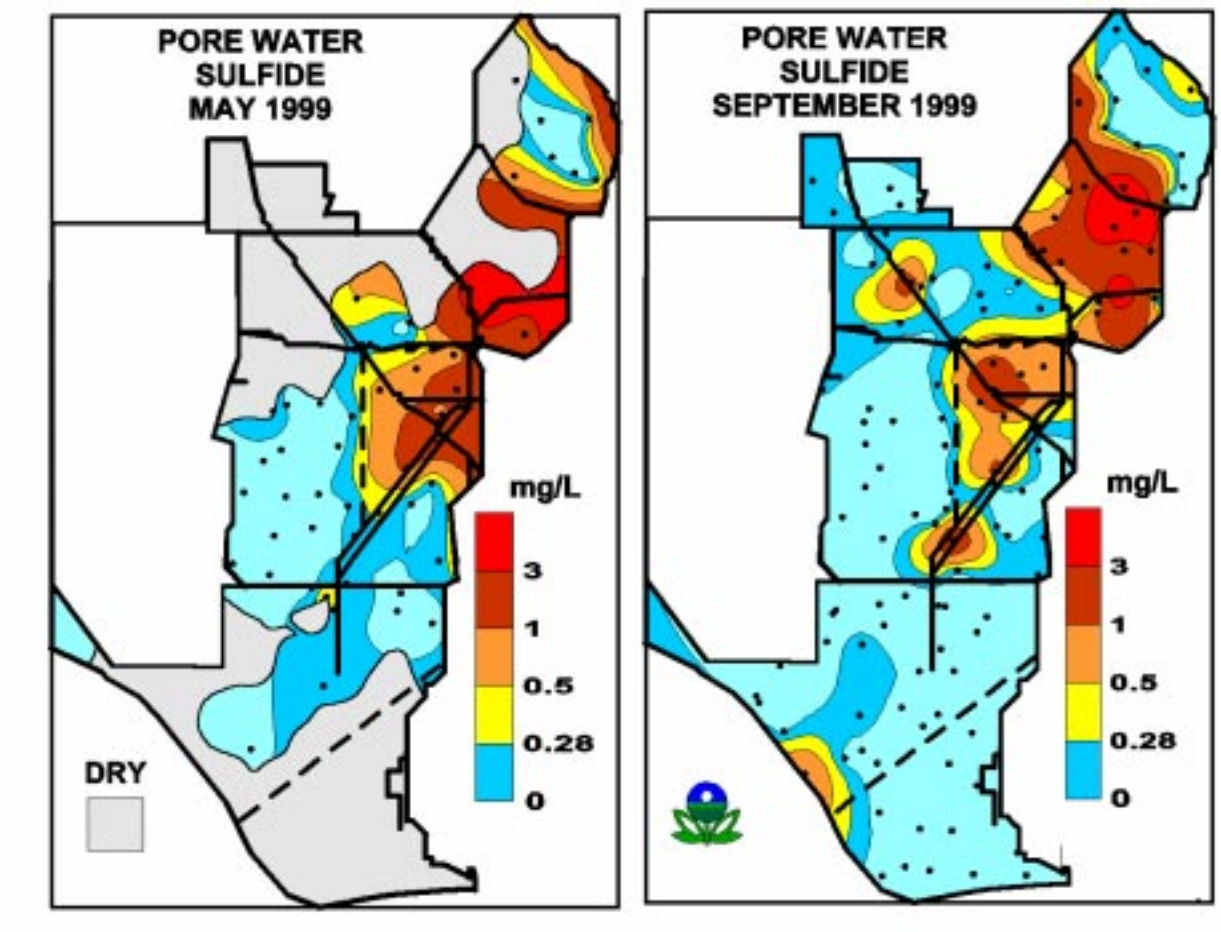


Figure 6.28. Surface plot of sulfide measured in pore water during wet and dry seasons in phase 2.

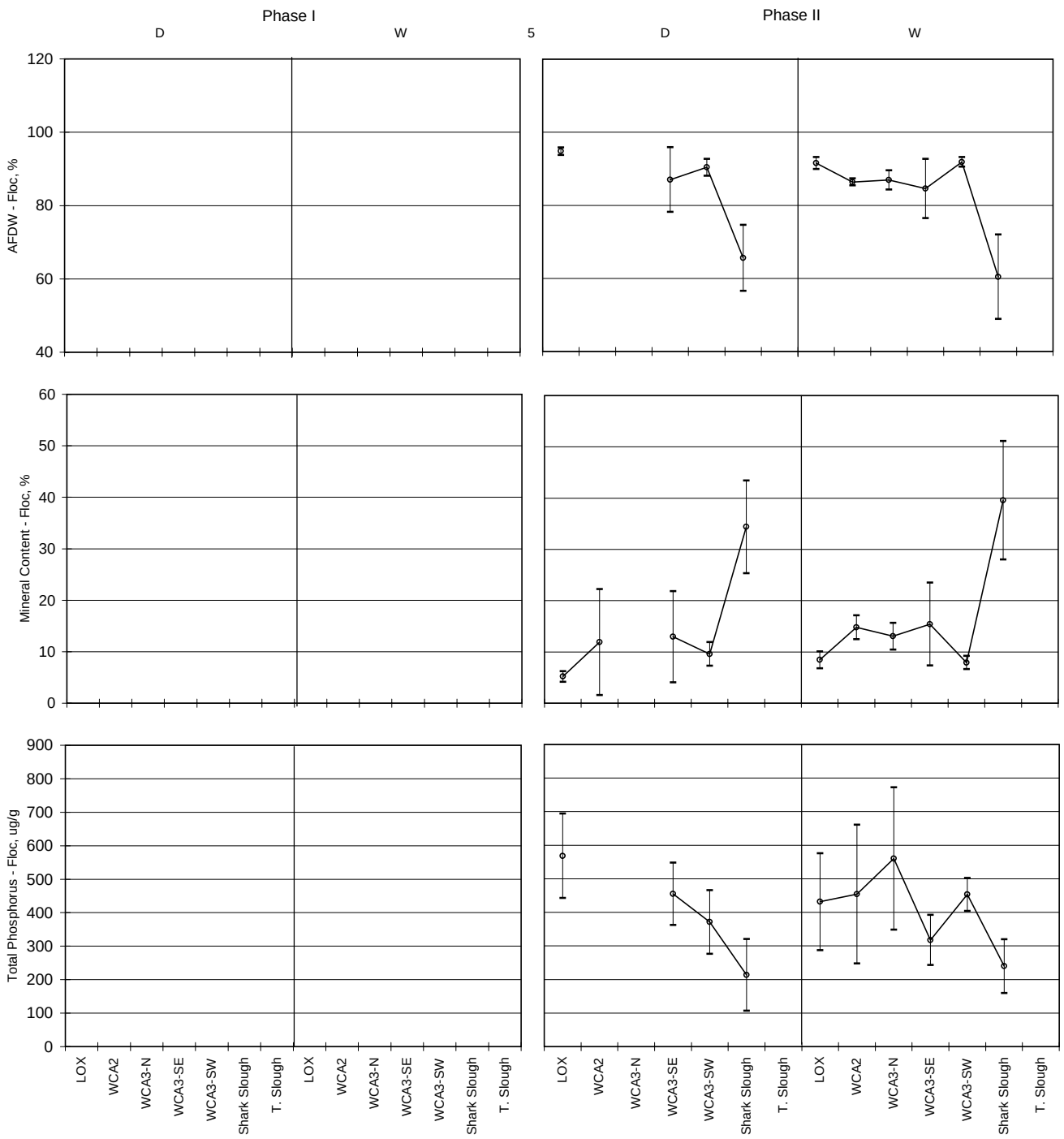


Figure 6.29. Median AFDW, mineral content, and total phosphorus in floc (with 95% confidence intervals) measured in subareas during wet and dry seasons in phases 1 and 2.

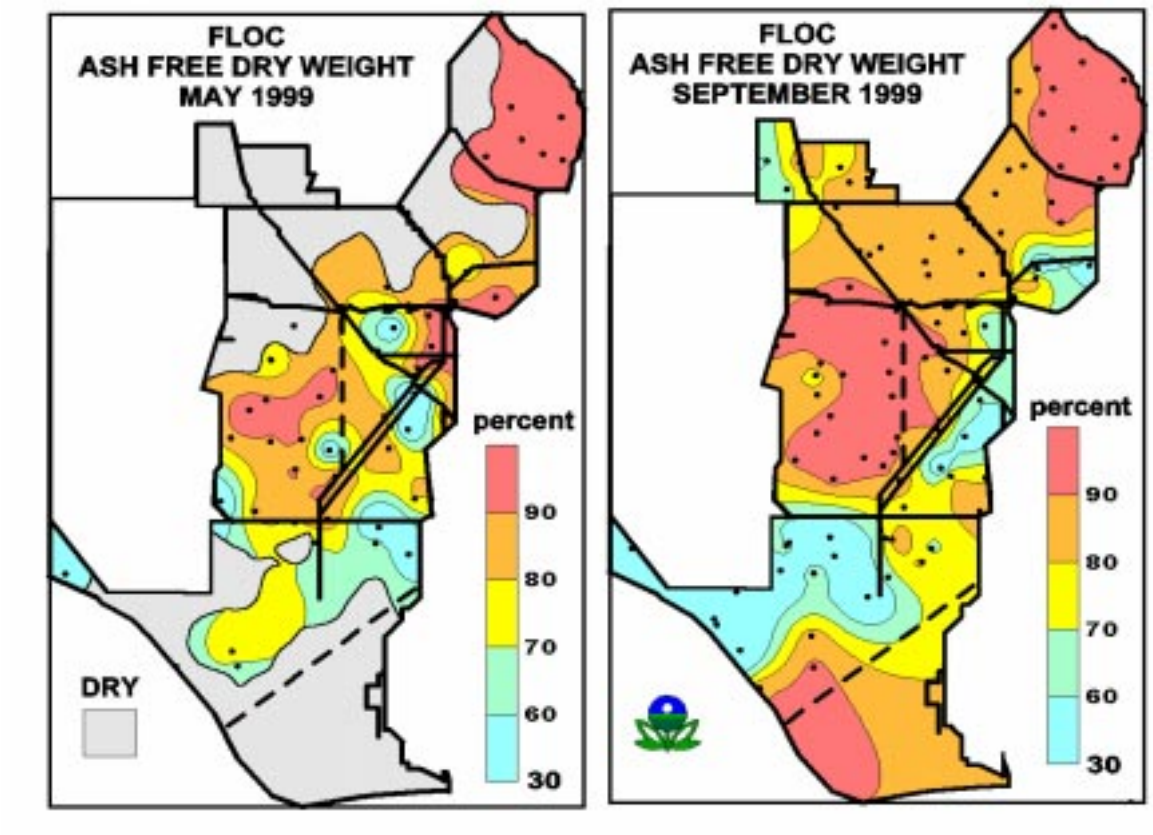


Figure 6.30. Surface plot of AFDW of floc measured during wet and dry seasons in phase 2.

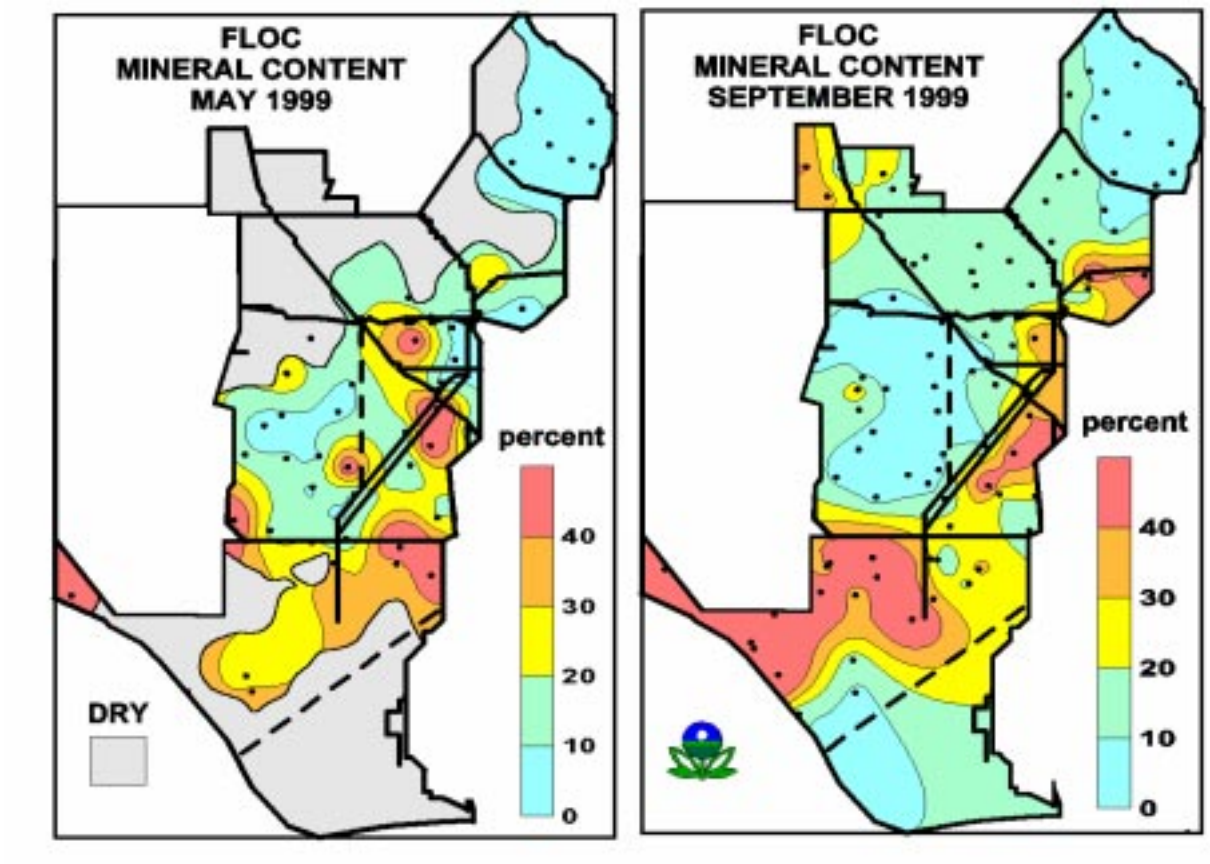


Figure 6.31. Spatial plots of floc mineral content measured during wet and dry seasons in phase 2.

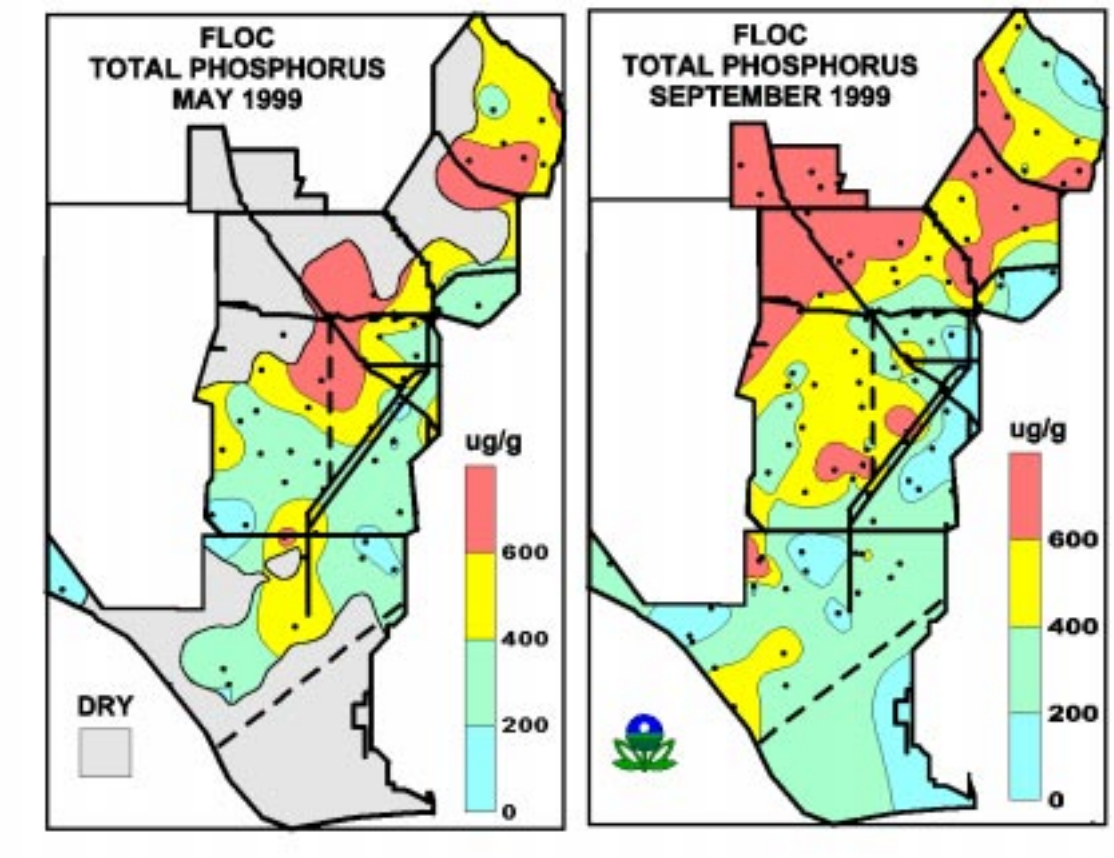


Figure 6.32. Spatial plots of total phosphorus in floc measured during wet and dry seasons in phase 2.

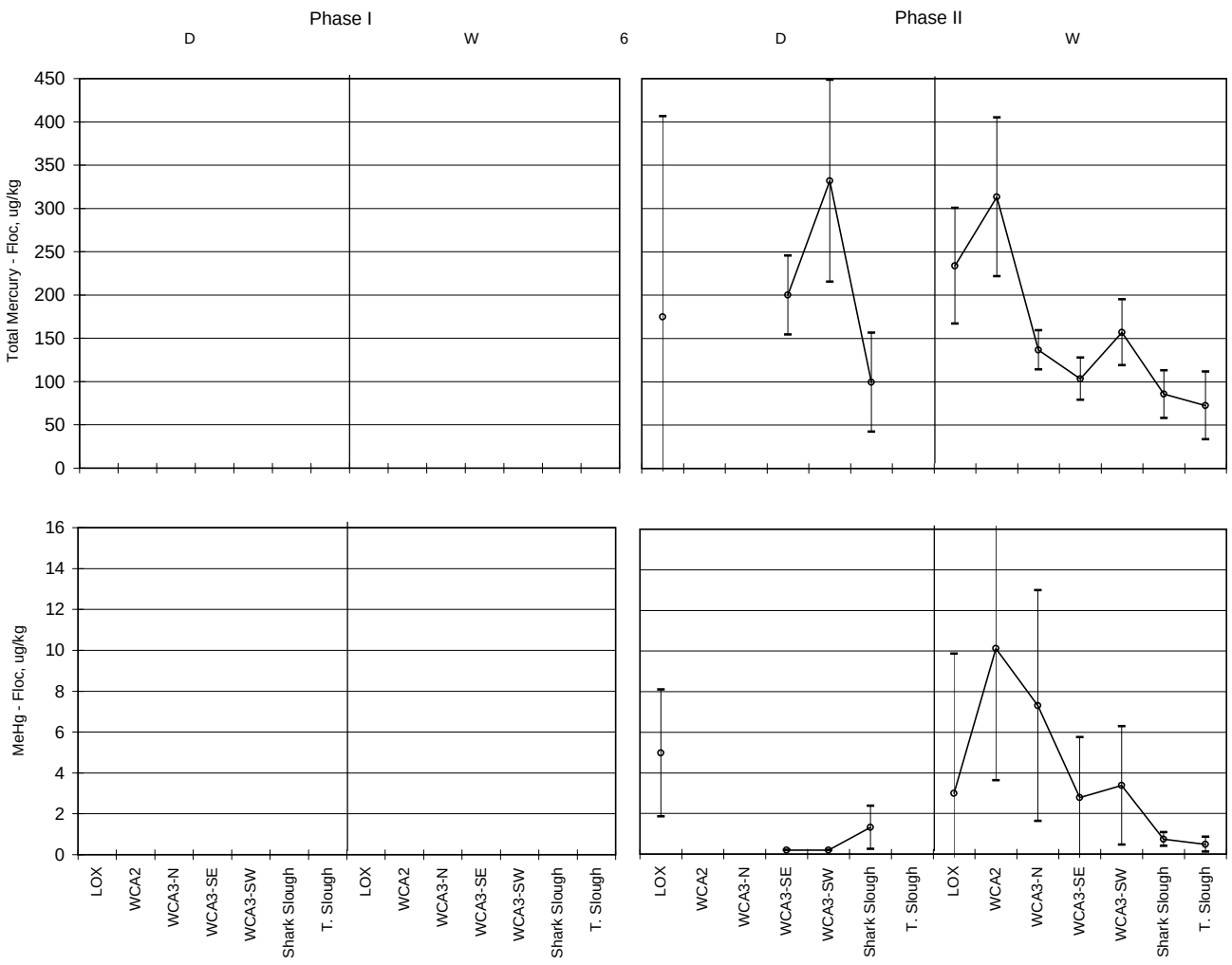


Figure 6.33. Median total mercury and methyl mercury in floc (with 95% confidence intervals) measured in subareas during wet and dry seasons in phases 1 and 2.

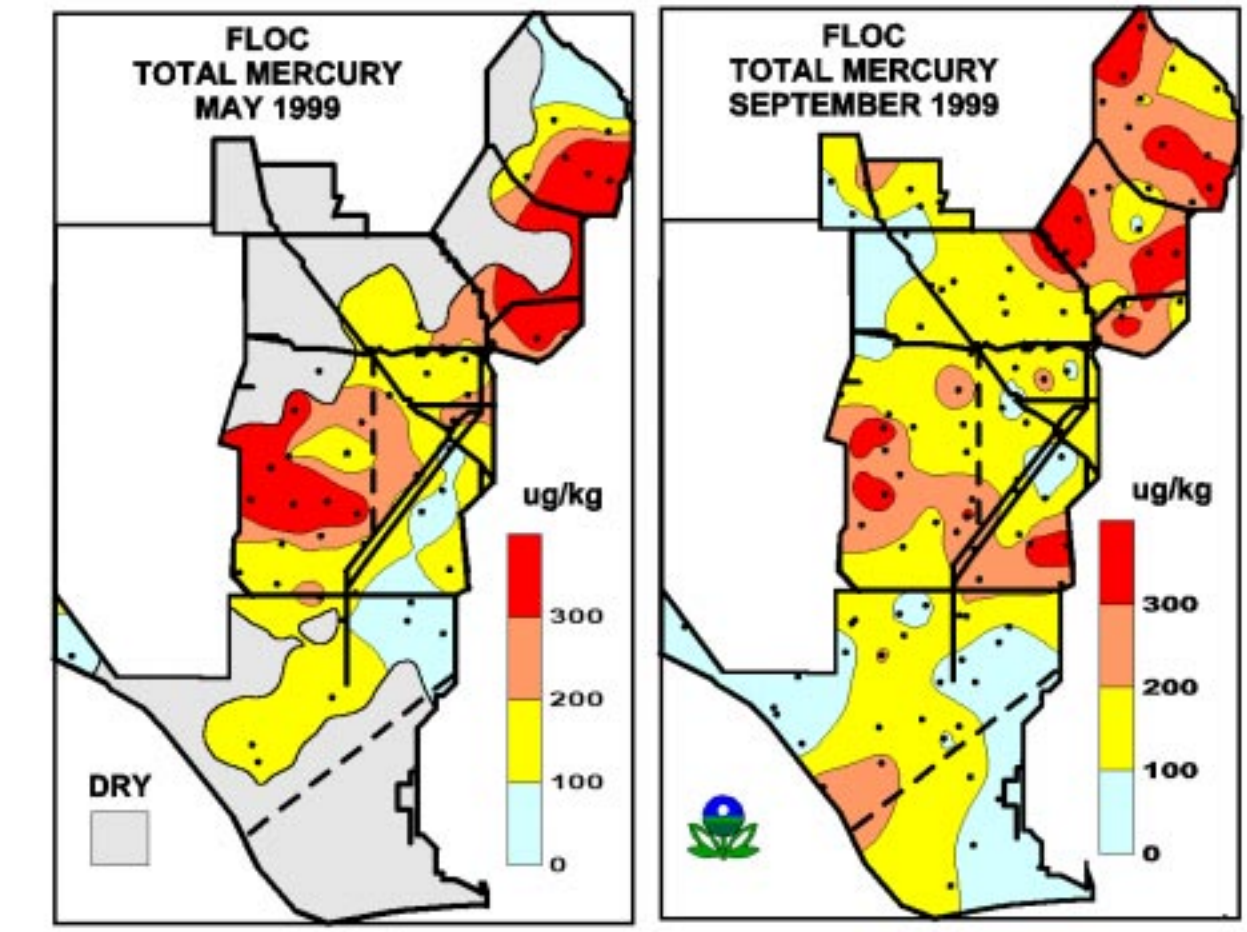


Figure 6.34. Spatial plots of total mercury in floc measured during wet and dry seasons in phase 2.

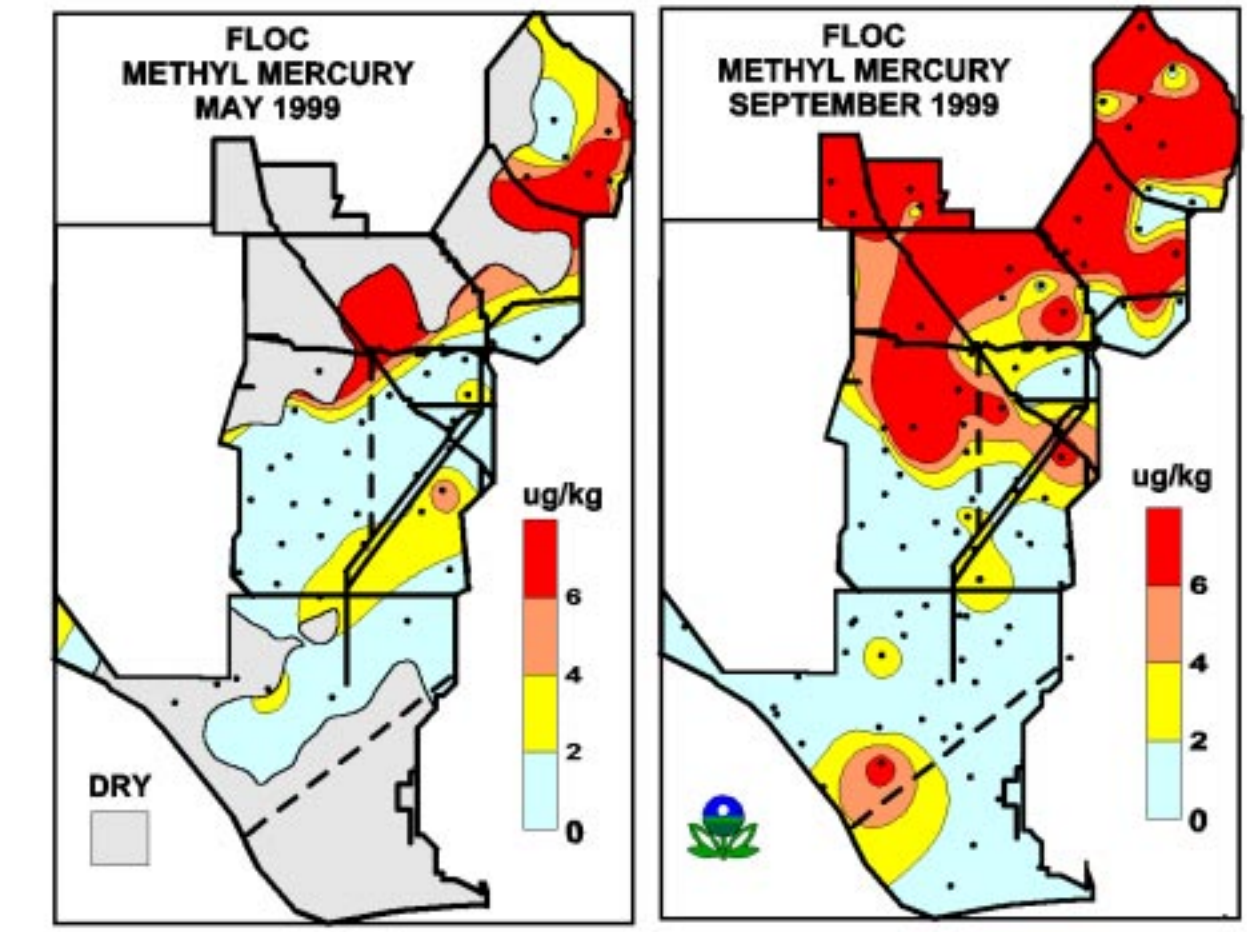


Figure 6.35. Spatial plots of methyl mercury in floc measured during wet and dry seasons in phase 2.

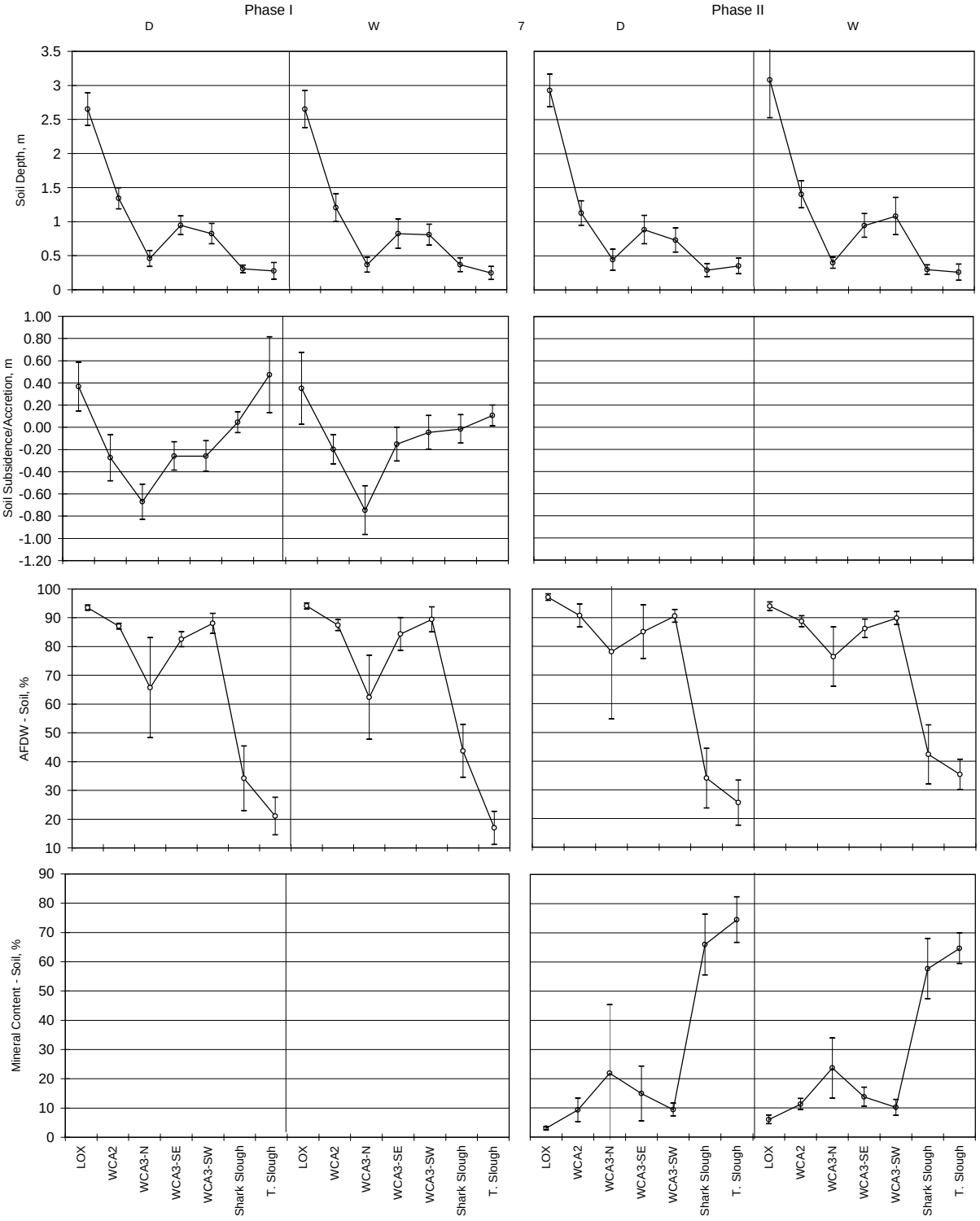


Figure 6.36. Median soil depth, soil subsidence/accretion, soil AFDW, and soil mineral content (with 95% confidence intervals) measured in subareas during wet and dry seasons in phases 1 and 2.

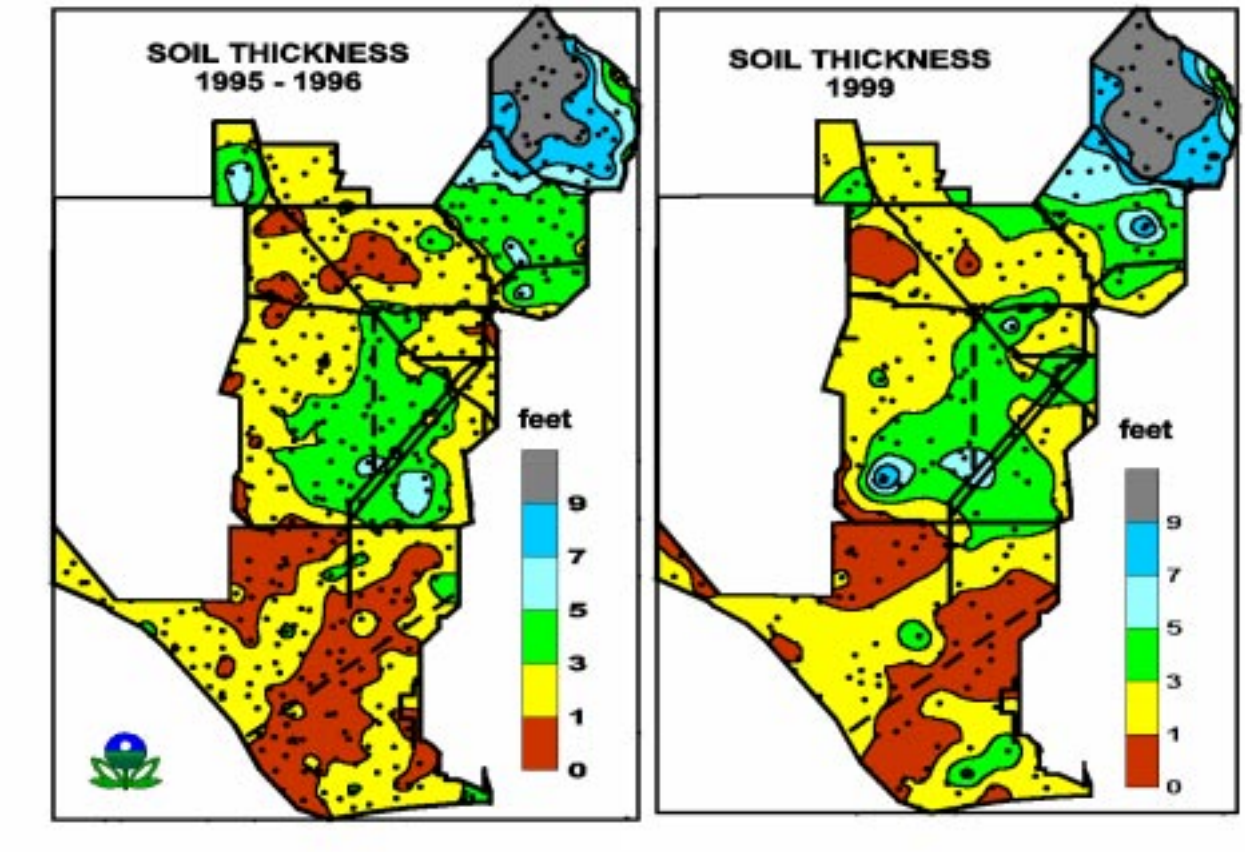


Figure 6.37. Spatial plots of soil thicknesses measured during phases 1 and 2.

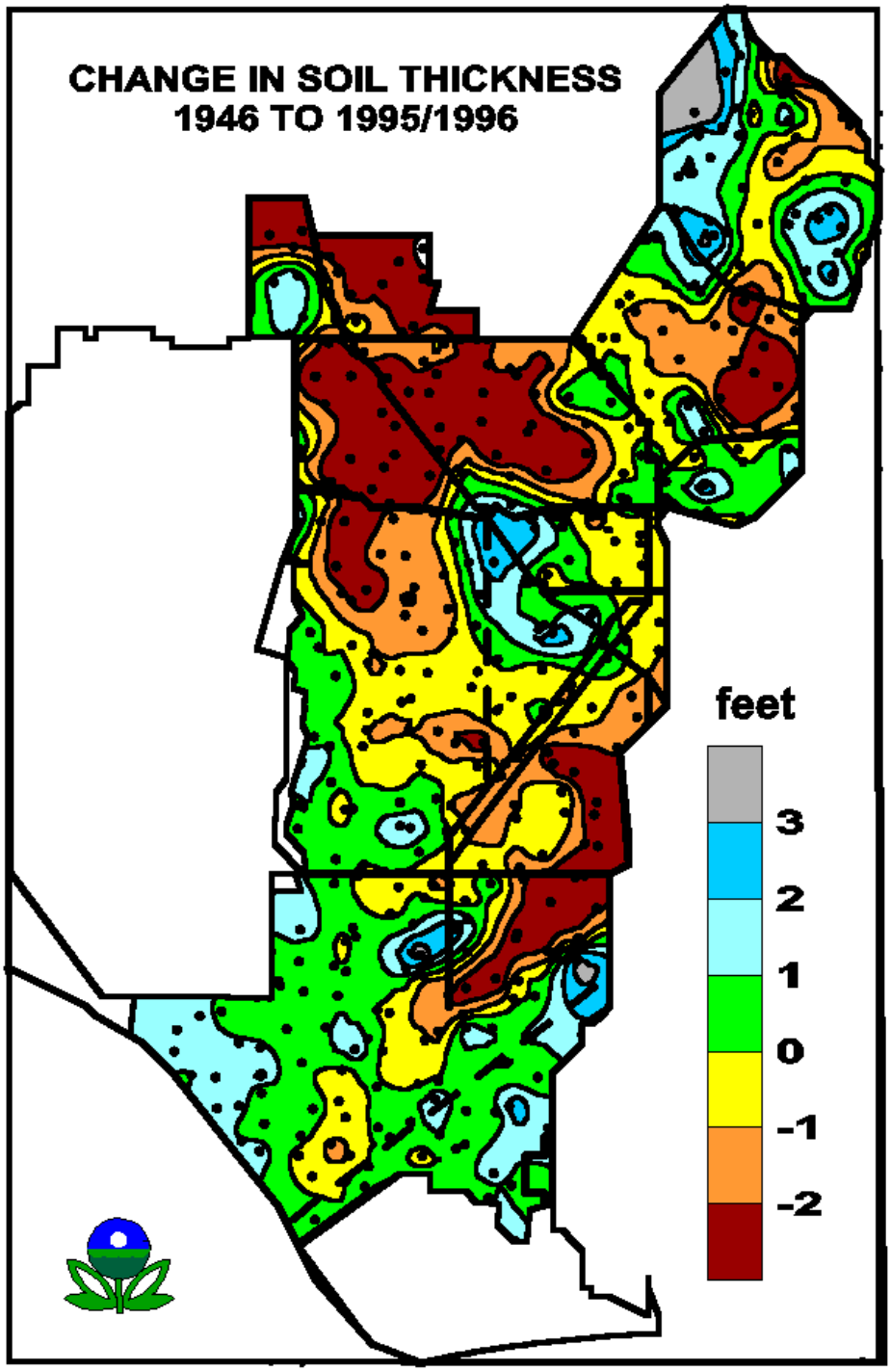


Figure 6.38. Spatial plot of soil subsidence and accretion from 1946 to 1995/1996.

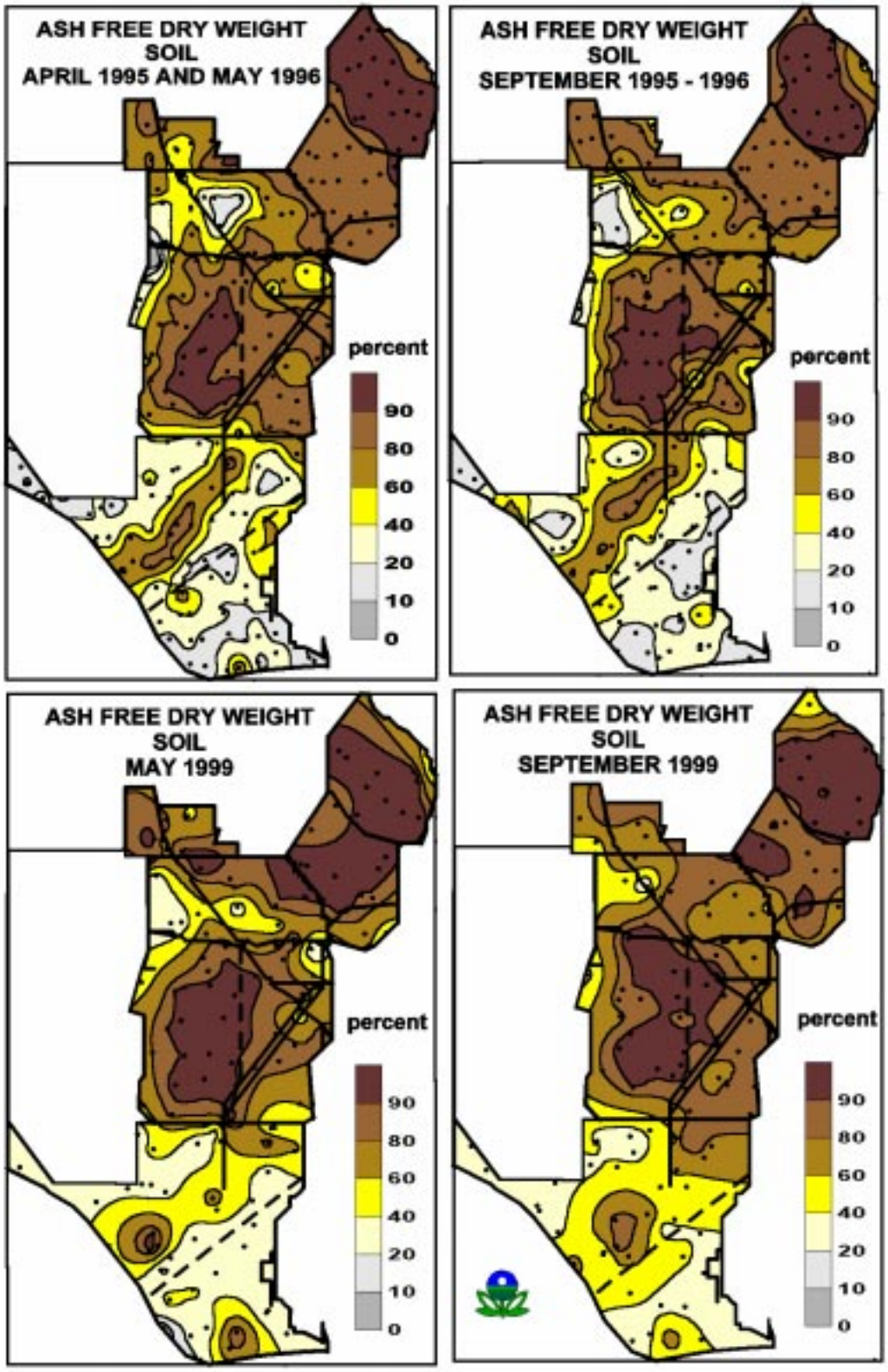


Figure 6.39. Spatial plots of soil AFDW measured during wet and dry seasons in phases 1 and 2.

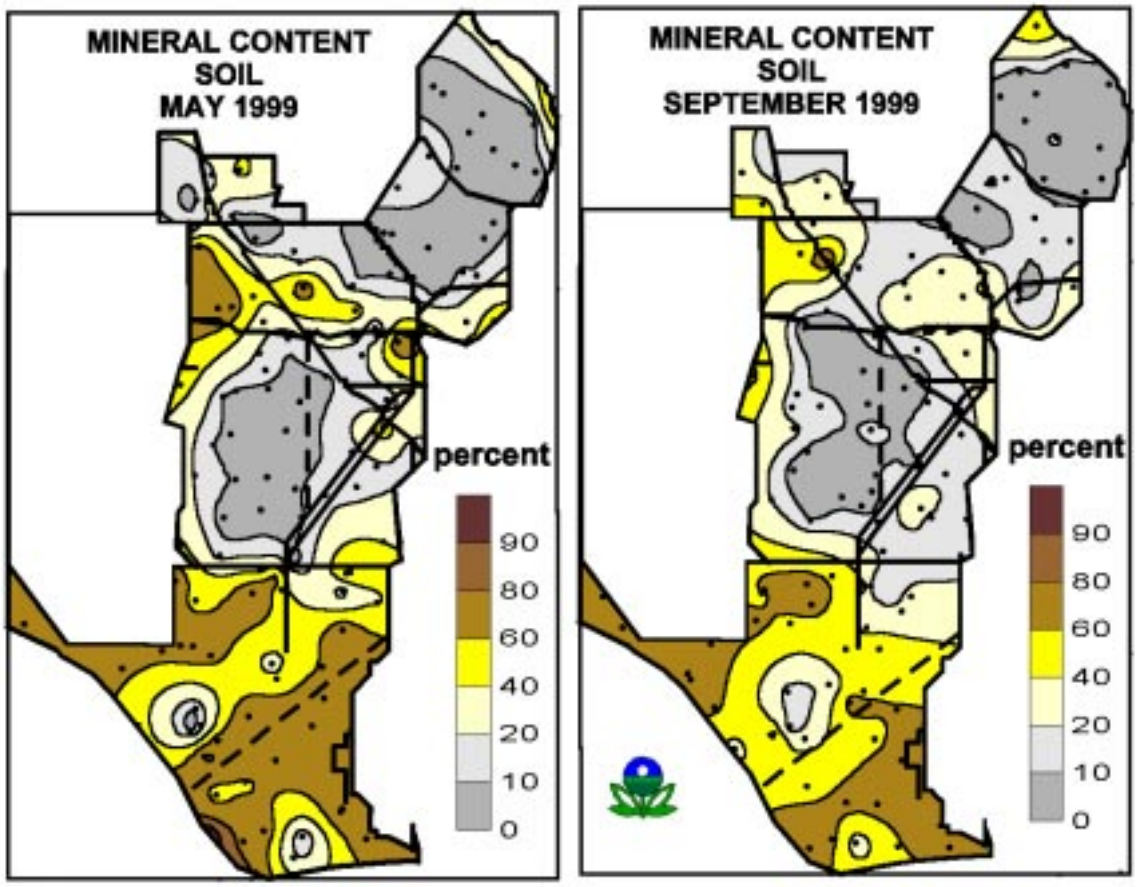


Figure 6.40. Spatial plots of soil mineral content measured during wet and dry seasons in phase 2.

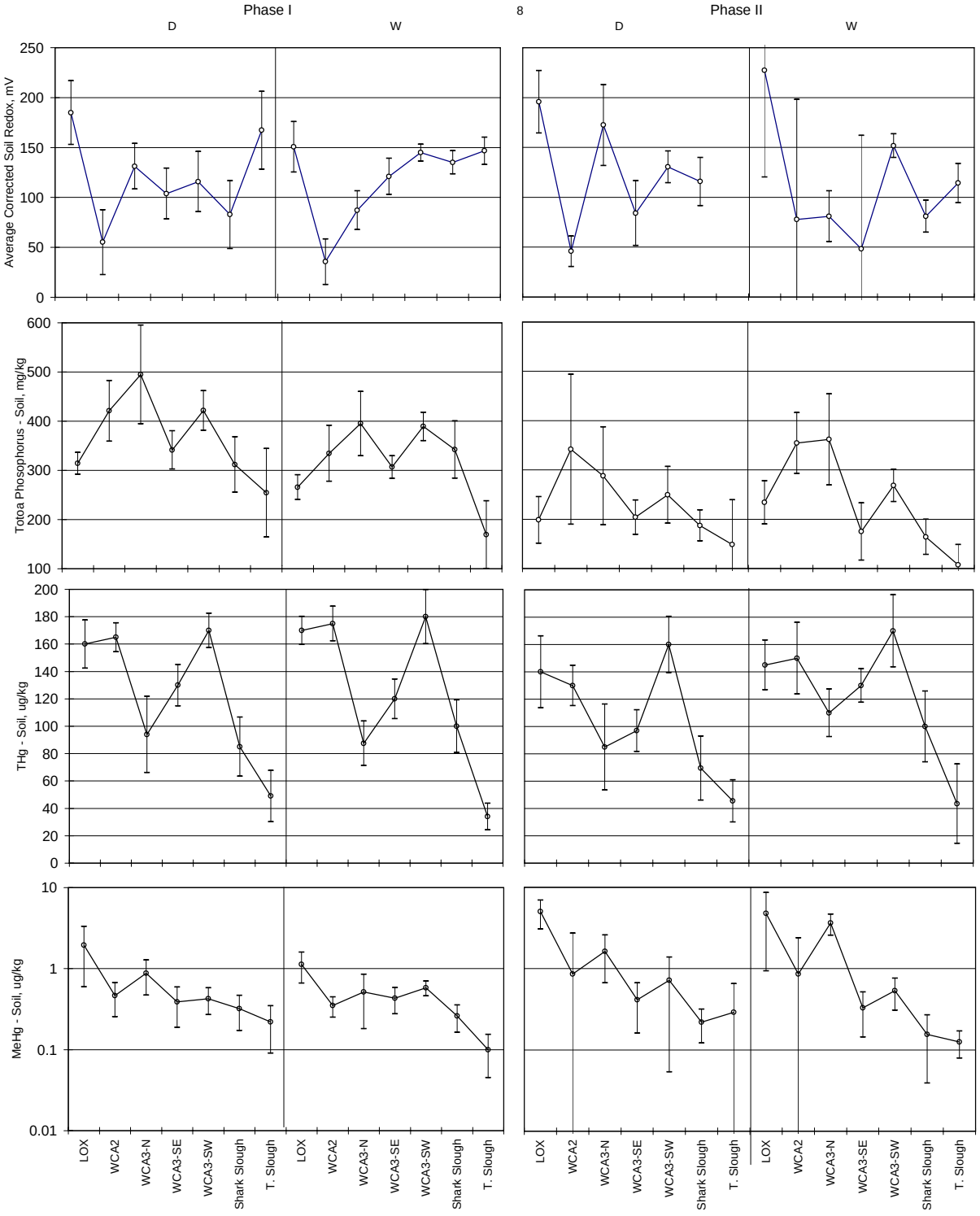


Figure 6.41. Median redox, total phosphorus, total mercury, and methyl mercury in soil (with 95% confidence intervals) measured in subareas during wet and dry seasons in phases 1 and 2.

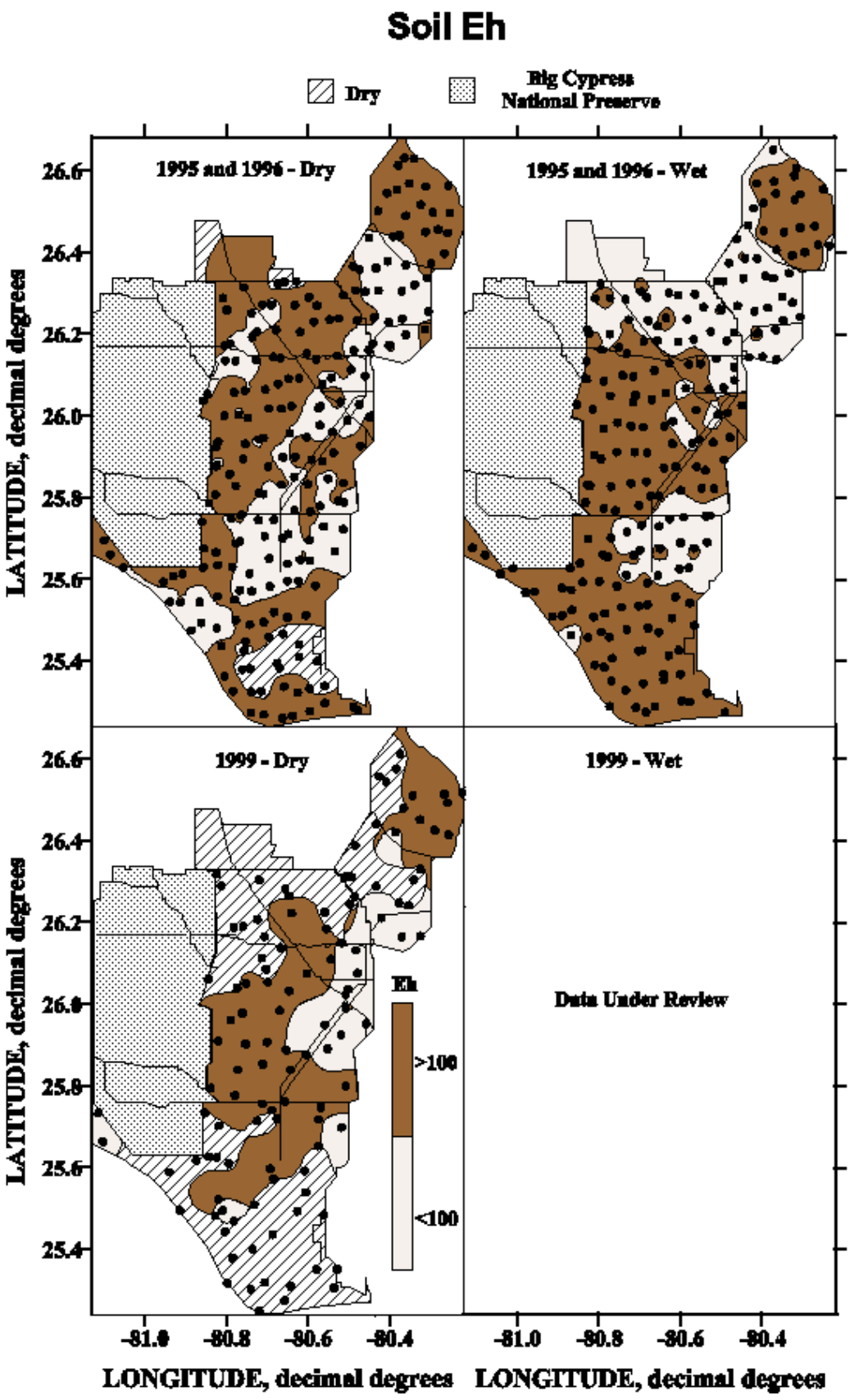


Figure 6.42. Spatial plots of soil Eh measured during wet and dry seasons in phases 1 and 2.

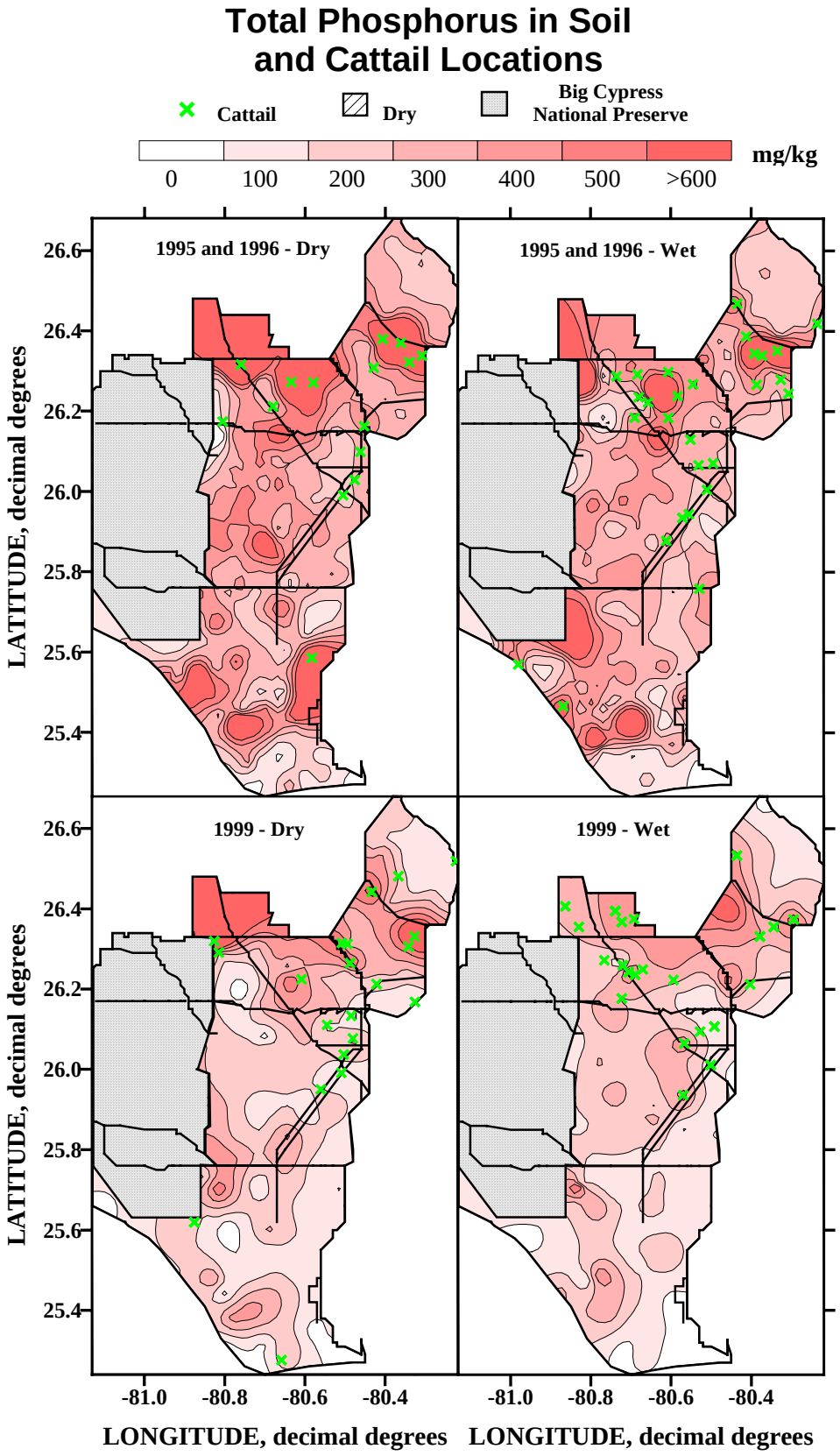


Figure 6.43. Spatial plots of total phosphorus measured during phases 1 and 2 indicating sites where cattails occurred.

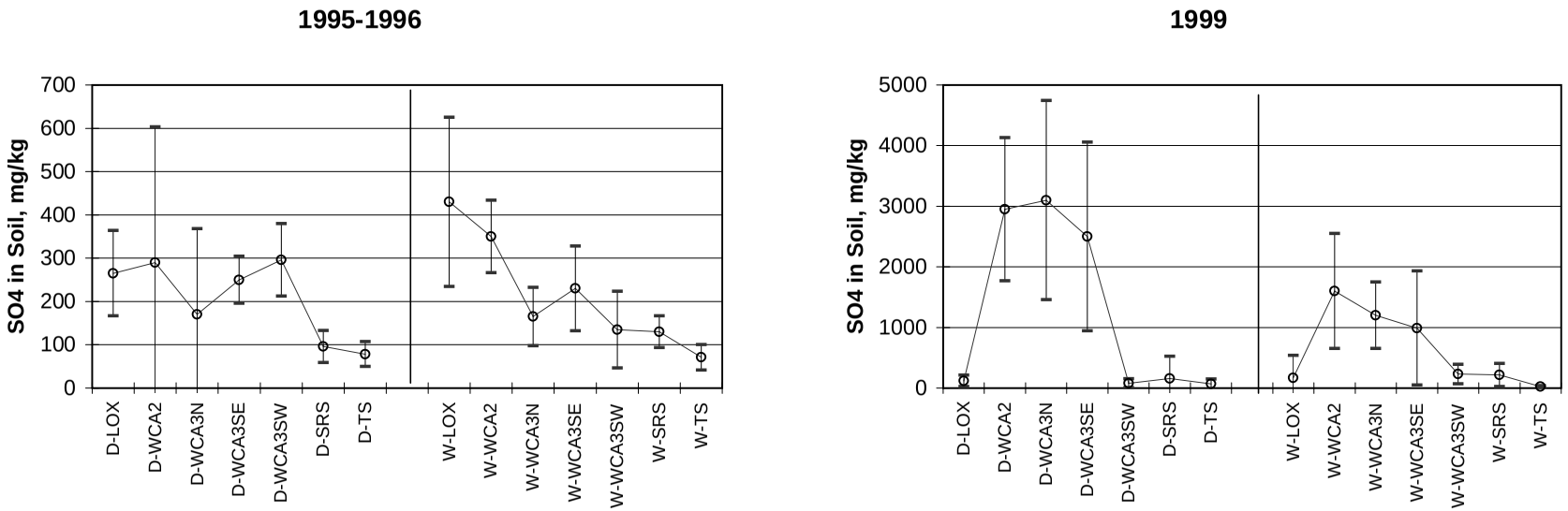


Figure 6.44. Median total sulfate in soil (with 95% confidence intervals) measured in subareas during wet and dry seasons in phases 1 and 2.

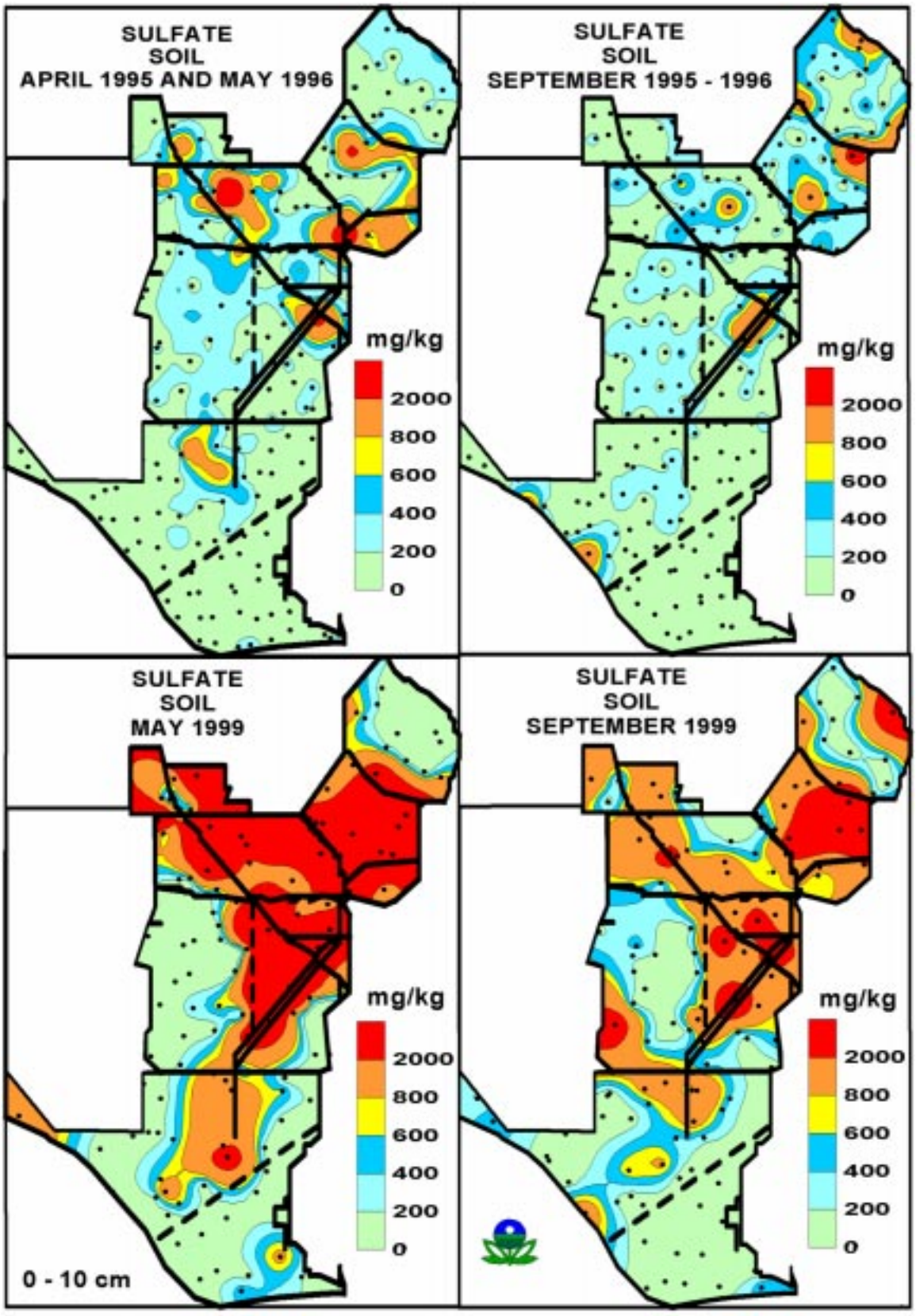


Figure 6.45. Spatial plots of total sulfate in soil measured during wet and dry seasons in phases 1 and 2.

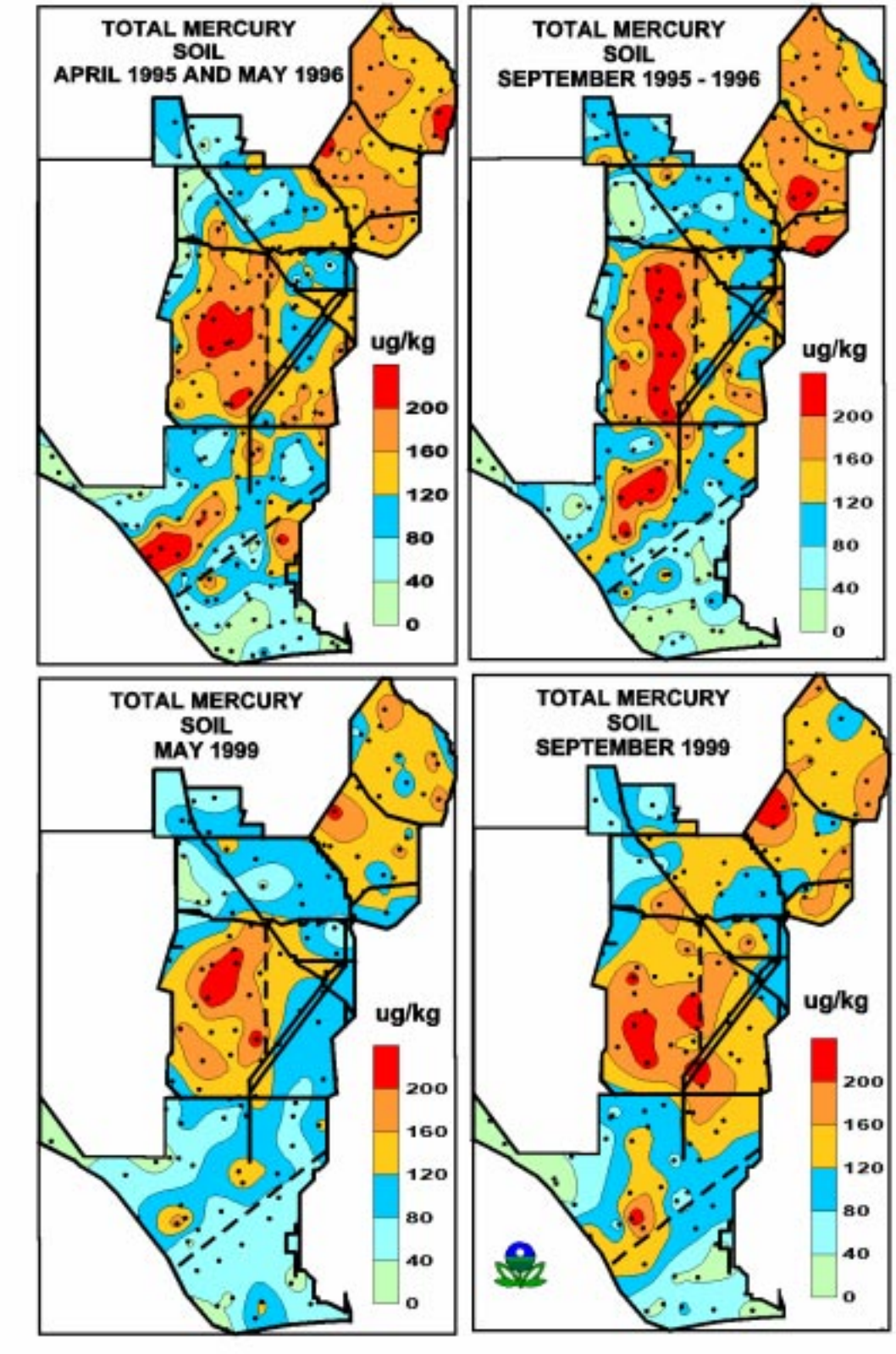


Figure 6.46. Spatial plots of total mercury in soil measured during wet and dry seasons in phases 1 and 2.

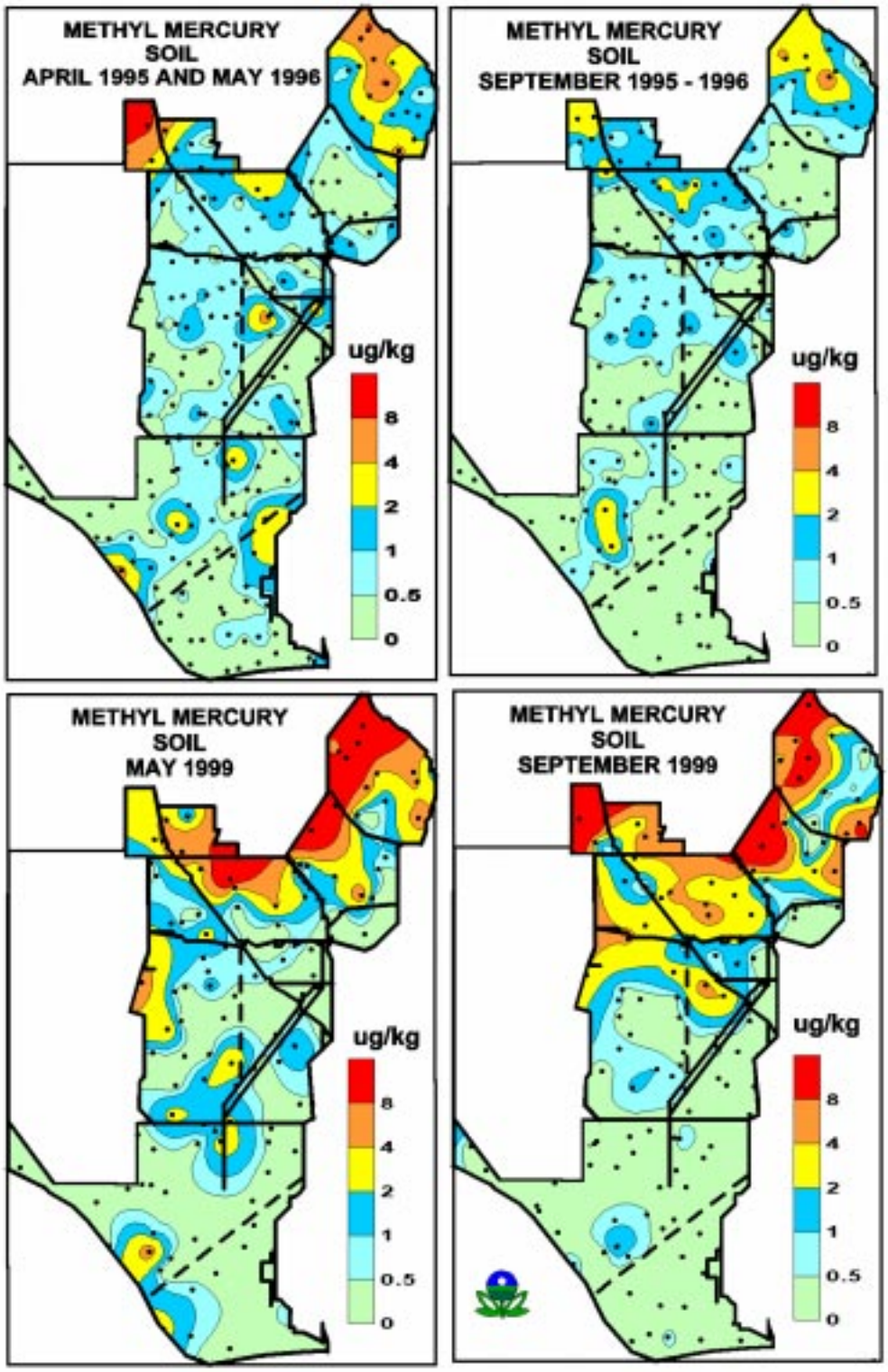


Figure 6.47. Spatial plots of methyl mercury in soil measured during wet and dry seasons in phases 1 and 2.

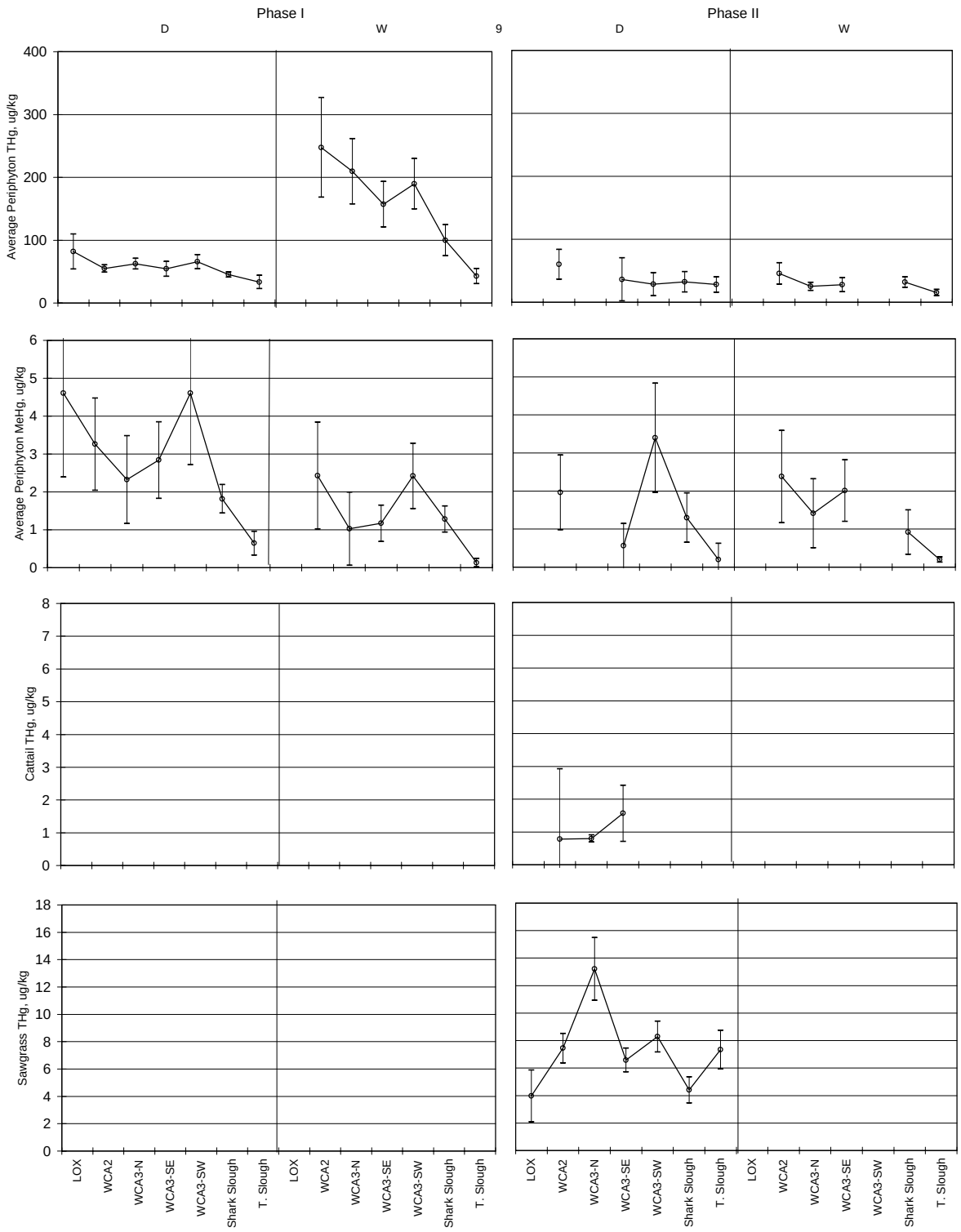


Figure 6.48. Median total mercury and methyl mercury in periphyton, and total mercury in cattails and sawgrass (with 95% confidence intervals) measured in subareas during wet and dry seasons in phases 1 and 2.

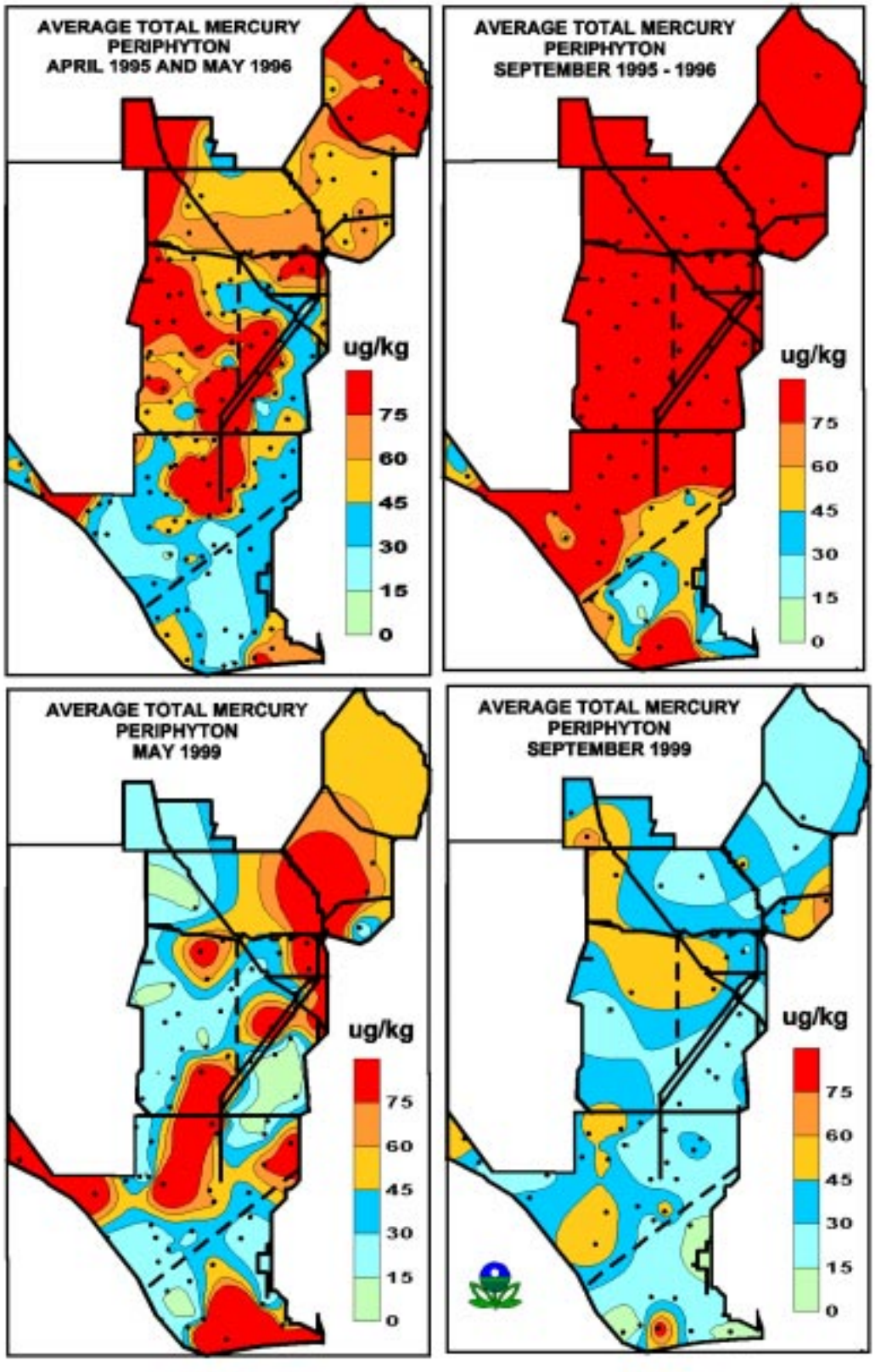


Figure 6.49. Spatial plots of average total mercury in periphyton measured during wet and dry seasons in phases 1 and 2.

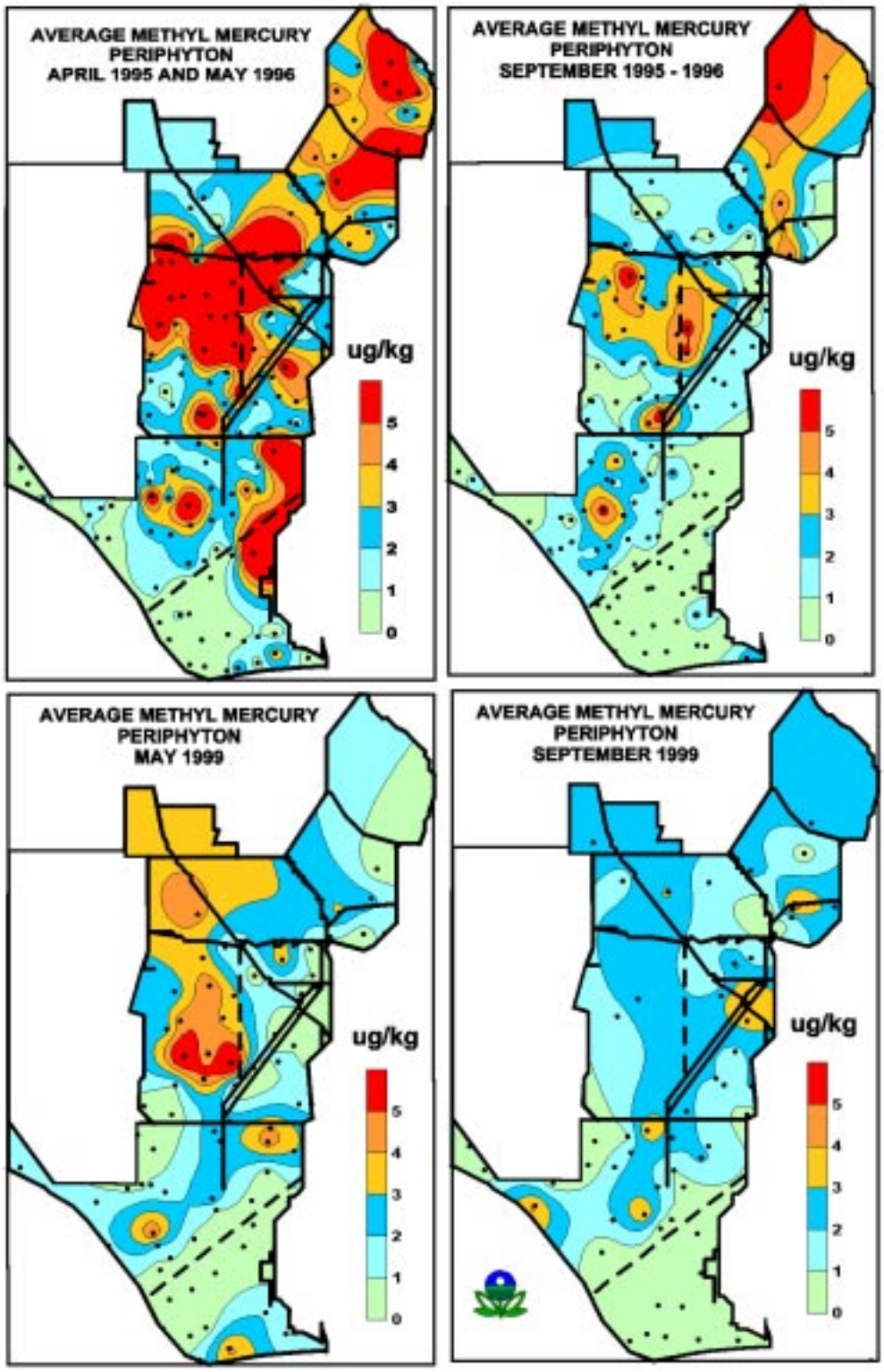


Figure 6.50. Spatial plots of average methyl mercury in periphyton measured during wet and dry seasons in phases 1 and 2.

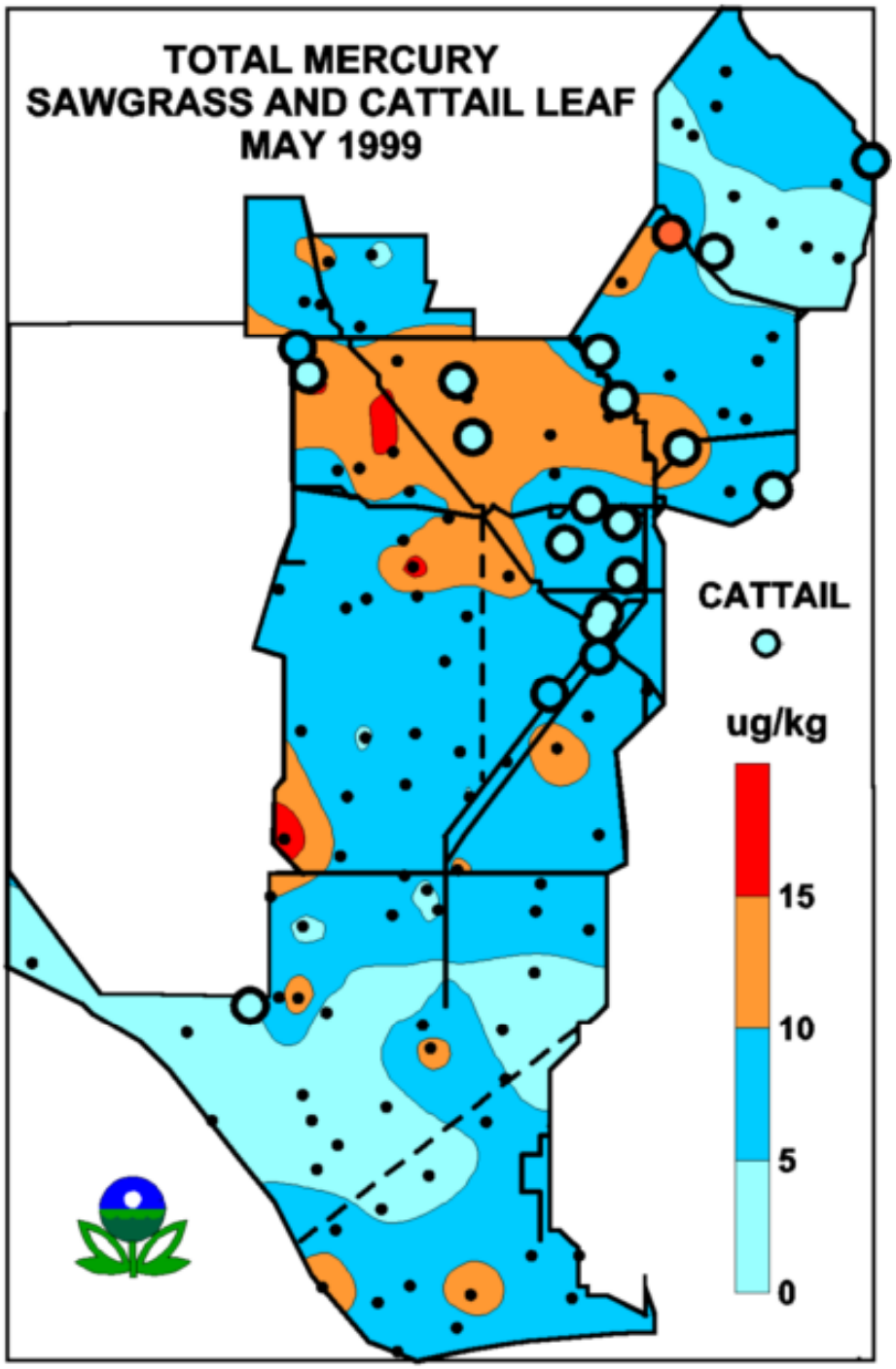


Figure 6.51. Spatial plots of total mercury in sawgrass measured May 1999 showing locations where cattails were collected with associated total mercury concentrations.

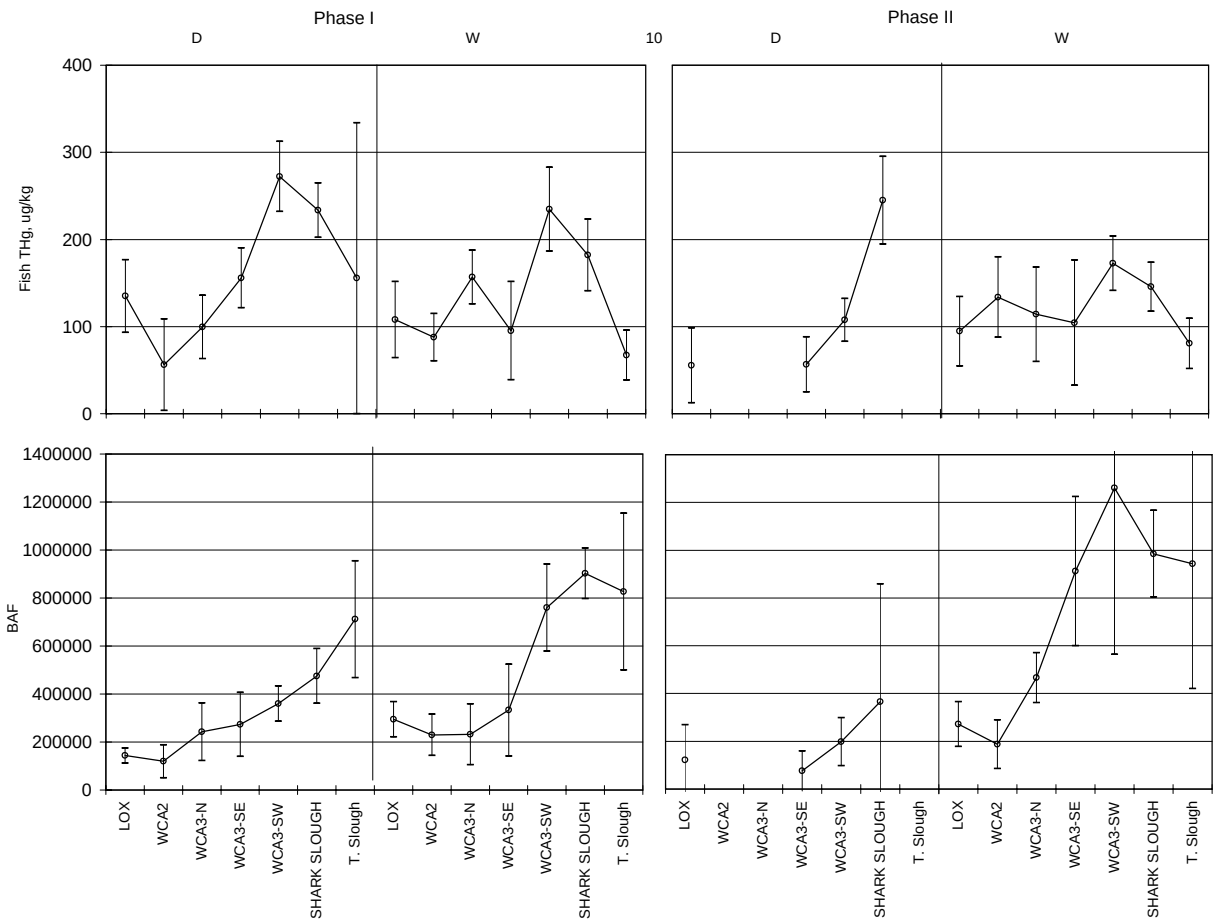


Figure 6.52. Median total mercury in gambusia and BAF measured in subareas during wet and dry seasons in phases 1 and 2.

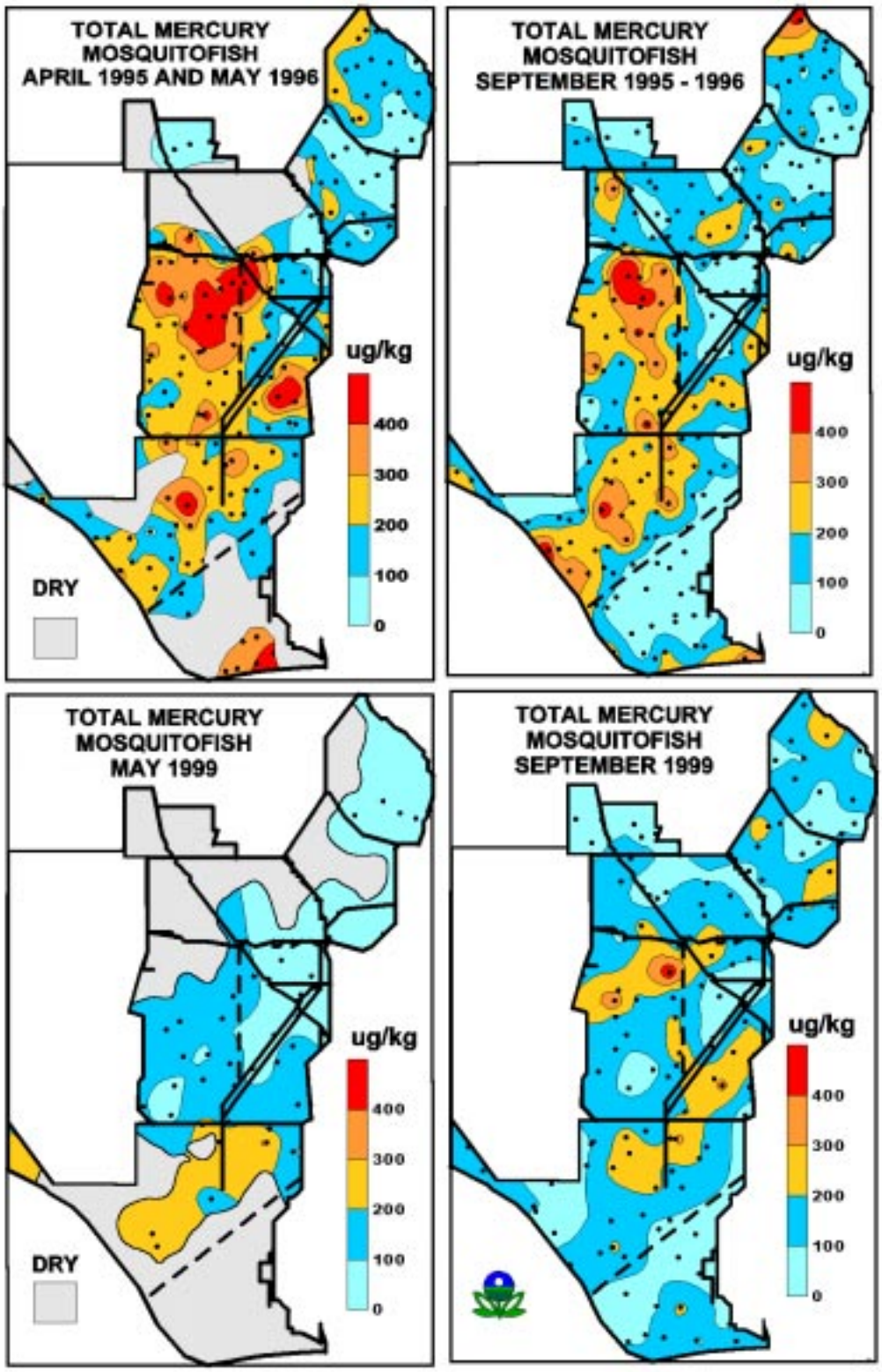


Figure 6.53. Spatial plots of total mercury in Gambusia measured during wet and dry seasons in phases 1 and 2.

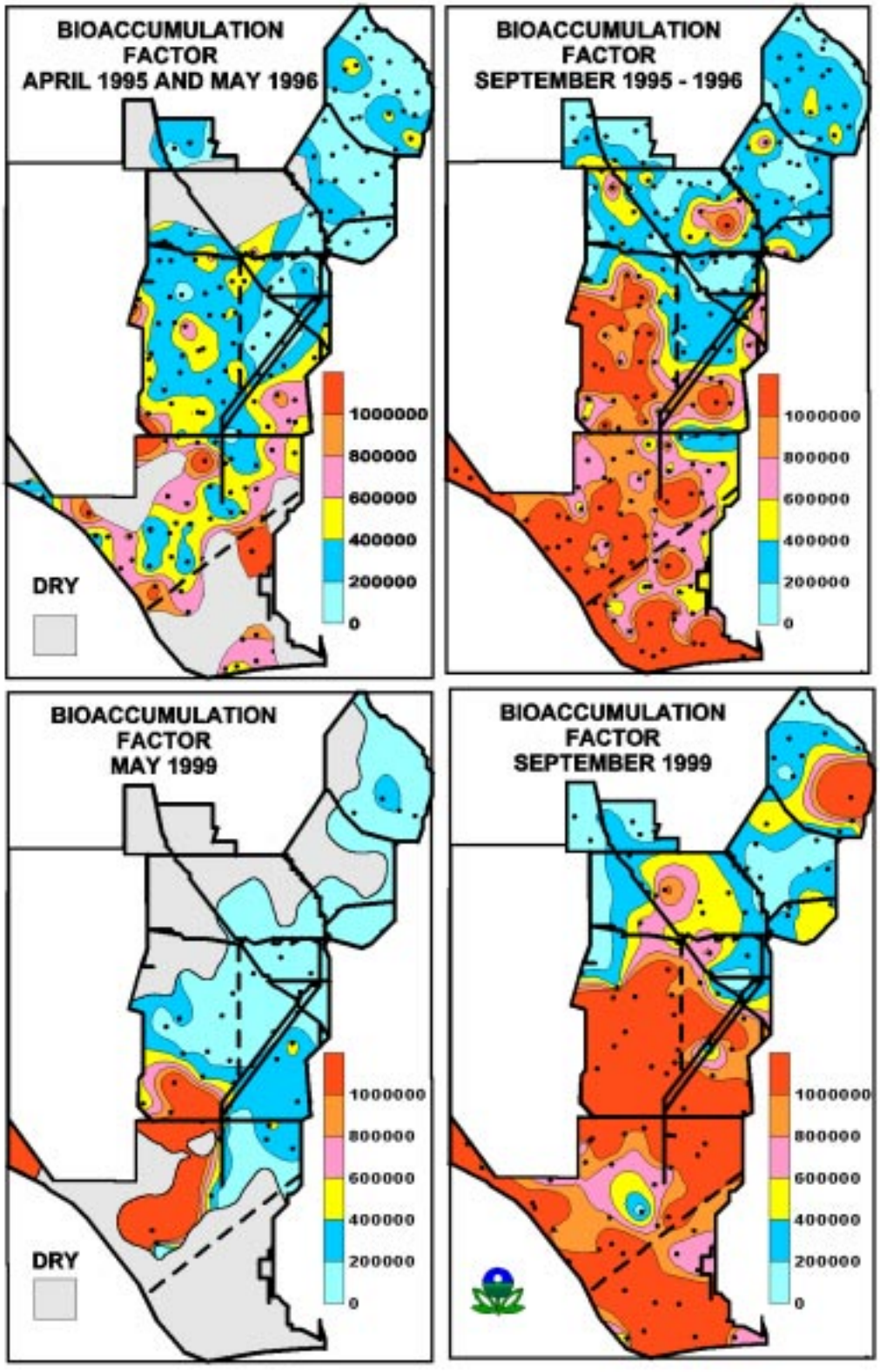


Figure 6.54. Spatial plots of BAF measured during wet and dry seasons in phases 1 and 2.

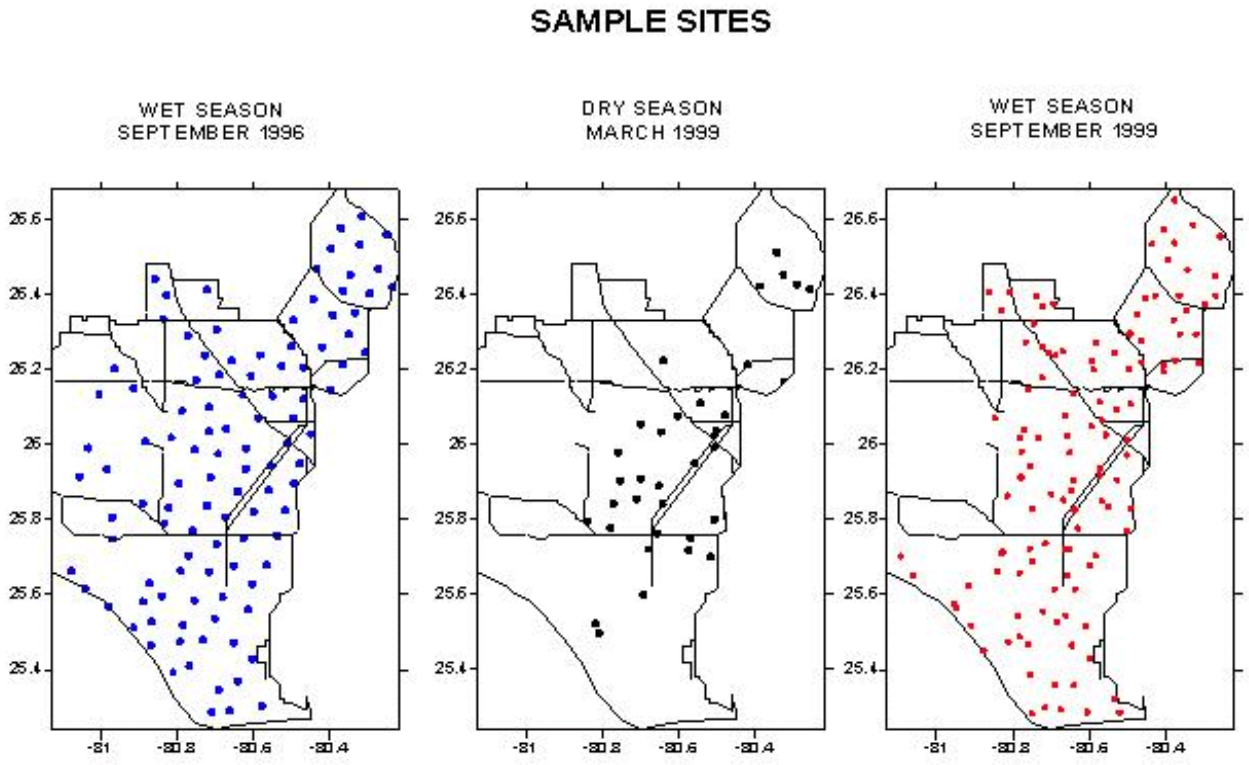


Figure 6.55 Maps of the synoptic sampling site locations where mosquitofish were collected for food habits analysis.

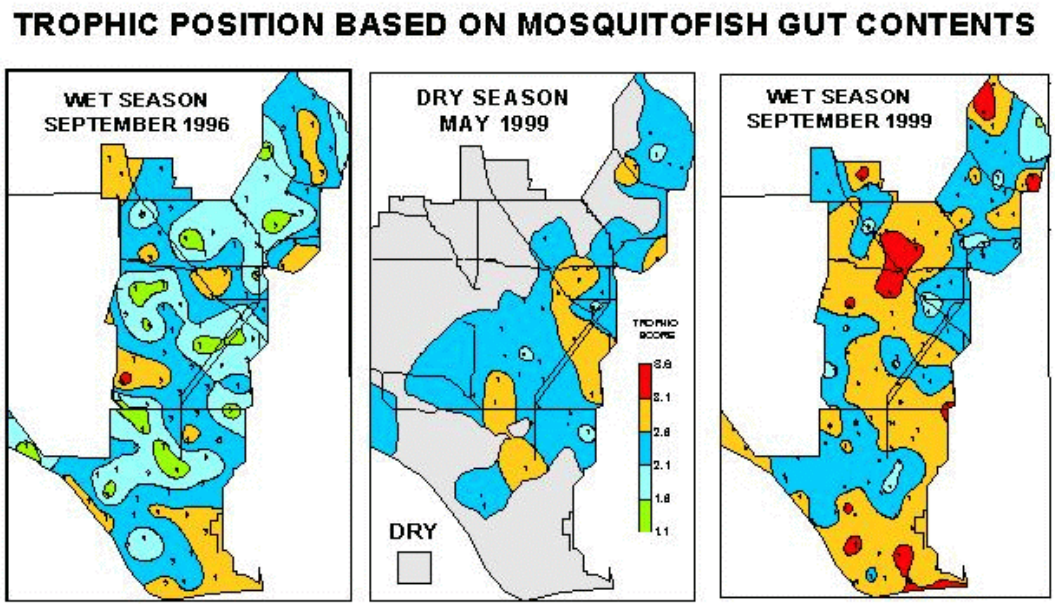


Figure 6.56 Maps of trophic score based on mosquitofish gut contents for each cycle sampled.

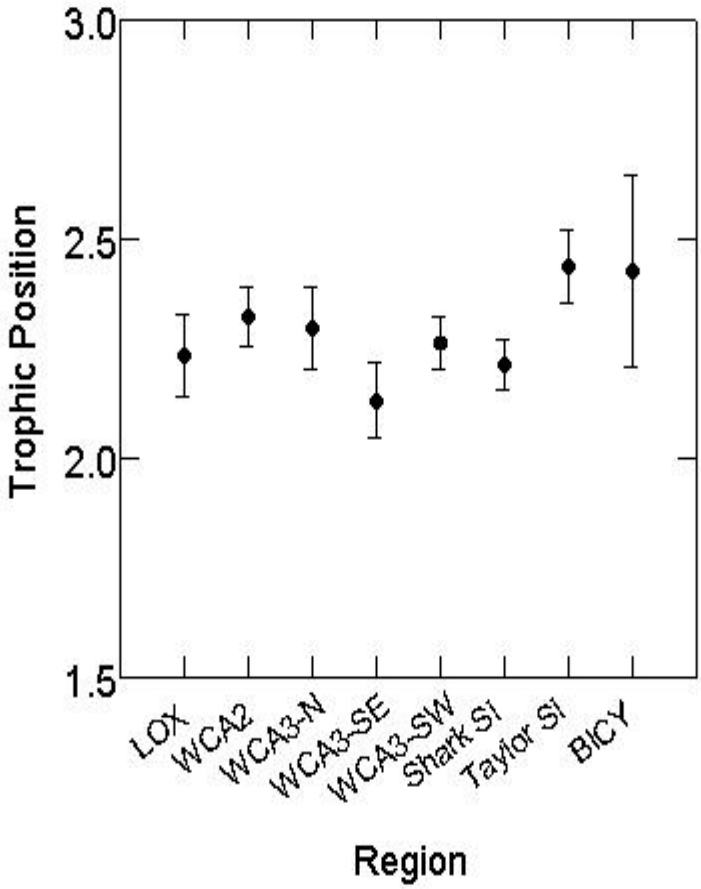


Figure 6.57 Least squares estimated means and 95% confidence intervals for trophic position of mosquitofish by study region. Abbreviations for the regions are: LOX (Loxahatchee NWR), WCA2, WCA3-N, WCA3-SE, WCA3-SW (Water Conservation Areas), Shark Slough, Taylor Slough, and BICY (Big Cypress National Preserve).

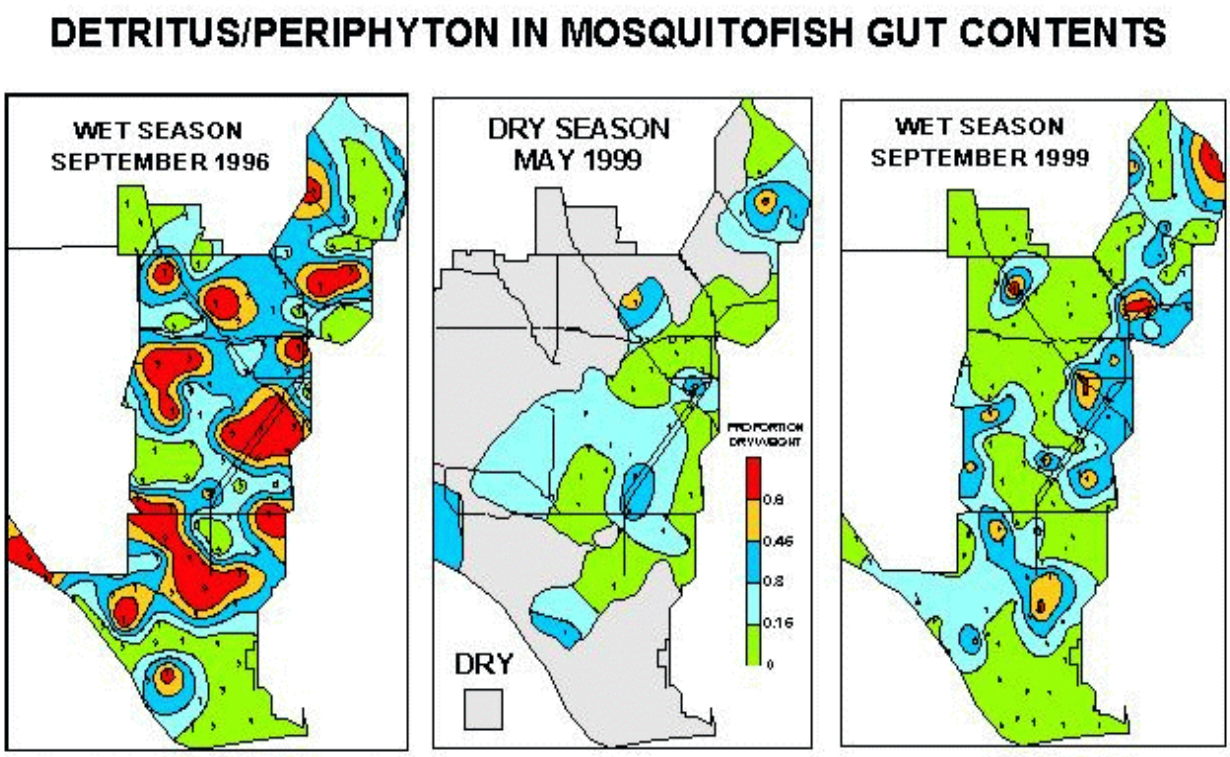


Figure 6.58 Maps of the frequency of detritus/periphyton in mosquitofish gut contents by cycle sampled.

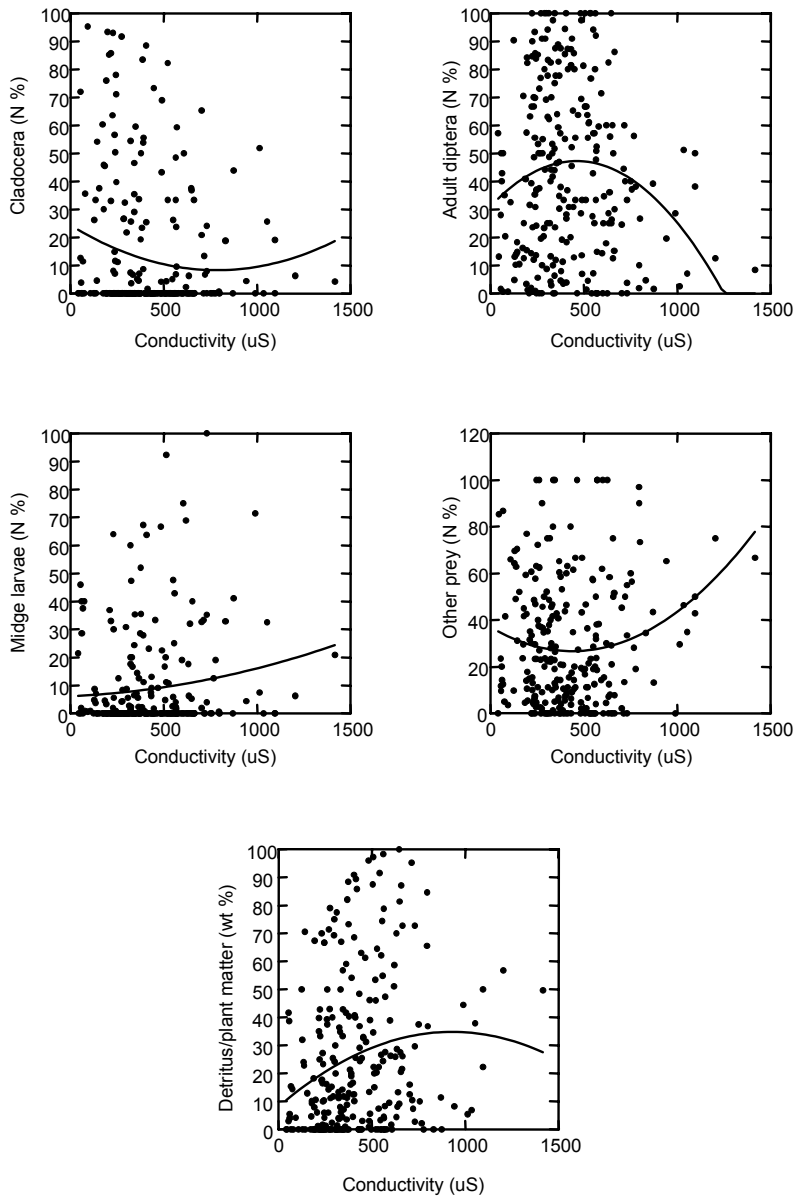


Figure 6.59 Relative abundance of each prey type plotted against conductivity at each site. A quadratic least-squares best-fit is plotted on each graph, all lines except midge larvae have slopes different than zero.

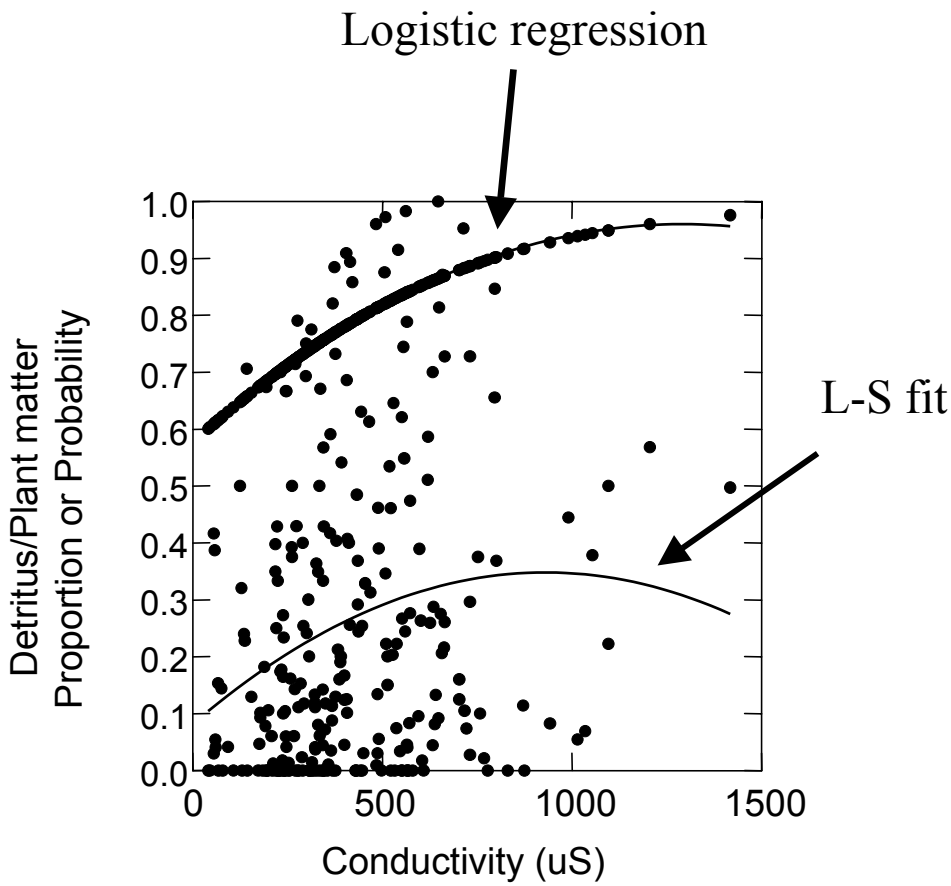


Figure 6.60 Detritus/plant matter in the diet of mosquitofish relative to conductivity at the collection site. The data points plotted indicate the proportion of detritus/plant matter in mosquitofish diets estimated by mass. The L-S (least-squares) best fit indicates the best estimate of proportion of this item in the diet. The logistic regression is probability of detritus/plant present (without regard to relative mass) in diet. Logistic regression indicates probability to increase from 0.6 to near 1.0 as conductivity increases from low to high. The logistic regression fits a binomial distribution using a maximum likelihood algorithm.

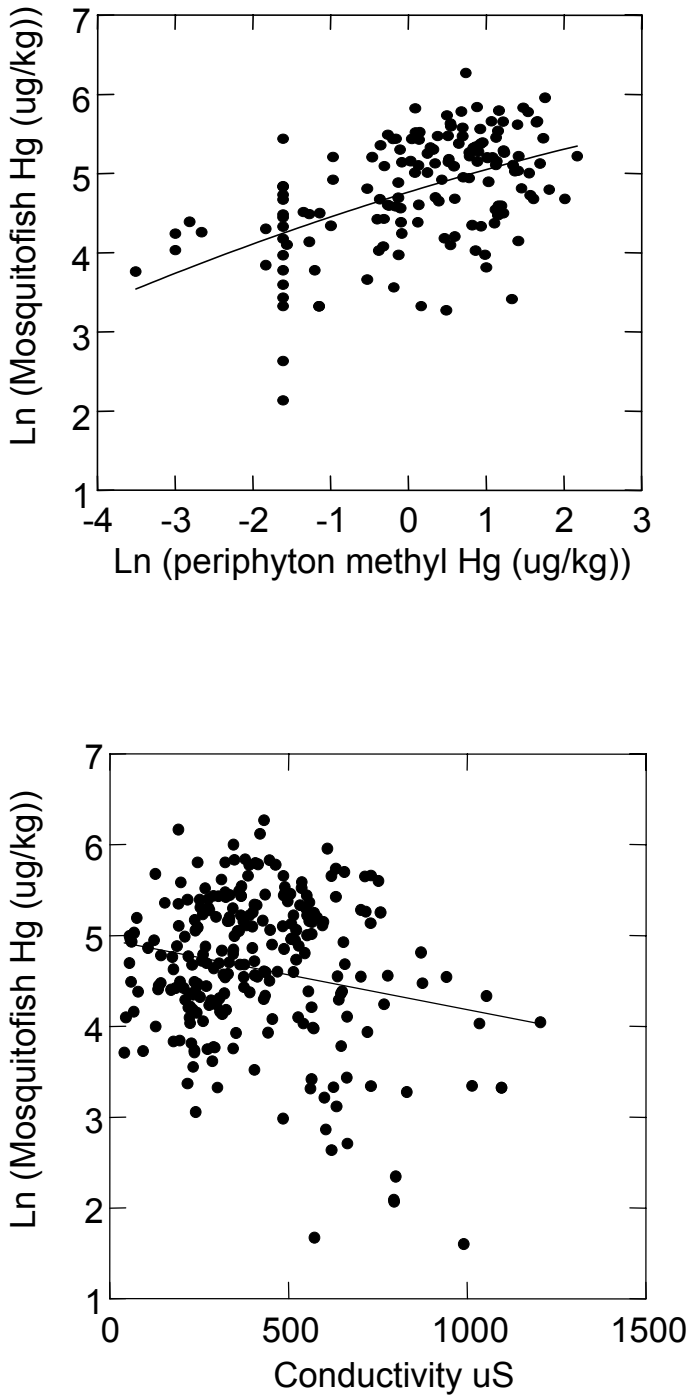


Figure 6.61 The relationship of the concentrations of total mercury in mosquitofish to methyl mercury in periphyton and conductivity at each site with least-squares best-fit lines.

Source	Effect	P	CD
Trophic score	-1.631	0.055	2.3%
Spatial division	-0.251	0.033	2.9%
Total P in water	0.352	0.120	1.5%

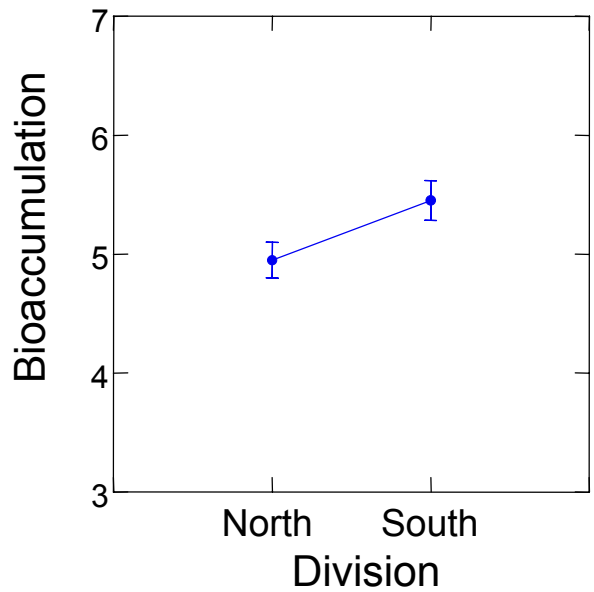


Figure 6.62. Mercury bioaccumulation estimated for mosquitofish collected south of I-75. North refers to fish collected in WCA3 between I-75 and Tamiami Trial while south refers to fishes collected south of Tamiami Trail in ENP. Mercury bioaccumulation = (fish total Hg) – (periphyton methyl mercury): N = 140; $R^2 = 0.055$.

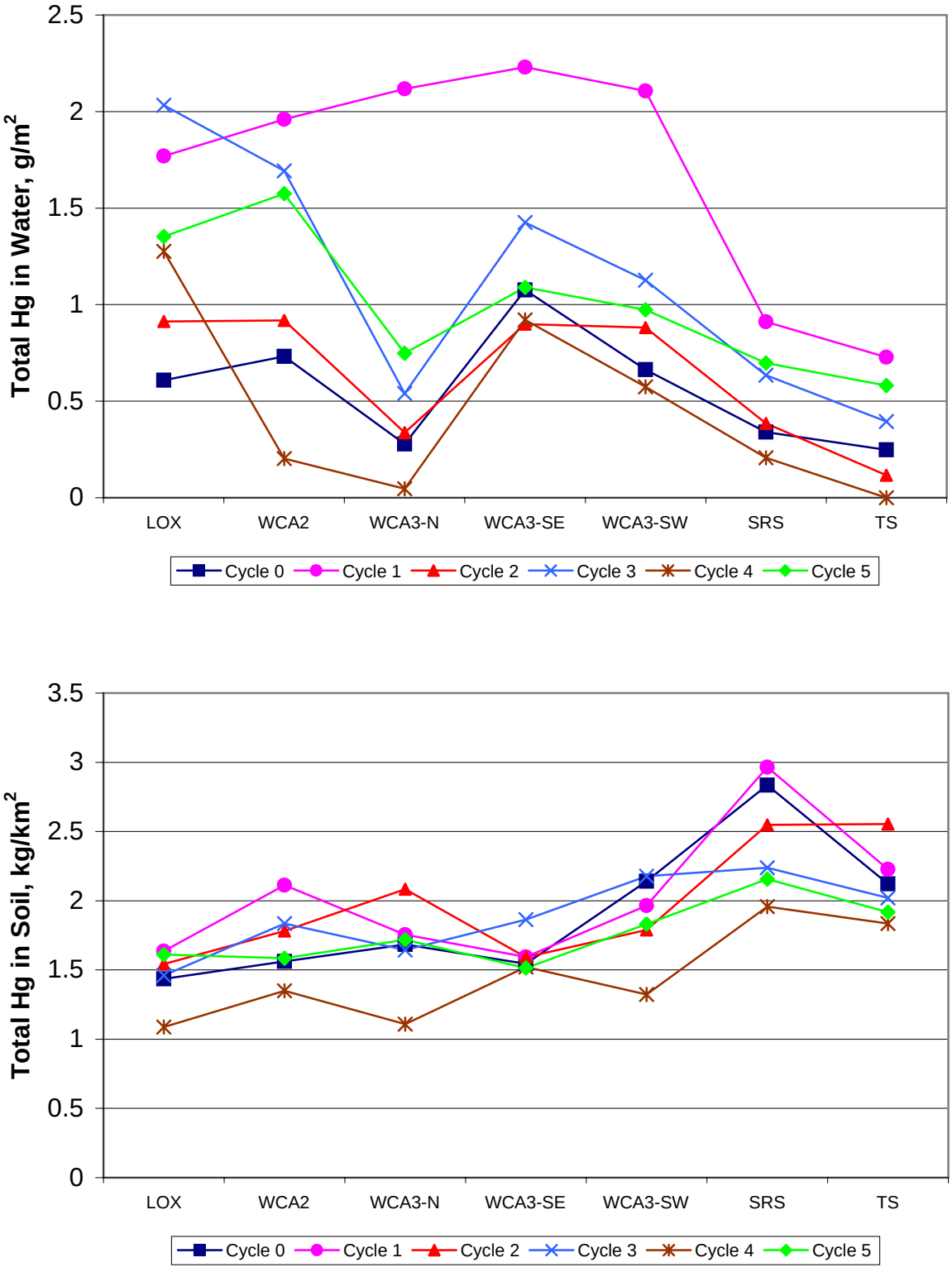
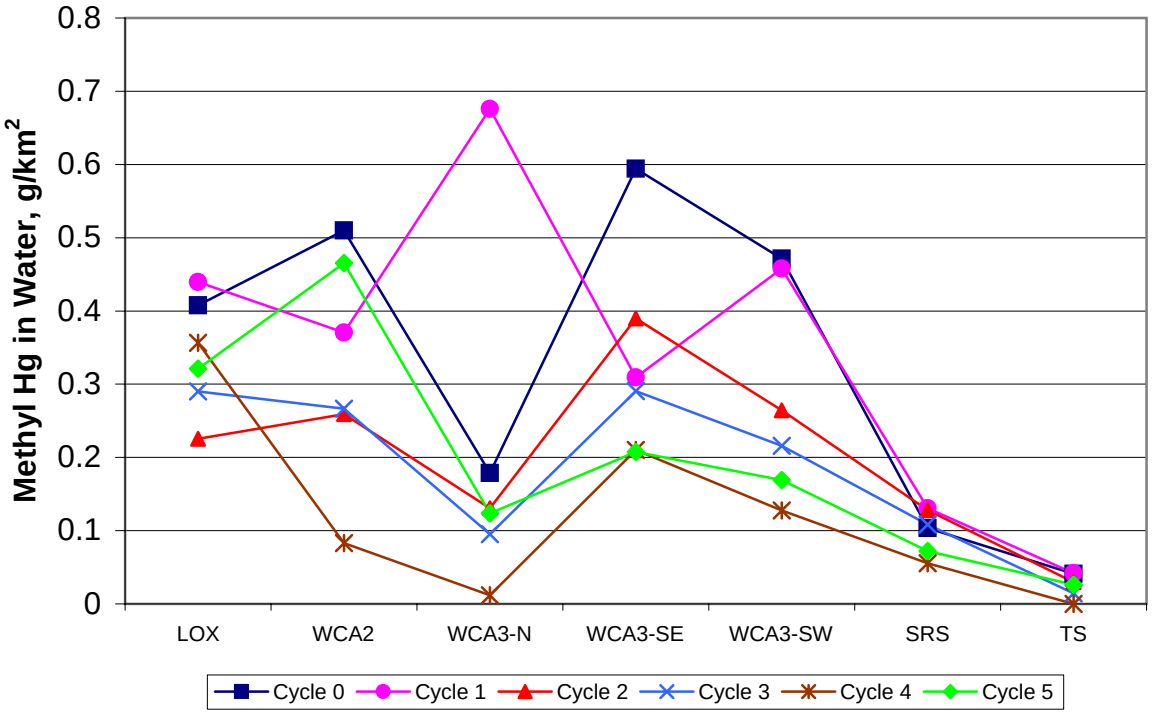


Figure 6.63. Total mercury mass estimates by marsh subarea and cycle in water (top) and soil (bottom).



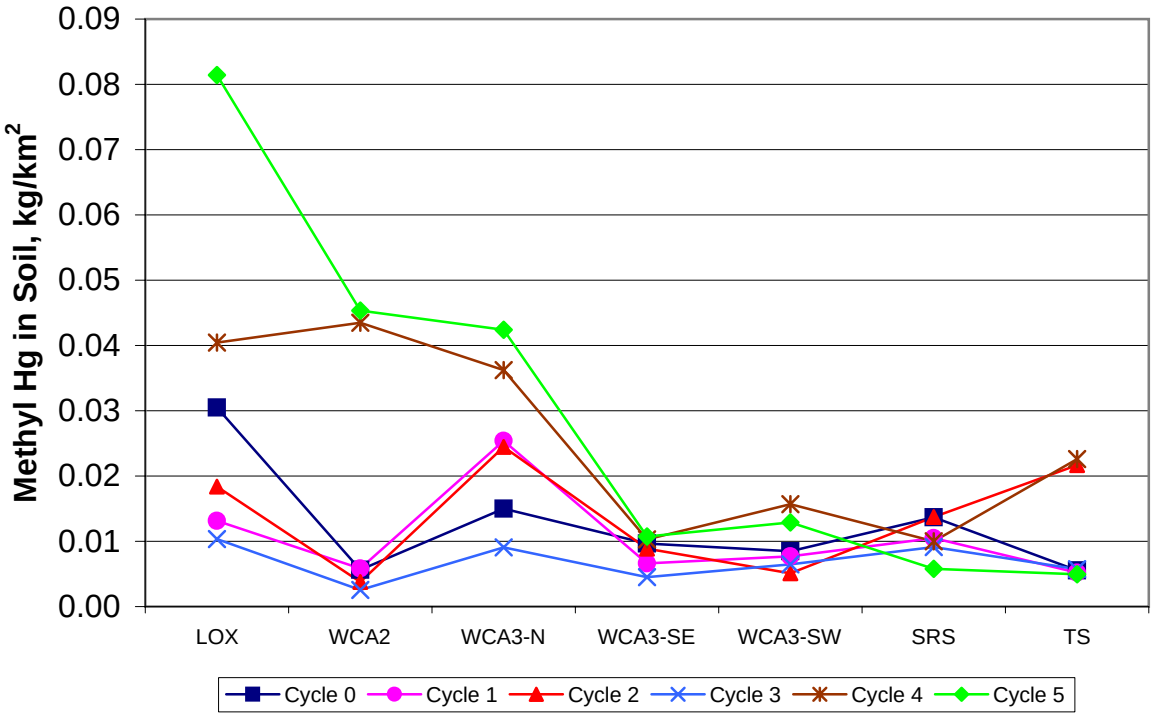


Figure 6.64. Methylmercury mass estimates by marsh subarea and cycle in water (top) and soil (bottom).

7.0 RISK HYPOTHESES ANALYSIS AND EVALUATION

7.1 Conceptual Models and Risk Hypotheses

7.1.1 South Florida Ecosystem Areas

Previous analyses and discussions have focused on seven subareas in South Florida that are demarcated by natural/artificial barriers or flow paths. This section will focus on three subareas in South Florida. The primary reason for decreasing the number of subareas from seven to three is to increase the number of sites or sample size per subarea considered in the analyses. With three subareas, there are typically 30 or more sites included in the analyses. This increased sample size provides greater explanatory power in statistical models formulated for these areas.

Alligator Alley (Interstate 75) and Tamiami Trail (US 41) form two barriers to flow from north to south through the South Florida Everglades ecosystem. The area north of Alligator Alley includes the Loxahatachee National Wildlife Refuge, WCA2, and the northern part of WCA3. The area between Alligator Alley and Tamiami Trail consists of WCA3, including both WCA3-SE and WCA3-SW. The area south of Tamiami Trail to the mangrove fringes is in the Everglades National Park and includes Shark Slough and Taylor Slough. Although there are control structures and culverts under Alligator Alley and Tamiami Trail to permit water movement, the northern portion of WCA3-SE and SW, between Alligator Alley and Tamiami Trail, is typically drier than natural conditions and the southern portion of this area, above Tamiami Trail, is wetter, with water ponding in the marsh just north of the Trail.

These three areas also respond differently to loadings from the EAA and other sources. Nutrient loading is greatest in the area north of Alligator Alley. As has already been shown, there is a mercury “hot spot” between the Alley and the Trail in WCA3-SW. The area south of the Trail in Shark and Taylor Sloughs has the lowest nutrient, sulfate and TOC concentrations, but intermediate fish mercury concentrations. Because these areas respond differently, management actions also are likely to elicit different responses in these three areas.

7.1.2 Conceptual Models

Different patterns in hydroperiod, water quality, soil constituents, and fish mercury became apparent during the Phase I analyses and were discussed in the Phase I report. Similar patterns were observed in the Phase II data. Based on these patterns, risk hypotheses were

formulated for each of these three areas. These risk hypotheses are illustrated by the conceptual models formulated for each of these three areas (Figure 7.1). The Phase I analyses focused primarily on the water pathways for mercury through the ecosystem. The Phase II analyses included potential soil pathways for methylmercury in addition to the water pathways. Other studies have indicated that the primary sites for methylation in the South Florida ecosystem are the soils, where porewater sulfide concentrations play a major role in controlling methylation rates (Benoit et.al., 1999, 2000; Gilmour et.al., 1998). Both the Phase I and Phase II studies represent synoptic surveys, rather than process-oriented studies. These surveys permit an evaluation of large scale patterns rather than small scale processes. However, comparisons with these smaller scale process studies can indicate the likelihood that the larger scale patterns result from these underlying processes.

Three conceptual models, hypothesized to describe the patterns and processes occurring in the South Florida Everglades ecosystem, are shown in Figure 7.1 and briefly discussed here. These risk hypotheses and pathways were evaluated using Principal Component Analyses (PCA) and path analyses, which are described in the next sections.

North of Alligator Alley, the system is dominated by the discharges from the EAA. TP, TN, TOC, sulfate, and sulfide concentrations were high in both the water and sediment. This is represented by the thick arrows shown in Figure 7.1. Total mercury concentrations in water were also relatively high in this area compared to areas south of Alligator Alley. Total mercury concentrations in soil, however, were moderate in WCA2 and low in WCA3-N compared to the other areas. Methylmercury concentrations north of the Alley were among the highest measured throughout the system during every season. However, fish mercury concentrations were relatively low in this area. Elevated sulfide and TOC concentrations likely act as ligands and chelate the mercury so that it is not readily available to aquatic organisms. High production in response to nutrient loading might also be contributing to biodilution of mercury through the aquatic food web.

Between Alligator Alley and Tamiami Trail, TP, TN, TOC, and sulfate concentrations decreased significantly. Methylmercury concentrations also decreased slightly, but fish mercury concentrations increased dramatically (Figure 7.1). The mercury “hot spot” for fish tissue concentrations occurred in this area. Both soil periphyton, floating periphyton mats and epiphytic assemblages were more abundant with a species composition that was more representative of the

historical Everglades assemblages, but still influenced by nutrient concentrations. Periphyton and floc (detritus) formed the base of the food web in this area. Uptake of methylmercury by the periphyton and sorption on to the floc provide a pathway for biomagnification of mercury through the food web. Periphyton methylmercury concentrations were highest in the southwestern portion of this area. Floc methylmercury concentrations were moderately high, but much lower than concentrations measured in the area North of Alligator Alley in WCA2 during the wet season.

South of Tamiami Trail, nutrient, TOC, and sulfate concentrations all decreased to levels more typical of the historical Everglades ecosystem (Figure 7.1). Methylmercury concentrations in both water, soil, floc, and periphyton were low; yet, fish mercury concentrations remained elevated. Fish mercury concentrations were only slightly lower in Shark Slough than at the hot spot in WCA3-SW. Bioaccumulation factors were highest in this area, indicating that food web complexity and biomagnification through the food web might be important processes for sustaining elevated mercury concentrations in fish. In addition, TOC and sulfide concentrations were lowest in this area, so the methylmercury that was produced, although in lower concentrations, might be more biologically available because of decreased interactions with these ligands.

7.1.3 Conceptual Model Testing

Several approaches were used to both develop and test the risk hypotheses or conceptual models. PCA was used to investigate the colinearity among variables and reduce the number of variables from 25-30 to 4-5 for additional consideration. General linear models (linear, stepwise and multiple regression models) were used to evaluate the relationship among various constituents demonstrated through laboratory or field process studies to influence the methylation of mercury and its subsequent transfer through the food web. Finally, structural equation models or path analyses were used to investigate multiple linkages and transfers through the ecosystem. The statistical approaches used and the rationale are included in Chapter 3, *Materials and Methods*.

7.2 Exploratory Analyses

A number of exploratory analyses were conducted to investigate the patterns observed in the data and gain a better understanding of the underlying processes that might be contributing to these patterns. Linear and multiple linear regression analyses were conducted based on the conceptual models described in previous section. In general, the relationships among total mercury in fish and other water quality variables, including methylmercury in periphyton were weak, with explained variance (i.e., R^2) coefficients of 0.1 to 0.36. Some of these relationships were statistically significant because of the large sample size, but were not considered ecologically significant.

7.2.1 South Florida Ecosystem

PCAs were conducted to investigate associations among variables for the entire South Florida ecosystem and for the three subareas defined by the location of Alligator Alley and Tamiami Trail (Tables 7.1 and 7.2, respectively). Comparing Phase I with Phase II for the entire South Florida marsh ecosystem indicated little association of fish total mercury with any combinations of inorganic, organic, or biotic variables (Table 7.1). During Phase I, the first principal component (PC) variables associated by media (i.e., water or soil) while the second PC variables showed an inverse association of biotic mercury and inorganic ligands in water with positive associations of soil methylmercury and soil periphyton methylmercury. During Phase II, there also was a general association by media of the first PC variables. The second PC associated primarily between biotic and abiotic variables. For Phase I, the first two components explained about 60% of the total variance, while in Phase II the first two components explained about 90% of the variance (Table 7.1). In both cases, only the first two components satisfied the Kaiser (1960) criterion with eigenvalues greater than 1 (i.e., if a factor does not extract at least as much information as the equivalent of one original variable, there is no reason to retain it).

Because of distinct north-south gradients and spatial patterns in many constituents, different associations or relationships among variables were expected within the three subareas. Therefore, PCAs were performed for the three subareas.

7.2.2 North of Alligator Alley

North of Alligator Alley, TOC, soil and water sulfate concentrations were closely associated with the first PC in both Phase I and Phase II, fish total mercury, and soil

methylmercury were associated with each other and inversely associated with TOC and sulfate in the first PC in both Phase I and Phase II (Table 7.2). The second PC variables were not closely associated in Phase I or II, although the soil variables were generally associated with the second PC in Phase I. The first two components explained about 55 to 60% of the variance for both Phase I and Phase II data. Periphyton assemblages typical of the Everglades ecosystem were rare north of Alligator Alley and were not considered in these analyses.

7.2.3 Alligator Alley to Tamiami Trail

Between Alligator Alley and Tamiami Trail, the first two components explained about 60% of the variance in Phase I, but almost 100% of the variance in Phase II. Associations among variables were different between Phase I and II in this subarea (Table 7.2). In Phase I, fish, floating periphyton, and water methylmercury were associated in the first PC. Soil variables such as soil periphyton methylmercury, soil sulfate, and AFDW also were associated in the first PC. Water variables (e.g., TOC, SO_4, floating periphyton methylmercury) were inversely associated with the soil variables in the second PC. In Phase II, the inorganic variables (with the exception of methylmercury in water) were closely associated with each other in the first PC (Table 7.2). The two periphyton groups were associated, and fish mercury and soil methylmercury were inversely associated with the other variables in the first PC. In the second PC, organic carbon variables (i.e., TOC, AFDW), sulfate variables, and periphyton variables were closely associated (Table 7.2). Total fish mercury and soil methylmercury were also associated in the second PC.

7.2.4 South of Tamiami Trail

South of Tamiami Trail, the first two components explained about 50% of the Phase I data and 80% of the Phase II data. In Phase I, fish and soil periphyton methylmercury, floating periphyton methylmercury and AFDW, and methylmercury in soil and water were associated variable pairs in the first PC (Table 7.2). In the second PC, soil periphyton and water methylmercury, sulfate in soil and water, and AFDW and soil methylmercury were associated pairs. Fish total mercury concentration was not closely associated with these pairs. In Phase II, fish mercury and TOC, and soil periphyton methylmercury and water sulfate pairs were associated with the first PC (Table 7.2). AFDW and soil methylmercury were inversely associated in the first PC. In the second PC, soil periphyton methylmercury and water sulfate

were again associated and AFDW and soil methylmercury were also inversely associated. Fish mercury was not closely associated with any of the variables.

In general, the ligands—AFDW, TOC, sulfate (surrogate for sulfide) in both soil and water—associated together. Associations among other variables were subarea and Phase specific. Because of the spatial patterns in these associations, structural equation models or path analyses were used to investigate the relationships among variables by Phase in these subareas.

7.3 Path Analysis

Structural equation models can be expressed with either standardized or reduced coefficients. Standardized coefficients are useful in evaluating the relative strength of the relationship among variables. Given a 1 unit standard deviation change in the independent variable, standardized coefficients represent the relative change in the dependent variable based on this unit change in standard deviation. For example, TP concentrations were associated with TOC concentrations North of Alligator Alley in both Phase I and Phase II (Figure 7.2). A one unit standard deviation change in TP in Phase I results in a 0.32 unit standard deviation change in TOC during Phase I and 0.74 unit standard deviation change in TOC in Phase II. The association between TP and TOC in Phase II was over twice as strong as it was in Phase I. Standard coefficients are used in this and the following sections so that the relative strength of associations among variables can be compared.

7.3.1 North of Alligator Alley

The risk hypotheses and conceptual model for the area north of Alligator Alley are shown in Table 7.3 and Figure 7.1, respectively. North of Alligator Alley, organic carbon, nutrient, and sulfate loading from the EAA dominated the area. TOC, TP, SO_4, Cl concentrations and conductivity were high in this area (See Chapter 6.0). Water quality patterns in the Refuge also reflected some of this loading, but primarily around the perimeter, with the interior of the Refuge being dominated by precipitation loadings. The Refuge is typically an acidic, oligotrophic system. However, during 1999, the Refuge also dried and exhibited water quality patterns that indicated that EAA loadings were influencing water quality in the interior of the Refuge. However, because the Refuge usually has water quality and flow patterns that are distinct from

the rest of the area north of Alligator Alley, the Refuge was treated separately in these analyses. The path analyses north of the Alley included primarily sites in WCA2 and WCA3-N.

During Phase I, there were positive associations among TP, TOC and methylmercury in water (Figure 7.2). The strength of the association between TOC and methylmercury (0.29) North of Alligator Alley was similar to the strength of association between TP and TOC (0.32) in Phase I. The TP-TOC association was over twice as strong in Phase II as in Phase I. During Phase II, however, the association between TOC and methylmercury concentrations in water was not statistically significant (Figure 7.2). Total mercury concentrations in water were positively associated with methylmercury concentrations in water during both Phases, but over twice as strong in Phase II.

Methylmercury concentrations in water were associated directly with total mercury concentrations in fish during both Phases (Figure 7.2). The methylmercury-fish mercury association, however, was about twice as strong in Phase II compared with Phase I. During Phase I, there was a negative association between TOC concentrations in water and fish total mercury.

Soil methylmercury concentrations were associated with soil TP concentrations in both Phases (Figure 7.2). However, in Phase I, the soil methylmercury concentrations were also associated with AFDW or organic carbon content of the soil. With the exception of the water sulfate-soil sulfide relationship, there were no statistically significant interactions between surface water and soil constituents north of Alligator Alley. Periphyton occurrence north of Alligator Alley was too sparse during both Phase I and II to be considered in the structural equation models (Figure 7.2).

In general, the associations among constituents north of Alligator Alley were relatively simple and linear. Chemical constituent concentrations were high in this area, reflecting the TP, TOC, and sulfate loadings from the EAA.

7.3.2 Alligator Alley to Tamiami Trail

Between Alligator Alley and Tamiami Trail, there were decreases in organic carbon, nutrient, and sulfate concentrations, but significant increases in fish total mercury concentrations in both phases. Methylmercury concentrations decreased only slightly below the elevated methylmercury concentrations measured north of Alligator Alley.

A greater number of pathways were statistically significant in the area between Alligator Alley and Tamiami Trail (Figure 7.3). During Phase I, there was a positive association of TP with TOC and negative association between TOC and water depth. TOC was also positively associated with methylmercury concentrations in water, but the association was over twice as strong as observed north of Alligator Alley. Both water depth and TP were positively associated, albeit weakly associated, with methylmercury concentrations in this area. Total mercury concentrations in water also were weakly associated with methylmercury concentrations. Sulfate was negatively associated with total mercury and methylmercury in this area.

Periphyton abundance was greater between the Alley and the Trail in Phase I and there was a positive association between methylmercury concentrations in water and floating periphyton mats (Figure 7.3). The strength of this association between methylmercury and floating periphyton was similar to the strength of the association between methylmercury and fish. However, there was no statistically significant association between floating periphyton methylmercury concentrations and fish total mercury concentrations, but there was a positive association between soil periphyton methylmercury concentrations and fish total mercury concentrations. The soil periphyton association, however, was about half the strength of the relationship between methylmercury concentrations in water and fish total mercury concentrations. There was an inverse relationship between soil periphyton methylmercury concentrations and water depth (Figure 7.3). There also was an association between soil methylmercury concentrations and fish total mercury concentrations, but no statistically significant association between soil methylmercury and soil periphyton methylmercury concentrations. Soil carbon content (AFDW) was positively associated with soil periphyton methylmercury concentrations. During Phase I, soil methylmercury concentrations were associated with soil TP and total mercury concentrations, but not carbon content.

During Phase II, there was also a positive association of TOC with TP concentrations and a negative association of TOC with water depth (Figure 7.3). As in the area north of Alligator Alley, during Phase II, there was no statistically significant association between methylmercury and TOC concentrations in water. Methylmercury concentrations were positively associated with sulfate and total mercury concentrations. Sulfate concentrations in water were positively associated with sulfide concentrations in soil, but negatively associated with total mercury concentrations in water, while sulfide in water was negatively associated with water depth. Soil

sulfate concentrations were positively associated with sulfide concentrations in both soil and water. Soil methylmercury concentrations were positively associated with soil TP concentrations and negatively associated with carbon content. There was no statistically significant association between soil methylmercury and fish total mercury concentrations during Phase II (Figure 7.3).

The periphyton abundance was insufficient during Phase II to evaluate periphyton associations with any other constituents (Figure 7.3). Fish total mercury concentrations were positively associated with methylmercury concentrations in water, and negatively associated with sulfide concentrations in both water and soil.

Compared to the area north of Alligator Alley, the complexity of pathways among constituents increased significantly, in both phases, between the Alley and Tamiami Trail. These pathways reflected by positive and negative (inverse) associations among constituents, with the primary inverse relationships occurring between sulfate and other constituents such as total mercury and methylmercury in water. There were also multivariate relationships between fish mercury concentrations and other constituents in both water and soil.

7.3.3 South of Tamiami Trail

South of Tamiami Trail, concentrations of all constituents are low, with the exception of fish total mercury concentrations. The pathways and interactions among constituents increased in complexity based on statistically significant pathways.

During Phase I, almost all associations among constituents were positive (Figure 7.4). There were positive associations between TP and TOC concentrations; between water depth, TP, TOC, total mercury, sulfate, and methylmercury concentrations; and between TP, TOC, and floating periphyton (PU) methylmercury concentrations (Figure 7.4). There was a positive association between TOC in water and soil periphyton methylmercury associations. There was a positive association between methylmercury in floating periphyton and soil methylmercury, but no associations between methylmercury in water and either periphyton assemblage or between soil methylmercury and soil periphyton (Figure 7.4). Fish total mercury concentrations were positively associated with methylmercury concentrations in water and in soil, but not with either periphyton assemblage.

Soil methylmercury concentrations were positively associated with carbon content and total mercury concentrations and negatively associated with sulfate concentrations (Figure 7.4).

During Phase II, almost all the associations were positive (Figure 7.4). The only negative association was between water depth and sulfide concentrations in water. TP and TOC were positively related and TOC, total mercury and sulfate were positively related to methylmercury concentrations in water (Figure 7.4). Soil methylmercury concentrations were positively associated with soil total mercury concentrations (Figure 7.4). Soil methylmercury concentrations were positively associated with soil total mercury concentrations. Methylmercury concentrations in water, soil, and floc were positively related to fish mercury concentrations. Fish total mercury concentrations also were positively associated with water depth and sulfide concentrations in the water. Unfortunately, there were insufficient periphyton assemblages at the sampling sites to evaluate periphyton associations with either chemical or biological constituents.

Regardless of the Phase, the associations among constituents were complex and positive in the oligotrophic area south of Tamiami Trail. Increased or decreased concentrations of almost any constituents in this area would be expected to result in a corresponding increase or decrease in methylmercury and fish total mercury concentrations.

The path analysis indicated that interactions among chemical and biological constituents are critical in understanding and managing mercury contamination in the South Florida Everglades ecosystem. In addition to multiple interactions, these relationships also change spatially throughout the system as the constituent concentrations change. This set of structural equation models provides one representation of the system, but there are other sets of risk hypotheses that also might be useful in understanding how the system responds to changes in water depth, nutrient, sulfate and TOC loading.

7.4 Alternative Risk Hypotheses and Paths

7.4.1 Alternative Structural Equation Models

Path analysis does not test causality, but rather whether the underlying data support the proposed model structure. Because several models might be supported by the data, it is useful to compare among the different model structures. For example, the explained variance (R^2) for two equations describing pathways for mercury in fish were similar in the Phase I area between Alligator Alley and Tamiami Trail (Figure 7.5).

For one model structure, the positive associations were among methylmercury in water, soil, and soil periphyton and fish total mercury concentration (Figure 7.5). For a second model structure, there were negative associations between TOC and sulfate with fish total mercury concentrations and a positive association between methylmercury concentrations in water and fish total mercury concentrations (Figure 7.5). The strength of the associations between methylmercury in soil and soil periphyton was similar to the strength of the negative associations between TOC and sulfate and fish total mercury concentrations. The strength of the association between water methylmercury concentration and fish mercury was stronger in the second equation than in the first, but both explained similar portions of the variance in fish mercury concentrations.

These two structural equation models indicate that it is likely there are alternative pathways for methylmercury from its formation to fish tissue concentrations. For example, the detritus food chain is not characterized in the Phase I or II data because constituents associated with this food chain or food web were not measured during the surveys. Alternative pathways through the producer and detrital food webs might be hypothesized because both water and soil methylmercury concentrations were associated with fish tissue mercury concentrations. Assessing food web dynamics is difficult with synoptic surveys, but these surveys clearly indicate the importance of considering selected process studies to investigate these associations. In addition, the spatial patterns apparent from the synoptic surveys provide insight into locations for conducting these studies.

7.4.2 Path Analysis Using Floc

During Phase II, floc samples were collected at all sites where water samples were collected. Floc sampling was described in *Chapter 3.0 Materials and Methods*. The floc samples might be used to represent a detritus-based food web. While the organisms feeding on detritus were not measured, these analyses might provide insight into detritus as a potential pathway for mercury bioaccumulation through the food web.

Additional structural equations were evaluated for floc relationships with other water quality constituents, water, soil, and periphyton methylmercury, and fish total mercury concentrations. Four additional structural equations were evaluated to determine whether the data supported hypotheses about the factors influencing floc methylmercury concentrations and

the potential for floc methylmercury to influence fish total mercury concentrations. Only one equation was significant and only in the area south of Tamiami Trail. Methylmercury concentrations in floc were positively associated with fish tissue mercury concentrations.

7.4.3 Path Analysis for WCA3-SE and WCA3-SW

Phase I and Phase II data in the area between Alligator Alley and Tamiami Trail were combined to increase the sample size. With a larger number of sites, the power of the statistical analyses was increased, which permitted investigating differences between WCA-SE and WCA-SW. The path analyses for these two areas indicated there were significant differences in the pathways and factors associated with various mercury species (Figure 7.6).

The WCA3-SE area is associated with the dominant flow path through the South Florida Everglades ecosystem. In WCA3-SE, TOC had a strong negative association with fish total mercury concentrations, a relatively weak positive relationship with total mercury concentrations in water, and a moderate positive relationship with methylmercury concentrations in water. There was an inverse relationship between water depth and TOC and between total mercury and sulfate concentrations in water in this area (Figure 7.6). There was no statistical association between soil constituents and water constituents.

In WCA3-SW, TOC interactions with methylmercury concentrations were weaker and there was no statistically significant relationship between TOC and fish mercury concentrations. The sulfur interactions were more pronounced in WCA3-SW than they were in the SE area, with a relatively weak, but positive relationship between sulfate and methylmercury concentrations, and a weak inverse relationship between total mercury and sulfate concentrations in water and water depth and sulfide concentrations by water. Water sulfate concentrations also were positively associated with porewater sulfide concentrations in the soil. In WCA3-SW, the soil carbon content (AFDW) was inversely related to soil methylmercury concentrations. In previous analyses, soil carbon content showed a positive relationship with methylmercury, if a statistical relationship was observed.

In both areas, methylmercury concentrations in water were the only mercury species associated with fish total mercury concentrations. Unfortunately, there were insufficient soil periphyton assemblages in each separate area to investigate periphyton associations with any statistical rigor.

Based on the path analyses, it appeared that TOC influenced the bioavailability of methylmercury in the southeastern portion of WCA3, while sulfate reduction and sulfur dynamics appeared to be more important in influencing the bioavailability of methylmercury in the southwestern portion of WCA3. While both sulfate and TOC are present in both areas, the relative importance of these constituents and their interactions did appear to vary between the two areas.

7.5 Synthesis

In general, the statistically significant paths associated with mercury dynamics were relatively sparse in the area north of Alligator Alley, where TOC, TP, sulfate, sulfide, and other chemical constituent concentrations were high; relatively complex between the Alley and the Trail, where chemical constituent concentrations were changing dramatically; and nearly all positive in the area south of Tamiami Trail, where chemical constituent concentrations were low, the system was ultra-oligotrophic, and biological food web complexity was high. There was no single constituent or path that represented the dominant relationship throughout the South Florida Everglades ecosystem.

The paucity of significant interactions north of Alligator Alley needs to be considered cautiously. Regression and association analyses are based on gradient or variation in responses. TOC and sulfate concentrations north of Alligator Alley are high, with less variability than is found in other subareas within the system.

In all areas and in both Phases, water depth was associated with a number of constituents that influenced methylmercury species. Although water depth is not equivalent to hydroperiod, it might serve as a surrogate, which would indicate that system mercury responses might be expected to be influenced by hydroperiod. In addition, there were interactions among the inorganic ligands, TOC, sulfide, and soil organic content (AFDW) and total mercury, and methylmercury in water and soil. The interactions among hydropattern and nutrient, organic carbon, and sulfate loadings from the EAA, with mercury contamination change from north to south in the South Florida ecosystem.

“Top down” versus “bottom up” is a concept used to explain how control of patterns and processes in aquatic systems changes during eutrophication or as nutrient loading to a system increases (Carpenter et al., 1985 and 1995). Some of these ecological attributes are compared

between oligotrophic and eutrophic systems in Table 7.4. The comparison is relevant because eutrophication, in part, affects mercury contamination patterns and processes and because the South Florida ecosystem shows the entire gradient from eutrophic in the north to oligotrophic in the south. The concept is a useful analog for understanding mercury contamination.

Oligotrophic systems can be viewed as "top-down" controlled ecosystems. Characteristics of top-down control are: 1) nutrient cycles are tightly coupled because nutrients are limiting; 2) biotic-abiotic interactions control the response of the ecosystem; and 3) the variability in biomass production is relatively small, varying by a factor of only 4 to 5 over the year (Table 7.4). Oligotrophic systems usually have a seasonal renewal of nutrients, such as during the rainy season. The predictability of the response of oligotrophic ecosystems is relatively low because there are multiple factors that control the interactions among biotic and abiotic constituents and we don't understand these interactions very well (Table 7.4).

Eutrophic systems can be viewed as "bottom-up" controlled ecosystems. Characteristics of bottom-up control include: 1) nutrient cycles are leaky and decoupled from higher levels in the food chain; 2) physical factors such as inflow, hydrodynamic mixing and sedimentation control system responses; and 3) there typically are large variations in biomass production, varying by over an order of magnitude throughout the year (Table 7.4). Nutrients are supplied primarily through inflows and are relatively continuous throughout the year. The predictability of the system response is relatively high. Statistical relationships between nutrient loads and biomass can be developed (i.e., Vollenweider-type nutrient loading models) (Table 7.4).

North of Alligator Alley, the marsh is eutrophic, chemical constituent concentrations are high (e.g., TP, TOC, SO_4), and chemical interactions appear to control mercury bioavailability and bioaccumulation (i.e., bottom-up), the food web in this eutrophic area is likely impacted by the organic loadings.

South of Tamiami Trail, the marsh is oligotrophic, chemical constituent concentrations are low, and biotic-abiotic interactions are likely much more tightly coupled (i.e., top-down). Although methylmercury concentrations are low, more of this methylmercury is likely biologically available and bioaccumulated and biomagnified through the food web. The methylmercury BAF is significantly higher in this area than in the north.

Between the Alley and the Trail, the system is in transition between a eutrophic and oligotrophic ecosystem. Productivity is still stimulated by nutrients, but chemical interactions

and interferences with methylmercury bioavailability and bioaccumulation have decreased, methylmercury concentrations are high, and mosquitofish mercury concentrations are at their highest values. Food webs are likely more tightly coupled, contributing to the elevated fish mercury concentrations. Transition areas typically are dynamic and have characteristics of both eutrophic and oligotrophic ecosystems.

Understanding some of the eutrophication processes helps our understanding of mercury contamination. For example, the path analyses indicated that the area between Alligator Alley and Tamiami Trail was dynamic, with multiple pathways and interactions among chemical constituents, methylmercury in water and soil, periphyton, and fish mercury concentrations. North of Alligator Alley, where the system was eutrophic and chemical constituent concentrations were high, the pathways were simple. South of Tamiami Trail, where the system is oligotrophic, the pathways are relatively complex, with both floc and water methylmercury concentrations associated with fish mercury concentrations. These analyses indicated that both detrital and autotrophic pathways contributed to fish mercury concentrations. Brumbaugh et al. (2001) also found that the associations of fish mercury were strongly correlated with water methylmercury concentrations in a national study of 21 NAWQA watersheds.

Table 7.1. Eigenvectors for the first (PC1) and second (PC2) principal components between Phase I and Phase II for selected variables.

Constituent	Phase I		Phase II	
	PC1	**PC2**	**PC1**	**PC2**
THg-Fish	0.23	0.59	0.03	-1.00
Soil Periphyton-MeHg	0.60	0.41	0.69	-0.56
Float Periphyton-MeHg	0.78	0.17	0.63	-0.72
TOC	0.67	-0.34	0.86	0.51
SO_4	0.44	-0.72	0.86	0.21
MeHg	0.86	-0.12	0.88	0.46
AFDW-Soil	0.79	0.20	0.91	-0.40
SO_4-Soil	0.55	-0.43	0.83	0.55
MeHg-Soil	0.61	0.36	-0.37	0.66
% Variance Explained	41	17	53	36

Table 7.2. Eigenvectors for the first (PC1) and second (PC2) principal components from analysis of biotic and abiotic characteristics for the three subareas in Phase I and Phase II.

Constituent	Phase I		Phase II	
	PC1	PC2	PC1	PC2
North of Alligator Alley				
Thg-Fish	-0.42	0.35	0.63	0.54
TOC	0.77	0.22	-0.76	0.19
SO_4	0.89	-0.03	-0.79	0.22
MeHg	0.13	0.69	0.15	0.83
AFDW	-0.11	0.52	0.44	0.57
SO_4-Soil	0.53	0.54	-0.78	0.44
MeHg-Soil	-0.51	0.61	0.51	-0.11
% Variance Explained	31	23	38	23
Alligator Alley to Tamiami Trail				
THg-Fish	0.73	0.36	-0.44	0.90
Soil Periphyton - MeHg	0.56	0.56	0.76	0.65
Float Periphyton - MeHg	0.75	-0.25	0.82	0.57
TOC	0.61	-0.52	0.99	0.10
SO_4	0.12	-0.72	0.90	-0.44
MeHg	0.81	-0.38	0.99	0.14
AFDW	0.51	0.38	1.00	0.07
SO_4-Soil	0.49	0.16	1.00	-0.03
MeHg-Soil	0.39	0.12	-0.54	0.84
% Variance Explained	35	18	72	28
South of Tamiami Trail				
THg-Fish	0.51	-0.04	0.76	0.54
Soil Periphyton - MeHg	0.49	-0.36	0.90	0.19
Float Periphyton - MeHg	0.73	0.21	–	–
TOC	0.24	-0.20	0.76	0.36
SO_4	0.03	0.74	0.98	0.17
MeHg	0.58	-0.33	-0.02	-0.89
AFDW	0.75	0.35	0.58	-0.82
SO_4-Soil	-0.34	0.77	0.33	-0.33
MeHg-Soil	0.67	0.40	-0.57	0.82
% Variance Explained	28	19	46	34

Table 7.3. Structural equations and risk hypotheses.

North of Alligator Alley
TOC = C + TP + Water Depth THg-W = TOC+SO_4+S^{2-} THg-Soil = AFDW+SO_4-Soil+S^{2-}Soil MeHg-W = C+TOC+SO_4+TP+THg+S^{2-}+Water Depth+SO_4-Soil MeHg-Soil = C+AFDW+$_{S2-}$+TPS+THgS+SO_4-Soil S^{2-}-W = C+SO_4+Water Depth+SO_4-Soil S^{2-}-Soil = C+SO_4+AFDW+SO_4-Soil THg-FISH = C+TOC+SO_4+S^{2-}+Water Depth+MeHg-W+MeHg-Soil+S^{2-}-Soil+SO_4-Soil
Alligator Alley to Tamiami Trail
TOC = C + TP + Water Depth THg-W = TOC+SO_4+S^{2-} THg-Soil = AFDW+SO_4-Soil+S^{2-}Soil MeHg-W = C + TOC+SO_4+TP+THg+S^{2-}+Water Depth+SO_4-Soil MeHg-Soil = C+AFDW+$_{S2-}$+TPS+THgS+SO_4+SO_4-Soil S^{2-}-W = C+SO_4+Water Depth+SO_4-Soil S^{2-}-Soil = C+SO_4+AFDW+SO_4-Soil MeHg-PU = C+TOC+SO_4+S^{2-}+TP+MeHg-W+MeHg-Soil+Water Depth+SO_4-Soil MeHg-PS = C+TOC+SO_4+S^{2-}+TP+AFDW+TP-Soil+MeHg-W+MeHg-Soil+S^{2-}+Water Depth+SO_4-Soil THg-FISH = C+TOC+SO_4+S^{2-}+Water Depth+MeHg-W+MeHg-Soil+S^{2-}-Soil+SO_4-Soil THg-Fish = C+TOC+SO_4+S^{2-}+Water Depth+MeHg-W+MeHg-Soil+MeHg-PU+SO_4-Soil THg-Fish = C+TOC+SO_4+S^{2-}+Water Depth+MeHg-W+MeHg-Soil+MeHg-PS+SO_4-Soil THg-Fish = C+TOC+SO_4+S^{2-}+Water Depth+MeHg-W+MeHg-Soil+MeHg-PU+MeHg-PS+SO_4-Soil
South of Tamiami Trail
TOC = C + TP + Water Depth THg-W = TOC+SO_4+S^{2-} THg-Soil = AFDW+SO_4-Soil+S^{2-}Soil MeHg-W = C + TOC+SO_4+TP+THg+S^{2-}+Water Depth+SO_4-Soil MeHg-Soil = C+AFDW+S^{2-}+TPS+THgS+SO_4+SO_4-Soil S^{2-}-W = C+SO_4+Water Depth+SO_4-Soil S^{2-}-Soil = C+SO_4+AFDW+SO_4-Soil MeHg-PU = C+TOC+SO_4+S^{2-}+TP+MeHg-W+MeHg-Soil+Water Depth+SO_4-Soil MeHg-PS = C+TOC+SO_4+S^{2-}+TP+AFDW+TP-Soil+MeHg-W+MeHg-Soil+S^{2-}+Water Depth+SO_4-Soil THg-FISH = C+TOC+SO_4+S^{2-}+Water Depth+MeHg-W+MeHg-Soil+S^{2-}-Soil+SO_4-Soil THg-Fish = C+TOC+SO_4+S^{2-}+Water Depth+MeHg-W+MeHg-Soil+MeHg-PU+SO_4-Soil THg-Fish = C+TOC+SO_4+S^{2-}+Water Depth+MeHg-W+MeHg-Soil+MeHg-PS+SO_4-Soil THg-Fish = C+TOC+SO_4+S^{2-}+Water Depth+MeHg-W+MeHg-Soil+MeHg-PU+MeHg-PS+SO_4-Soil

Table 7.4 Comparison of processes and patterns between oligotrophic and eutrophic systems.

Ecological Attribute	Oligotrophic Systems	Eutrophic Systems
Controlling Factors	"Top-down"	"Bottom-up"
Nutrient Cycling	Tightly coupled nutrient cycles-algae-grazers-microbes, regenerated in water columns	Loose nutrient cycling–decoupled from higher food chain, supplied from inflow, sediment cycling
Forcing Functions	Biotic-abiotic interactions	Physical factors–inflow, hydrodynamic mixing
Temporal Patterns	Relatively small biomass variability	Large biomass variability
Nutrient Requirements	Seasonal renewal	Continuous supply
Predictability	Low-multivariate relationships among biomass and controlling factors not well understood	High-statistical relationships between nutrient loads and biomass

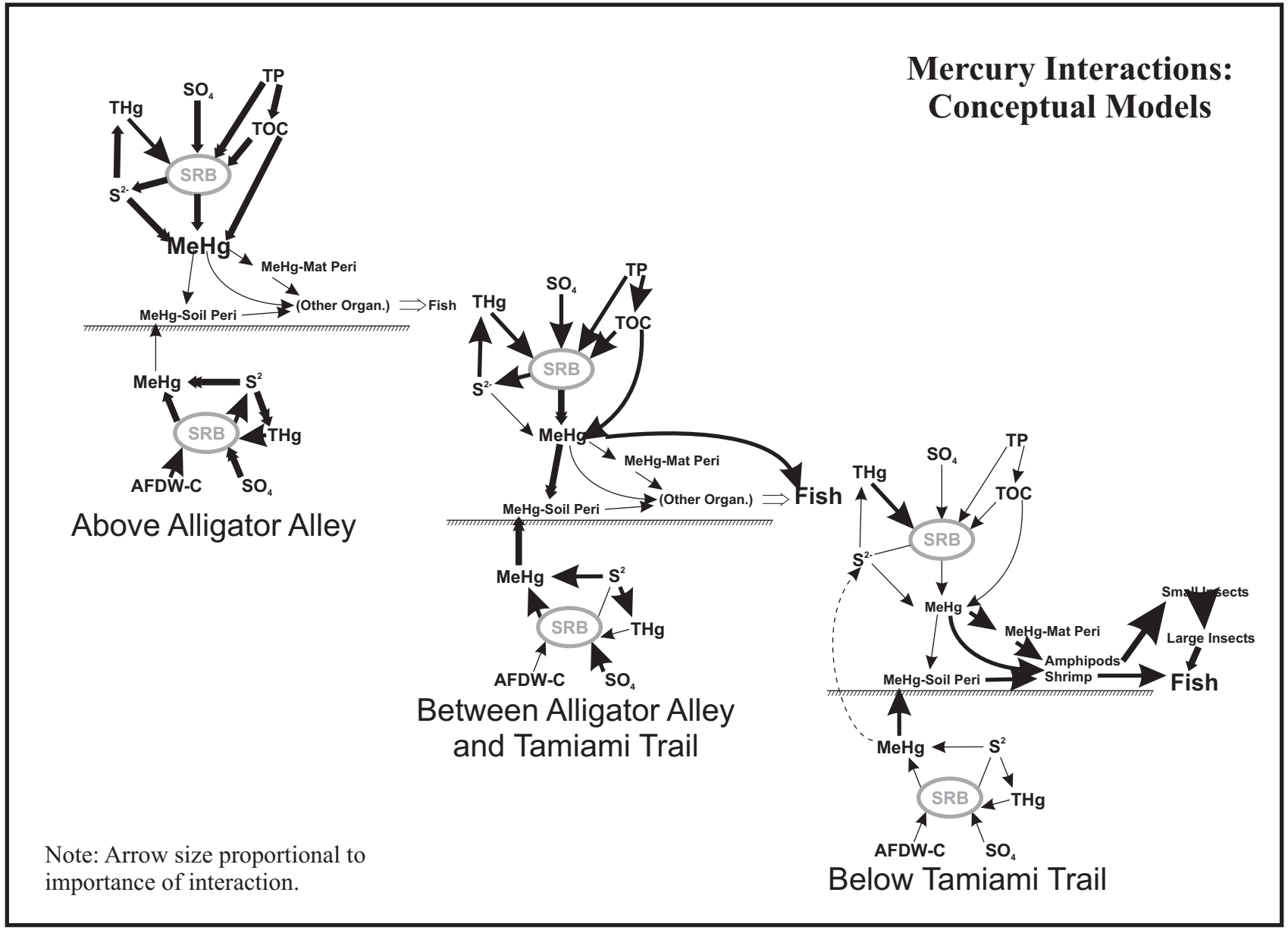


Figure 7.1. Conceptual models of mercury interactions in three areas of South Florida formed by Alligator Alley (I-75) and Tamiami Trail (US Hwy 41).

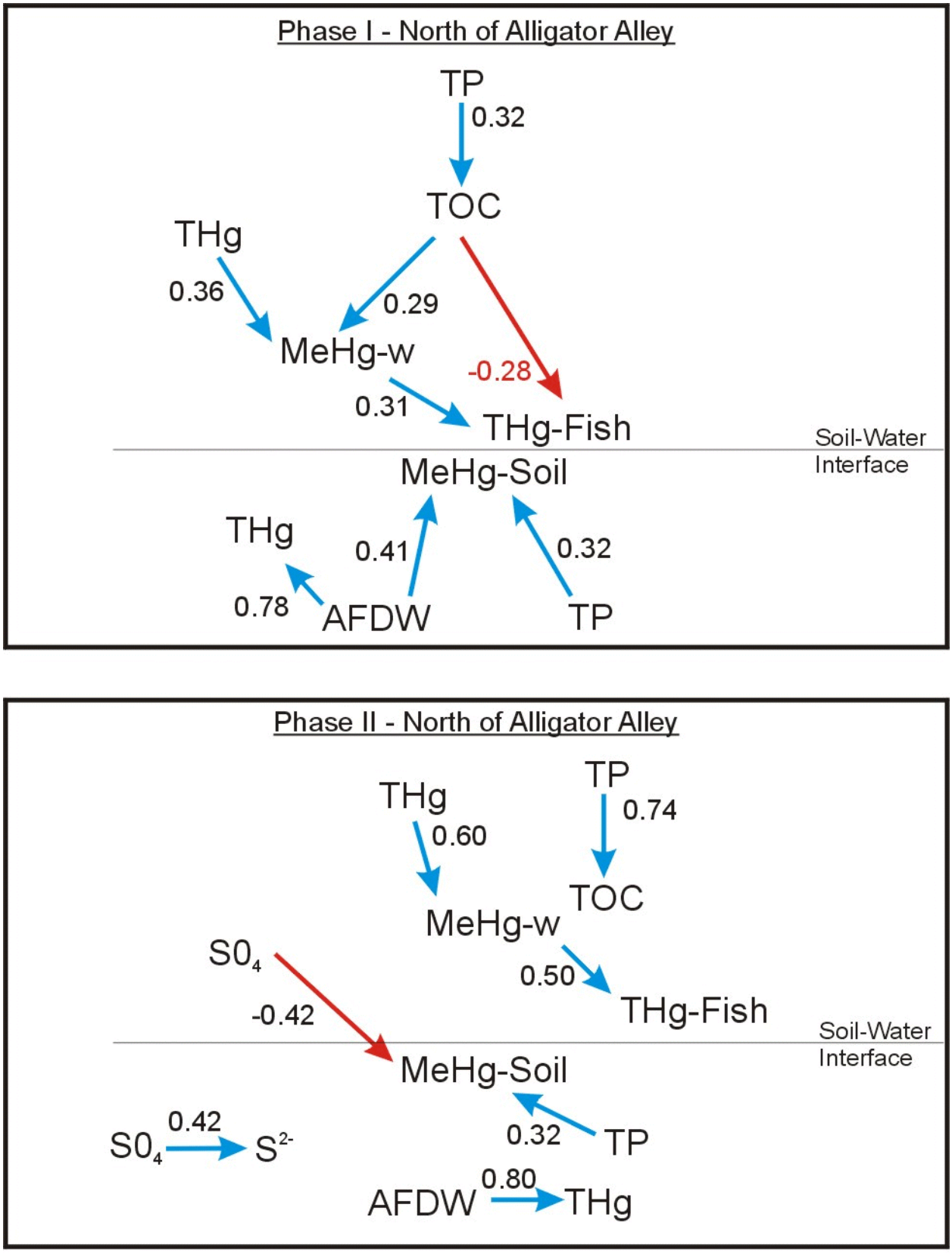
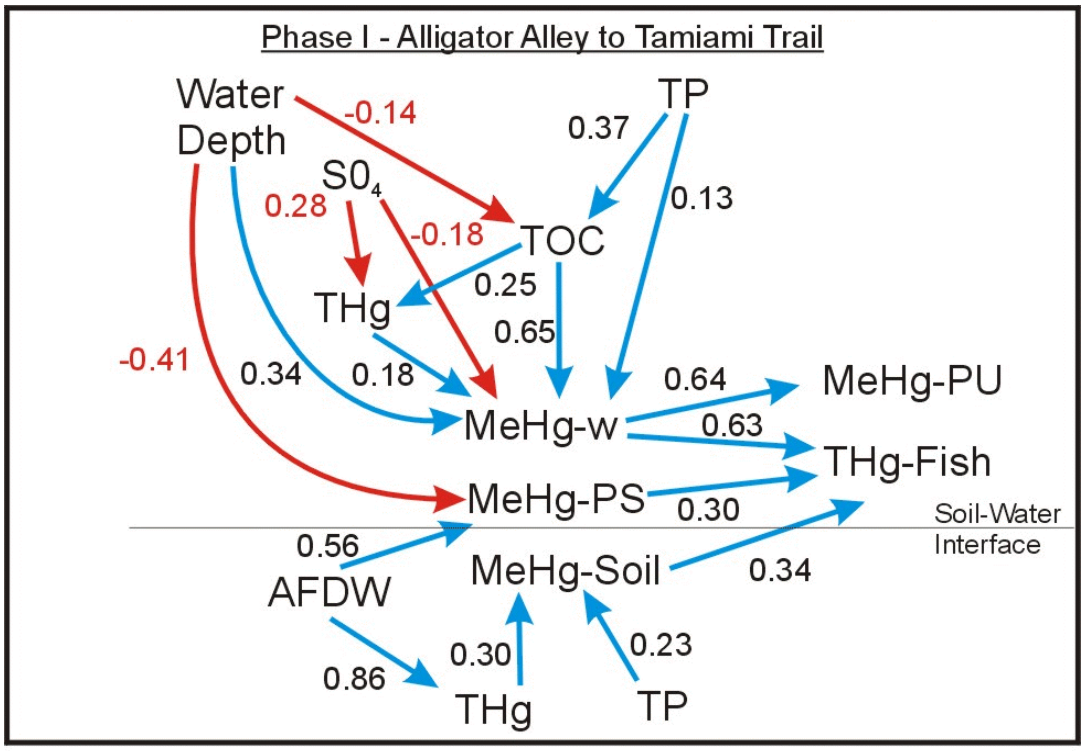


Figure 7.2. Phase I, II path analyses for the area north of Alligator Alley.



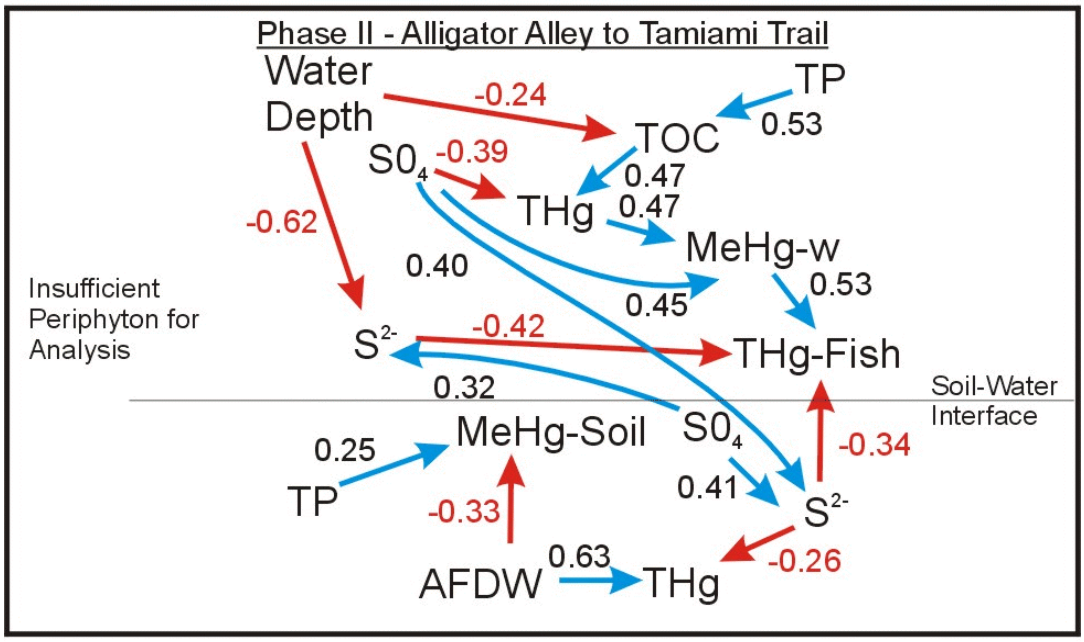


Figure 7.3. Phase I, II path analyses for the area between Alligator Alley and Tamiami Trail.

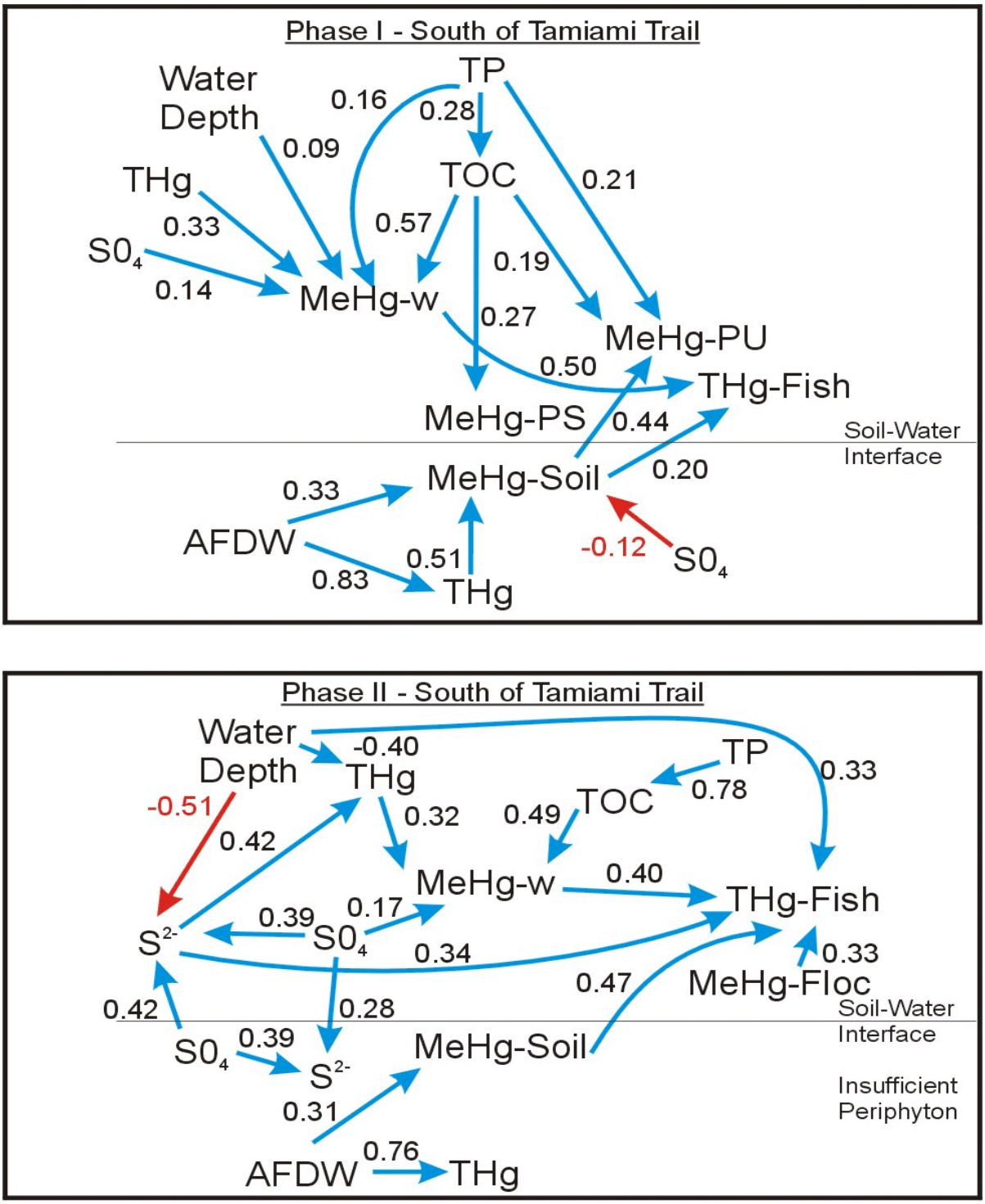


Figure 7.4. Phase I, II path analyses for the area south of Tamiami Trail.

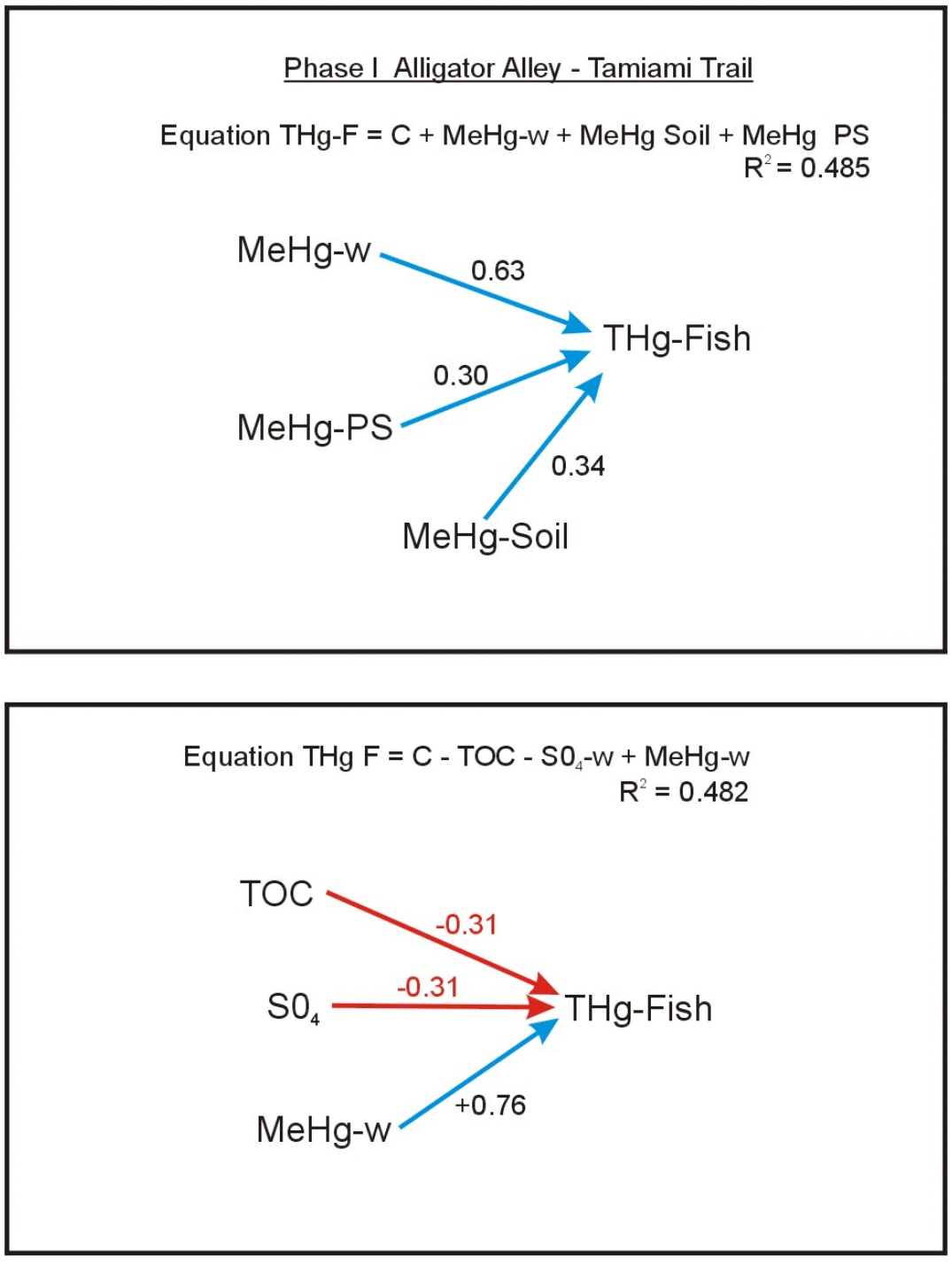


Figure 7.5. Alternative path analysis for pathway for fish total mercury in Phase I area between Alligator Alley and Tamiami Trail.

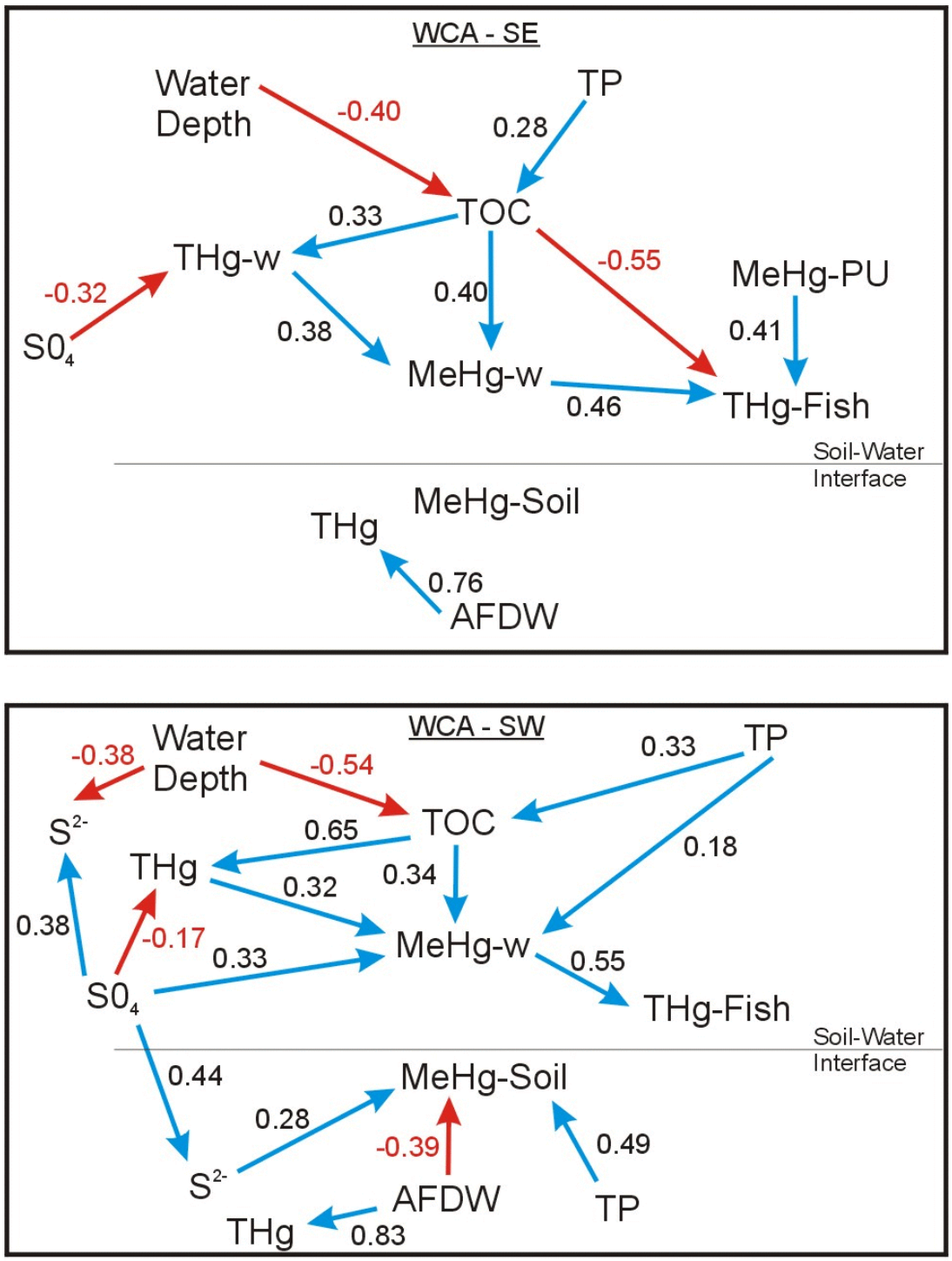


Figure 7.6. Phase I, II path analyses for WCA3-SE and WCA3-SW.

8.0 MERCURY ECOLOGICAL RISK ASSESSMENT

An early conclusion from the South Florida Ecosystem Assessment Project was that the greatest threat to the Everglades ecosystem was to assume that the problems facing the Everglades are independent, and that these problems can be managed independently (Stober et al 1996, 1998). The complex interactions among and effects of ecosystem level stressors in the Everglades is exemplified in an assessment of ecological risk from mercury. This chapter presents the ecological risk assessment for mercury in prey fish in the South Florida Everglades ecosystem. Specifically, it (1) presents an overview of the approaches and models used to assess the risks associated with mercury in the Everglades ecosystem; (2) summarizes the results of the Everglades mercury ecological risk assessment for prey fish; (3) demonstrates the ability of the mercury ecological risk assessment models to evaluate effects of management scenarios being proposed for the restoration of the Everglades ecosystem (e.g., restoration of hydrology, nutrient reduction via agricultural BMPs, and mercury emissions reductions) on mercury concentrations in Everglades biota; and (4) discusses how this risk assessment links to or serves as the foundation for the probabilistic mercury risk assessment for wading birds being conducted by the South Florida Water Management District. As additional large-scale data are collected, process studies completed, and monitoring of the Everglades ecosystem continues, the Everglades mercury ecological risk assessment will be refined so that the risks to prey fish and wading birds and benefits associated with proposed management alternatives can be evaluated more fully.

8.1 EPA Ecological Risk Assessment Framework

The EPA ecological risk assessment framework (EPA 1992) was used as the foundation for the large-scale South Florida Ecosystem Assessment project and early assessment of mercury in the South Florida Everglades ecosystem. This framework consists of three principal phases: problem formulation, analysis, and risk characterization. The ecological risk assessment framework was used because it provided a flexible, yet scientifically defensible approach for conducting this large-scale, multi-stressor ecosystem assessment. The iterative format of the framework was also consistent with the adaptive management approach being used to restore the South Florida Everglades Ecosystem.

As part of the problem formulation phase, a precursor conceptual model of mercury cycling in the Everglades was developed following a review of available data and information on mercury in the Everglades ecosystem. This conceptual model (Figure 7.1) shows the factors as well as the interactions and linkages between the factors that were thought to contribute to mercury in the Everglades ecosystem. Specifically, the precursor conceptual model suggested that the deposition of anthropogenic sources of mercury from local, regional, and global emissions, combined with specific nutrient inputs from the EAA, were creating conditions conducive to mercury methylation, accumulation, and biomamagnification through the food chain. Several testable mercury hypotheses were developed from this initial model. These hypotheses guided data collection by the US EPA during the Phase I assessment (i.e., from 1993 through 1996) (Stober et al. 1998).

In 1998, EPA published a final set of draft guidelines in the Federal Register for conducting ecological risk assessment (EPA 1998). These guidelines, which were built on the 1992 risk assessment framework, retained the major phases of the ecological risk assessment framework, but changed the terminology and steps within the phases. These changes were made to guide ecological assessments both on local and landscape scales. Such assessments often require the integration of physical, biological, and chemical stressors.

The ecological risk assessment guidelines provide decision-makers with an approach for considering available scientific information along with social, legal, political, or economic information or factors when selecting a course of action. The initial ecological risk assessment framework for the Everglades was modified to incorporate the terminology and the approaches of the 1998 ecological risk assessment guidelines (EPA 1998).

8.2 Problem Formulation

8.2.1 Spatial and Temporal Patterns of Mercury

The data collected during the Phase I assessment (Stober et al. 1998) and by others (e.g., Florida Game and Fish Commission and the University of Florida) documented the spatial extent of, and temporal changes in, mercury concentrations in water, soils, fish, and wading birds in the Everglades ecosystem. These studies showed that mercury “hot spots” in prey fish species (*Gambusia sp.*), periphyton, and wading birds occur within the Central portion of WCM3 (Stober et al. 1998) and that the spatial and temporal distribution of these hot spots is a function

of the complex interaction between hydroperiod, nutrient status, food web complexity, individual species life cycle requirements, and other factors. The Phase I study indicated there were no apparent discharge point sources of mercury (e.g., EAA) and atmospheric deposition was the primary source of mercury in the Everglades. The previous chapters discussed the pertinent information on the sources of mercury, exposure pathways, factors and processes affecting mercury exposure, and observed mercury concentrations in mosquitofish (*Gambusia sp.*).

Since the Phase I assessment, mercury concentration data also have been collected in largemouth bass (Lange et al. 2001), a top predator fish species. Lange et al. (2001) showed that similar to *Gambusia*, mercury concentrations in largemouth bass in the Everglades was highly variable spatially, with highest concentrations observed from the Central Everglades basin.

Rumbold et al. (1999) completed a probabilistic risk assessment of mercury in wading birds. This study concluded that mercury risks to wading birds in the Everglades also varied spatially and temporally. Comparing the exposure distributions for wading birds with feeding ranges limited to the Central Everglades basin to exposures integrated over the whole Everglades ecosystem, Rumbold et al. (1999) found that those birds foraging in the Central Everglades basin were at higher risk (i.e., 75% vs 35%) of exceeding the NOAEL than when exposure distributions over the whole Everglades were used. Specifically, wading bird colonies located in the mercury hot spots in the central portion of the Everglades between Alligator Alley and Tamiami Trail, were at higher risk than those colonies that feed elsewhere in the Everglades ecosystem. The data from Lange et al. (1999) and Rumbold et al. (1999) support work by Stober et al. (1998) indicating that there are spatial differences in mercury in ecological receptors in the Everglades. Moreover, these studies point to the importance of selecting sampling locations for estimating risks in the Everglades ecosystem. Rumbold et al. (1999) stated “recognition of scaling issues is critical in evaluating risk in environments with spatially highly variable concentrations, i.e., hot spots.”

These studies also point to the importance of temporal scales when interpreting data and the importance of evaluating ecosystem characteristics over longer temporal scales than a few years. For example, both Lange et al. (2001) and Frederick et al. (1999) indicate mercury concentrations in largemouth bass and Great Egret chick feathers, respectively, have been declining since about 1994. Mercury emissions have declined since 1989, which might have contributed to reduced mercury deposition over the Everglades ecosystem. However, from 1995

to the present, precipitation has also declined annually, which significantly affects mercury deposition. Pollman et al. (2001) stated they found no statistically significant trends in mercury deposition from 1994 to the present. Data collected over a longer period of time are needed to validate whether this decreasing trend is real and statistically valid.

8.2.2 Assessment Endpoints

Historically, largemouth bass and other top predator fish species, that routinely were consumed, represented the primary assessment endpoints for the mercury risk assessment for the Everglades. These endpoints were selected primarily because of human health concerns associated with fish consumption and widespread fishing consumption advisories throughout the Everglades since 1992. In 1989, when an endangered Florida panther was thought to have died from mercury toxicity, ecological receptors also became endpoints of concern.

Initial ecological assessment endpoints for the Everglades mercury ecological risk assessment included the Florida panther, the American alligator, and the Everglades wading bird populations. The public's desire to protect these species was a driving factor behind the selection of these species as the initial assessment endpoints for the mercury ecological risk assessment. Specifically, concerns over the survival of the endangered Florida panther (Roelke et al. 1991), declines in wading bird populations since the 1930s (Ogden 1994), and studies showing the potential effects of mercury accumulation in the food web on reproductive success of wading birds (Fredrick et al. 1999, Fredrick et al. 1997, Fredrick and Spalding 1994) were important drivers in the selection of these species as ecological assessment endpoints.

Both the Florida panther and the American alligator have been reevaluated as assessment endpoints for the mercury risk assessment because of a number of confounding factors. Other ecosystem stressors, such as PCBs, inbreeding, reduced population size, and habitat loss have lead to the elimination of the Florida panther as an assessment endpoint. Similarly, the American alligator has not been retained as an ecological assessment endpoint for the mercury risk assessment. Fish species such as mosquitofish and largemouth bass, and wading birds, including the Great egret, great blue heron, wood stork, and anhinga, predominately a fish eating species, are likely to become the final assessment endpoints for the mercury ecological risk assessment in the South Florida Everglades ecosystem. Wading birds in particular are emerging as the group at

highest risk from mercury in the Everglades further supporting this group of species as mercury ecological risk assessment endpoints.

8.2.3 Conceptual Model

An initial conceptual model of mercury cycling in the Everglades was used to guide the Phase I activities. This conceptual model described the sources of mercury to the South Florida Everglades ecosystem, how it entered the ecosystem, processes and factors affecting and controlling mercury methylation and bioavailability, and factors that result in direct exposure, indirect exposure, and biomagnification through the food chain. The Phase I results were reported previously in Stober et al. (1998) along with data gaps and needs for refining the conceptual model. Specifically, process studies to elucidate methylation/demethylation in the Everglades and effects of other stressors on these processes were identified as important in understanding transport and availability of mercury in the Everglades. Critical path analyses for top terrestrial predators also was identified as being needed (Stober et al. 1998). Based on the Phase I study (Stober et al. 1998) and results of mercury process studies and food web studies (Cleckner et al. 1998, 1999; Gilmour et al. 1998, 2000; Hurley et al. 1998; Krabbenhoft et al. 2000; Loftus et al 1998), the Everglades mercury conceptual model was refined.

8.2.4 Design and Planning

From the onset, the South Florida Ecosystem Assessment project has been designed to utilize an ecological risk assessment approach to evaluate the effects of and interactions between the multiple stressors present in the Everglades ecosystem. Both large scale collection of data and local process or site specific data and multiple lines of evidence developed through data analysis are used to support, refute, and revise the risk hypotheses for mercury.

Since the early 1990s, many studies have been conducted by cooperating agencies to collect the scientific data needed to complete the ecological risk assessment. During Phase II, the focus on the data collection in the South Florida ecosystem by EPA was to more fully evaluate the interactions and linkages between the principal variables within the mercury conceptual models both spatially and temporally in 1999 during two seasons: cycle 4 (the dry season) and cycle 5 (the wet season). As described in previous chapters, data collection activities in 1999

were focused within the marsh using the probability sampling approach used previously for the Phase I studies.

8.3 Analysis

The analysis phase of an ecological risk assessment includes two principal activities: exposure characterization, which is the contact or co-occurrence of a stressor with a receptor, and ecological effects characterization, or the measure of an effect. Exposure characterization describes sources of stressors, their distribution in the environment, and their contact or co-occurrence with ecological receptors. Ecological effects characterization evaluates stressor-response relationships or evidence that exposure to stressors causes an observed response.

8.3.1 Measures of Exposure

Mercury concentrations in mosquitofish and in largemouth bass are two measures of wading bird exposure to mercury in the Everglades. Data collected throughout the Everglades ecosystem during Phase I (1994 through 1996) (Stober et al. 1998) showed that MeHg concentrations in mosquitofish were lower in the area north of Alligator Alley and were higher in the central and southern areas. Similarly, Lange et al. (1999, 2001) showed that concentrations in largemouth bass also coincided with the mosquitofish hot spots (Figure 6.53). These areas of high MeHg concentrations in fish coincide with some of the largest breeding colonies for wading birds, e.g., the great egret and blue heron (Figure 6.53). Furthermore, the mercury hot spots for mosquitofish also coincided with wading bird rookeries where the highest concentrations of mercury were found in great egret chick feathers (Frederick et al. 1997).

Additional Phase II data, described in previous chapters, also showed that hydroperiod is an important factor influencing mercury concentrations in fish. Hydroperiod is an equally important factor influencing feeding behavior and therefore exposure in wading birds. This is particularly important on a seasonal basis and during some years when water depths in the South Florida ecosystem decrease during drier seasons or years of low precipitation. As indicated by Rumbold et al. (1999), “consideration of wading bird feeding habits and activity patterns is, therefore, essential in defining exposures integrated over different spatial scales.” Sampling of mosquitofish and largemouth bass to develop exposure distributions for wading bird populations

in the Everglades, therefore, must consider spatial and temporal variability in order to estimate risks to wading birds.

8.3.2 Measures of Ecosystem and Receptor Characteristics

Ecosystem and receptor characteristics were described in detail in Chapters 4 through 6. Consistent spatial distribution of MeHg in water, periphyton, mosquitofish, and wading birds indicates a consistency in the location of enhanced bioaccumulation and biomagnification in the food chain between Alligator Alley and Tamiami Trail, and south of Tamiami Trail through Shark River Slough.

8.3.3 Measures of Effects

Effects of MeHg in wildlife are summarized in a number of publications (Barr 1986; Bouton et al. 1999; Fredrick et al. 1999, 1997; Heinz 1979 Rumbold et al.1999; Nocera and Taylor 1998; and Wolfe et al. 1998). Effects of MeHg range widely from sublethal effects to the nervous system to effects on excretory, reproductive or immune system functions (Rumbold et al. 1999). Effects of MeHg at fairly low doses or as concentrations of mercury in the blood increase have been fairly well described for birds at the individual species level. These documented effects of MeHg in wildlife species however, generally come from laboratory studies, controlled mesocosm studies, and on individuals of a specific species. Although some field studies have been conducted in the Everglades ecosystem (Fredrick et al. 1997, Sepulveda et al. 1995), population and community level effects of MeHg, particularly on survival of fledglings and reproductive success, have not been documented.

MeHg effects in fish also are wide ranging. Changes in fish behavior, such as reduced feeding efficiency, occur when mercury concentrations exceed 6 ng/L MeHg. Other documented effects include transovarian mercury transfer (Weiner et al. 1996) and decreased condition index. Similar to bird data, this information is obtained from laboratory or mesocosm studies on single individuals, not in situ at the population level.

8.3.4 Exposure Analysis

The reduced structural equations shown in Table 8.1 can be used to estimate mercury concentrations in Gambusia for differing estimates of mercury deposition that might result from

emission controls. These reduced structural equations can also be used to assess the effects of nutrient reduction (e.g., TP) or hydrologic modifications to Gambusia mercury concentrations.

8.3.5 Ecological Response Analysis

Previous documentation indicates that wading bird colonies have been declining in the Florida Everglades since 1930s (Ogden 1994). Fredrick et al. (1999) showed that fledgling wading birds reared in colonies in these hot spots had elevated levels of liver mercury concentrations. Nevertheless, effects on wading bird populations and communities in the Everglades are not well documented.

The probabilistic mercury risk assessment for wading birds (Rumbold et al. 1999) provided the foundation for estimating wading bird exposure to mercury in the Everglades. Based on exposure distributions developed from data collected by Lange et al. (1999), wading birds with feeding ranges limited to the Central Everglades basin were at greater risk (i.e., 75% vs 35%) of exceeding the NOAEL than when exposure distributions over the whole Everglades were used. Specifically, wading birds colonies located in the mercury hot spots in the central portion of the Everglades between Alligator Alley and Tamiami Trail, were at higher risk than those colonies that feed elsewhere in the Everglades ecosystem. The probabilistic risk assessment and the Phase I and II studies point to the importance of sampling location selection for estimating risks in the Everglades ecosystem.

8.3.6 Exposure Profile

The reduced equations were used to project changes that might occur in methylmercury and total mercury in fish concentrations from changes in total phosphorus, sulfate and/or total mercury concentrations through management actions. Table 8.2 includes a comparison of the observed versus predicted constituent concentrations, projected changes in constituent concentrations following a reduction in total phosphorus to 10 Fg/L (5 Fg/L south of Tamiami Trail), reduction in sulfate to 0.5 mg/L (both in the area between Alligator Alley and Tamiami Trail and south of Tamiami Trail), reduction in total mercury to 1 ng/L, and finally with a simultaneous reduction in total phosphorus, sulfate and total mercury. Table 8.2 also includes a comparison of the observed median constituent concentrations with the reduced constituent concentration for the input variables for reference. Reduced equations were developed for both

Phase I and Phase II. However, only the Phase I reduced equations were used for the projections because the observed constituent concentrations were better represented using Phase I rather than Phase II equations.

North of Alligator Alley, the reduced equations overpredicted both water methylmercury and fish mercury concentrations compared with the observed values (Table 8.2). In part, this is probably because there was no significant relationship between sulfate/sulfide and the mercury species. Concentrations of both sulfate and sulfide are elevated north of Alligator Alley without a significant gradient across this area. Regression equations are based on gradients occurring in constituent values or concentrations. Reducing total phosphorus concentrations in this area resulted in a slight increase or no change projected in fish mercury concentrations (Table 8.2). Decreasing total mercury concentrations, however, did result in a projected decrease in both methylmercury and fish mercury concentrations.

Between Alligator Alley and Tamiami Trail, the reduced equations underpredicted fish mercury concentrations in Phase I and overpredicted fish mercury concentrations in Phase II (Table 8.2). Water methylmercury concentrations were slightly overpredicted, but, in general, observed versus predicted concentrations agreed within 0.1 ng/L. Changes in sulfate or total phosphorus concentrations resulted in similar projected changes in water methylmercury and fish mercury concentrations (Table 8.2). A greater change in both methylmercury and fish mercury concentrations were projected from the reduction in water total mercury concentrations. The greatest decrease in both methylmercury and fish mercury concentrations were projected by changing water total phosphorus and total mercury concentrations simultaneously (Table 8.2). Reducing water total phosphorus, sulfate, and total mercury concentrations resulted in a smaller projected reduction in water methylmercury and fish mercury concentrations than the change in only total phosphorus and total mercury (Table 8.2). This is because there is an inverse relationship in the reduced structural equations between sulfate and water methylmercury concentrations between Alligator Alley and Tamiami Trail (Table 8.1). Sulfate is a surrogate for sulfide and reducing the sulfate concentration in this area of the marsh also results in lower sulfide concentrations, which are acting as a ligand on both inorganic and organic mercury in this area. Binding the inorganic mercury with higher sulfide concentrations in this area make less inorganic mercury available for diffusion across methylating bacterial cell membranes. In addition, binding the organic or methylmercury in this area makes it less biologically available

for uptake and biomagnification through the food web. If both sulfate and total phosphorus are reduced through the STA's, the reduction in fish methylmercury might be less that if just total phosphorus were reduced.

South of Tamiami Trail, the marsh is probably approaching historic background conditions. The relationships in the reduced structural equations are all positive (Table 8.1). Observed concentrations of water methylmercury were within 0.01 ng/L of predicted concentrations (Table 8.2). The equations overpredicted fish mercury concentrations, but were within 10 ug/Kg of observed fish mercury concentrations (Table 8.2). Reducing water total phosphorus, sulfate, or total mercury concentrations resulted in a projected decrease in water and fish methylmercury concentrations. The greatest decrease in water methyl and fish mercury concentrations resulted from a reduction in all three input constituents - total phosphorus, sulfate, and total mercury concentrations.

The reduced form structural equations provide a tool for projecting changes in methylmercury and fish mercury concentrations that might occur from potential management actions that reduce water total phosphorus, sulfate or total mercury concentrations. These are steady-state equations and do not provide estimates of the time to reach these concentrations. However, the equations do provide an additional tool for screening management actions and formulating hypotheses that can be tested through field research studies.

8.3.7 Stressor-Response Profiles

Results from the Exposure Profile will be integrated with the stressor-response profiles developed by Rumbold et al. (2000). These interactions have been initiated, but not yet completed. Crystal Ball simulations will be used to integrate the variance about the median concentrations projected for Gambusia to provide a range of exposure to the wading birds and subsequent response of the wading birds to decreased mercury concentrations in their diet.

8.4 Risk Characterization

Risk characterization is the final phase of an ecological risk assessment. During this phase, risk assessors estimate ecological risks, indicate the overall degree of confidence in the risk estimates, cite evidence supporting the risk estimates, and interpret the adversity of ecological effects (EPA 1998). Estimating risks from mercury contamination in the Everglades

must consider the potential effects of nutrient addition and hydropattern modification on methylation and biomagnification. For example, food web complexity in the Everglades has been affected by increased nutrient inputs (Loftus et al. 1998). However, as shown through the conceptual models and path analysis, the increased potential for mercury transfer through more complex food webs is not only correlated with the nutrient regime, but also dependent on a number of other ecosystem characteristics. Loftus et al. (1998) and Fink et al. (1997) also suggest that changing water depths may affect food web complexity, mercury concentrations in prey and predator fish, and feeding rates in wading birds.

Because risks are not independent but rather joint probabilities, multiple lines of evidence must be evaluated to characterize ecological risks from mercury.

It is clear from assessing the results from the reduced structural equations that mercury is influenced not only by mercury deposition, but also by nutrient loading, sulfate loading and hydroperiod modifications. Assessing the risk from mercury contamination, therefore, must consider the interactions with these other factors. The greatest risk for mercury contamination occurs not at the sites with the greatest nutrient and sulfate loading, but at those sites that have moderate increases in nutrient and sulfate concentrations and that are pulsed by changes in hydropattern or water depth. Additions to these sites appear to stimulate the methylation process by continually providing a supply of sulfate and organic carbon (both through loading and through oxidation during dry periods) for methylating bacteria and that have relatively complete food webs. Although there are interactions of inorganic and organic mercury with ligands (e.g., sulfide, organic carbon), these interactions are not as strong as they are in the higher nutrient and sulfate areas to the north. Therefore, even though there is some binding by ligands, higher net methylmercury production results in more methylmercury being biologically available for uptake through the food web. The greatest reduction in mosquitofish mercury concentrations occurred in the oligotrophic portion of the marsh south of Tamiami Trail. This area is considered to be approaching the historical constituent concentrations that previously existed in the Everglades. Based on existing information, it can not be determined what historical mercury concentrations were in the Everglades. However, because wading birds were historically distributed throughout a greater area of the Everglades ecosystem, their risk from mercury might have been lower because they might not have been concentrated in the areas with the highest mercury concentrations.

Table 8.1. Reduced form structural equations used to project changes in *Gambusia* mercury concentrations based on selected management actions.

Phase I Equations
North of Alligator Alley $TOC = 10^{\wedge}(1.26 + 0.13 \text{ Log}_{10} (TP))$ $MeHg\text{-}w = 10^{\wedge}(-1.24 + 0.64 \text{ Log}_{10} (TOC) + 0.40 \text{ Log}_{10} (THg))$ $THg\text{-}fish = 10^{\wedge}(3.33 - 0.63 \text{ Log}_{10} (TOC) + 0.46 \text{ Log}_{10} (MeHg\text{-}w))$
Alligator Alley to Tamiami Trail $TOC = 10^{\wedge}(1.07 - 0.06 \text{ Log}_{10} (Depth) + 0.18 \text{ Log}_{10} (TP))$ $THg\text{-}w = 10^{\wedge}(-0.13 \text{ Log}_{10} (SO_4) + 0.38 \text{ Log}_{10} (TOC))$ $MeHg\text{-}w = 10^{\wedge}(-2.56 + 0.16 \text{ Log}_{10} (TP) + 0.38 \text{ Log}_{10} (Depth) + 1.62 \text{ Log}_{10} (TOC) - 0.15 \text{ Log}_{10} (SO_4) + 0.29 \text{ Log}_{10} (THgw))$ $MeHg\text{-}soil = 10^{\wedge}(-2.86 + 0.54 \text{ Log}_{10} (TP\text{-}soil) + 0.41 \text{ Log}_{10} (THg\text{-}soil))$ $MeHg\text{-}PS = 10^{\wedge}(-3.97 - 0.66 \text{ Log}_{10} (Depth) + 0.01 (AFDW))$ $THg\text{-}fish = 10^{\wedge}(3.03 + 0.57 \text{ Log}_{10} (MeHg) + 0.28 \text{ Log}_{10} (MeHg\text{-}soil) + 0.15 \text{ Log}_{10} (MeHg\text{-}PS))$
South of Tamiami Trail $TOC = 10^{\wedge}(1.06 + 0.16 \text{ Log}_{10} (TP))$ $MeHg\text{-}w = 10^{\wedge}(-2.45 + 0.18 \text{ Log}_{10} (TP) + 0.12 \text{ Log}_{10} (Depth) + 1.18 \text{ Log}_{10} (TOC) + 0.17 \text{ Log}_{10} (SO_4) + 0.67 \text{ Log}_{10} (THgw))$ $THg\text{-}soil = 10^{\wedge}(1.44 + 0.01 (AFDW))$ $MeHg\text{-}w\text{-}soil = 10^{\wedge}(-1.64 + 0.007 (AFDW) + 0.84 \text{ Log}_{10} (THg\text{-}soil) - 0.10 \text{ Log}_{10} (SO_4\text{-}soil))$ $THg\text{-}fish = 10^{\wedge}(2.55 + 0.38 \text{ Log}_{10} (MeHg\text{-}w) + 0.12 \text{ Log}_{10} (MeHg\text{-}soil))$

Table 8.2. Comparison of observed versus predicted constituent concentrations using the reduced form structural equations. Projected constituent concentrations potentially resulting from nutrient BMPs and mercury emission controls changing water TP and/or THg concentrations.

Constituent	Phase I							Phase II						
	Observed Median	Predicted	Projected				All	Observed Median	Predicted	Projected				All
			TP (10)	THg (1)	SO_4	TP & THg				TP (10)	THg (1)	SO_4	TP & THg	
North of Alligator Alley														
TOC	27.2	26.1	24.6	26.1	–	24.6	–	27.9	24.6	24.6	24.6	–	24.6	–
MeHg	0.51	0.67	0.63	0.46	–	0.45	–	0.41	0.61	0.61	0.45	–	0.45	–
THg - fish	106	226	231	192	–	196	–	109	226	226	196	–	196	–
Alligator Alley to Tamiami Trail														
TOC	20.9	18.9	18.2	18.9	18.9	18.2	18.2	18.5	17.5	18.5	17.5	17.5	18.5	18.5
THg-w	1.9	2.8	2.8	1	1	1	1	1.6	2.8	2.9	1	1	1	1
MeHg-w	0.46	0.50	0.46	0.37	0.46	0.34	0.42	0.32	0.39	0.45	0.29	0.33	0.33	0.38
MeHg-soil	0.43	0.27	0.27	0.27	0.27	0.27	0.27	0.42	0.20	0.20	0.20	0.2	0.20	0.20
THg - fish	217	177	168	150	168	142	160	117	147	159	124	135	134	146
South of Tamiami Trail														
TOC	16.5	16.5	14.8	16.5	16.5	14.8	14.8	11.0	15.0	14.8	15.0	15.0	14.8	14.8
MeHg-w	0.22	0.21	0.17	0.14	0.17	0.11	.09	0.11	0.12	0.12	0.09	0.11	0.09	.08
THg - soil	75	56	56	56	56	56	56	64	64	64	64	64	64	64
MeHg - soil	0.26	0.70	0.70	0.70	0.70	0.70	0.70	0.19	0.87	0.87	0.87	0.87	0.87	0.87
THg - fish	178	189	173	162	173	147	134	146	156	154	141	152	140	136

9.0 POLICY AND MANAGEMENT IMPLICATIONS

Seven management and policy-relevant questions guided this project. One of the primary objectives of this project was to provide scientifically sound information to answer these questions and contribute to management decisions on the South Florida Everglades ecosystem. This is an interim assessment, so not all of these questions can be fully answered, but at least partial answers can be provided for each question.

9.1 Hydroperiod Management

Findings

- The surface water coverage of the six synoptic surveys ranged from 44 to 100% of the ecosystem area, considering both dry and wet seasons.
- A surface area to volume curve was calculated, which indicated the long hydroperiod marsh covered about 4,200 km^2.
- The remaining short hydroperiod marsh from 4,200 km^2 to 5,500 km^2 (1,300 km^2) requires twice the water volume to inundate this area compared to the volume of water covering the long hydroperiod marsh.
- The shortest hydroperiod marsh is located in northwestern WCA3-N and Taylor Slough.
- The area of ponding estimated during the 1999 dry season indicated that if ponding of water north of the Tamiami Trail roadway were eliminated, the wet prairie/slough habitat in the marsh would be reduced by about 400 km^2.

Management Implications

- Water management changes to restore sheet flow in this system is a noble goal, but based on the surface area to volume curve, significant volumes of water will be required to achieve 100% surface water coverage of the ecosystem in the dry season.
- Annual drought cycles are a natural occurrence and some will be more severe than others. Large volumes of water continuously supplied will be required to make ecologically significant differences in surface water coverage when system storage capacity is low.
- Ponding in the system increases the wet prairie/slough refugia where aquatic organisms remain during droughts. Careful consideration should be given before any actions to reduce these areas are carried out.

- There may be insufficient volume to reestablish sheet flow in chronically drought prone short hydroperiod areas of the system. This does not mean that additional flow in central and eastern WCA3-N would not begin reversing the soil loss which has occurred there over the last 50 years. However, the build up of peat soil will occur most rapidly if continuous surface water coverage is maintained.

- The water and soil quality gradients identified in this study must be considered before plans are implemented to divert contaminated water farther downstream in this system with the result of making water quality deteriorate over a larger area of the ecosystem.

- There are macrophyte and periphyton community indicators of hydropattern modifications developed in this study that can be used to assess the effectiveness of future restoration efforts prior to and following implementation.

9.2 Nutrient Loading

Findings

- The median concentrations of total phosphorus in water decreased from 1995-96 to 1999, however, the change was not statistically significant across the ecosystem. The greatest change among the subareas was found in WCA2 and WCA3-N.

- Maximum water total phosphorus concentrations occurred in WCA3-N where the median TP concentrations declined from 16 to 11.4 ppb over the intervening three year period.

- Nutrient loading appeared to increase across the northwestern portions of WCA3-N and WCA3-SW in 1999, even though it decreased in other subareas.

- The increased water TP concentrations in WCA3-SE and WCA3-SW during the 1999 dry season probably resulted from phosphorus transport from WCA3-N because a wildfire that occurred in WCA-3N two weeks prior to sampling transformed plants and peat into phosphorus-rich ash.

- The extent of marsh area with TP in water <10 ppb has continued to increase over time, from 41% in 1995 to 78% in 1996 and 87% in 1999.

- The extent of marsh area with TP in water <15 ppb has likewise continued to improve from 65% in 1995 to 87% and 93% in 1996, and 1999, respectively.

- The extent of marsh area with TP in water >50 ppb remained at 2%.

- Median TP concentrations in soil decreased from 350 mg/kg in 1995-96 to 250 mg/kg in 1999.

- Median wet season soil TP concentrations were lower in Loxahatchee, WCA3-N, WCA3-SE, WCA3-SW and Shark Slough in 1999 versus 1995-96 while no change was evident in WCA2.

- The lowest median wet season soil TP concentrations consistently occurred in Taylor Slough

- Median wet season soil TP concentrations in WCA2 and WCA3-N were 350 and 400 mg/kg, respectively and are the subareas where the invasion of cattails is most prevalent.

- TP concentrations greater than 400 mg/kg occur along the EAA border of WCA2 and WCA3-N.

- Future changes in TP concentrations in water and soil require further monitoring to verify trends.

Management Implications

- The phosphorus control program, principally the Best Management Practices which have been in place since 1995, may be reducing the loading to the ecosystem.

- The decline in soil phosphorus concentrations in the less saturated downstream subareas is the area where an initial response to decreased loading is expected. The upstream heavily impacted subareas would be the last subareas expected to respond to decreased phosphorus loading.

- The invasion of the cattail community correlates with the high soil phosphorus in WCA2 and WCA3-N.

- Monitoring using the same methodology needs to continue in order to establish trends used to evaluate the effectiveness of the phosphorus control program.

9.3 Habitat Management

Findings

Remote Vegetation Assessment

- Remote sensing and GIS techniques were successfully used to assess vegetation patterns over the entire Everglades ecosystem.

- Areal summary statistics indicated spatial trends such as decreasing cattail coverage ranging from 12-17% in the north to 0.4% in the south.

- Plant communities identified in 1 km^2 plots, overlaid on the randomly selected sampling sites, adequately represented the vegetation cover in the Everglades. Comparison of remotely sensed estimates with existing database for ENP-Shark Slough and WCA3-N found the average difference in vegetation type percent cover estimates was 1.5% in ENP-SRS and 0.4% in WCA3-N. This demonstrated the data compatibility among USNPS and SFWMD vegetation mapping efforts.

- This effort establishes a baseline of conditions existing in 1994/1995 and a quantitative methodology for efficiently monitoring future vegetation patterns and assessing changes in the Everglades ecosystem over space and time.

Macrophyte Distributions and Morphology

- Because this study provides a quantitative evaluation of marsh macrophyte community types and their distributions across the Everglades ecosystem, it provides a background for evaluating community change during and after restoration.

- There are four major communities that are found across the entire ecosystem: sawgrass, waterlily-purple bladderwort, spikerush, and cattail. These communities differ in their hydroperiod/water depth, soil type, and nutrient levels. The dominant species within each community have different tolerances for soil TP.

- Sawgrass is the only community that occurs across the entire ecosystem; the other communities are more localized in their distributions.

- Although sawgrass was present throughout the Everglades, sawgrass morphology and density was correlated with changes in soil type. Controls on variations in density and morphology, as well as patchiness, represent areas for future research.

- Some communities that have been noted to be prominent historically did not appear as distinct communities in our analysis. For example, the Rhynchospora tracyi (beakrush) community did not form a distinct community in our clustering. These differences could represent a historical change in community composition in the ecosystem and/or could be a result of the quantitative nature of our analysis.

- Sagittaria lancifolia is found across a broad range of soil TP and soil organic content in the Everglades. We have shown in a parallel study that S. lancifolia leaf morphology provides an indication of soil nutrient level and water depth. Plants with broader laminae and shorter petioles are found in sites with higher nutrients, while plants with longer petioles are found in deeper sites with lower nutrients.

- The distribution of the major macrophyte communities can be used to monitor the effects of restoration actions.

Periphyton Distributions

This study demonstrated that diatom community metrics are associated with specific environmental changes and can be a useful tool in environmental monitoring. Diatom community metrics should be integrated into Everglades assessment protocols for the following reasons:

- Diatoms are ubiquitous in the Everglades yet species have non-random distributions. Baseline distribution data is now available for use in detecting environmental change.

- Diatoms are sensitive to environmental variation. Assemblage and species responses to spatial variation in ion content, nutrient availability and hydroperiod have been identified. Temporal models can be built from these spatially explicit data to predict community change under different management scenarios with a measurable degree of accuracy.

- Diatoms respond quickly to environmental change. Unlike many other biotic indicators, changes in diatom assemblage composition can happen over very short time scales (days to weeks) and, therefore, can provide sensitive early warning signals of impending ecosystem change.

- The taxonomic reference base generated from this survey will increase efficiency of future diatom inventories. Many surveys exclude diatom analyses because of perceived technical difficulties in collection and assessment. Currently available taxonomic databases should substantially reduce allocation of time and resources to identification. There are fewer species of diatoms in the Everglades than vascular plants. Given currently available reference materials, lack of technical expertise in this field is no longer a viable argument against diatom assessments, especially given their potential in environmental monitoring.

Management Implications

- A baseline of vegetative conditions using remote sensing, ground transect macrophyte community sampling, macrophyte morphology and periphyton communities has been established for monitoring and assessing future changes of the Everglades marsh habitat.

- The mosaic of plant communities across the ecosystem integrates the natural and the anthropogenic impacts imposed on this ecosystem.

- Changes in plant community response are of critical importance in evaluating the effectiveness of restoration practices.

- Indicator macrophyte and periphyton species have been identified which respond to multiple key interacting variables that can be used in assessing change.

- Each habitat methodology applied in this study has developed a unique and cost effective data set needed to track future habitat responses across the entire ecosystem.

9.4 Mercury Contamination

9.4.1 How Big is the Problem (Magnitude)?

Findings

- Over 60% of the marsh mosquitofish exceeded the proposed predator protection criteria for mercury.

- Less than 20% of the canal mosquitofish exceeded the proposed predator protection criteria for mercury.

- About 98% of the sampling sites had total mercury concentrations less than the mercury water quality criteria of 12 ppt (parts per trillion).

- Methylmercury concentrations in the water rarely exceeded 1 ppt, yet mercury concentrations in mosquitofish and largemouth bass exceeded 500 ppb and 1 ppm, respectively. This is a biomagnification factor of 500,000 to 1,000,000 times the methylmercury concentration in the water.

Management Implications

- The methylmercury criteria based on mercury concentrations in fish tissue (300 ppb) is appropriate because it considers bioaccumulation and biomagnification through the food chain.

9.4.2 What is the Extent of the Problem (Extent)?

Findings

- There is a hot spot in Water Conservation Area 3A, just below Alligator Alley, where methylmercury concentrations are highest in water, algae, fish, and wading birds. This hot spot has an area of over 200 square miles.

- There is an area that extends from this hot spot below Alligator Alley down through Shark River Slough in Everglades National Park in which fish and wading birds also have elevated mercury concentrations.

Management Implications

- By both magnitude and extent, fish, alligators, wading birds, the Florida panther, and other organisms in the marsh have greater mercury contamination than organisms in the canals. Focus management actions on the marsh.

- The mercury hot spot corresponds with an area in which wading birds breed and feed.

9.4.3 Is it Getting Better or Worse over Time (Trends)?

Findings

- A solid baseline (1993–1996) has been established to evaluate future trends. The comparative comprehensive monitoring in 1999 has provided the opportunity to begin trend assessment which can be compared to other more frequent trend monitoring in top predators to determine the status of mercury contamination in the Everglades ecosystem through time.

- During the past 10 years there has been an estimated decrease of greater than 95% in local atmospheric emissions in South Florida. There also has been a corresponding reduction in total mercury concentrations in surface water and declines in prey fish, largemouth bass and great egret chick feathers.

- Total mercury concentrations in prey fish greater than 200 ppb declined from a 40% exceedance in 1995-96 to a 20% exceedance in 1999. This indicates an approximate reduction of 50% in mercury in fish with the highest concentrations.

- Largemouth bass monitoring by FFWCC indicates a 66% decline in total mercury in fillets still exceeds the Florida fish consumption advisory of 0.5 ppm.

- Monitoring of great egret chick feathers by University of Florida scientists from 1994-2000 has shown a 73% decline in mercury.

Management Implications

- Maintain the EPA Region 4 monitoring program with seasonal sampling, but emphasize the marsh sites compared to the canals. Establish trend sites.

- Continue monitoring the great egret check feathers, largemouth bass, and mosquitofish to assess trends.

- The mercury problem did not occur overnight and it will not be corrected overnight. Long-term management practices will be required to fix the mercury problem.

- Monitoring is the only approach for assessing the effectiveness of management and restoration practices to control eutrophication, restore natural hydropattern changes, and eliminate mercury contamination.

9.4.4 What is Causing the Problem (Causation)

Findings

- The exact causes of mercury contamination in the South Florida ecosystem are unknown. However, it is likely the interaction of total phosphorus, TOC, and sulfate loading from the EAA, water depth, organic matter sources and production, food chain links and continued input of atmospheric mercury to the ecosystem control mercury contamination.

- The large scale spatial patterns of these environmental conditions have been established through the EPA Region 4 program, FFWCC fish sampling, and NPS/FL DEP wading bird sampling programs.

- Processes responsible for these large-scale patterns are being studied through the USGS ACME program, EPA and FL DEP atmospheric deposition studies.

Management Implications

- There is no "magic bullet" that can be implemented to control one factor and eliminate mercury contamination.

- Factors controlling mercury should be determined in the hot spot and compared with factors in other areas without extensive mercury contamination to develop effective management strategies.

- Controlling EAA loading of phosphorus, sulfate, and TOC concentrations might also reduce the mercury problem by reducing constituents that are influencing mercury contamination.

9.4.5 What are the Sources of the Problem (Sources)?

Findings

- Annual atmospheric mercury loading is from 35 to 70 times greater than mercury loading from the Everglades Agricultural Area.

- An EPA ORD study indicated municipal and medical waste incineration emissions had higher mercury concentrations than emissions from a coal-fired cement kiln.

Management Implications

- Local emissions are a significant source of inorganic mercury.
- Mercury emissions controls would reduce mercury loadings to the Everglades ecosystem.
- However, waste disposal is a multimedia problem. Controlling mercury emissions might create other problems such as disposal of solid waste, including not only the waste, but also the mercury removed from the emissions.

9.4.6 What is the Risk to the Ecosystem (Risks)?

Findings

- Mercury methylation is also controlled or influenced by hydropattern, habitat alteration, and food web complexity.
- Over 60% of the marsh area has mosquitofish with mercury concentrations that exceed the proposed predator protection level.
- Mercury concentrations are high, near toxic levels in wading bird livers and other organs but have been declining in largemouth bass and wading birds over the past 8 years.
- There is a 200 square mile hot spot where mercury contamination in biota is greatest, which corresponds with an area of wading bird rookeries.

Management Implications

- Biological species higher in the aquatic food chain are at risk from mercury contamination, even though the effects are subtle. Because mercury bioaccumulates, the risks increase over time. The longer management is delayed, the greater the risks.
- However, the greatest threat to the Everglades ecosystem is to assume the environmental problems are independent.

9.4.7 What Can We Do About The Problem (Management)?

Findings

- The SFWMD Everglades Nutrient Removal project removes nutrients and total and methylmercury from the inflow to the Project.
- Atmospheric mercury loading is much greater than mercury loading from the EAA stormwater.

Management Implications

- Controlling nutrient loading, hydropattern and habitat type should contribute to reducing the mercury contamination problem.
- Controlling local atmospheric mercury emissions has apparently reduced the mercury load to the South Florida Everglades ecosystem and the concentration in biota. However, there has been no apparent change in mercury deposition over the past 8 years.
- Emission controls have multimedia impacts and must be assessed as a multimedia issue, not as a single media issue.
- If the nutrients, sulfate and TOC concentration gradients, were decreased further and pushed upstream, the zone of impact where fish mercury is high could be reduced and might be outside the areas where wading birds concentrate for breeding, feeding, and with reduced emissions, the overall fish concentrations might be lower.

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APPENDIX A: Aerial Photo Vegetation Assessment in the Everglades Ecosystem

Aerial Photo Vegetation Assessment in the Everglades Ecosystem

Final Report

by

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ABSTRACT

Long-term monitoring of the Everglades ecosystem, including observations of plant communities over broad areas as indicators of biogeochemical change, can be implemented using remote sensing and geographic information system (GIS) techniques. The Center for Remote Sensing and Mapping Science (CRMS) at The University of Georgia has used these techniques in cooperation with the Everglades National Park (ENP) and the U.S. Environmental Protection Agency (EPA) Science and Ecosystem Support Division (Athens, Georgia) to conduct a vegetation assessment study in support of the EPA South Florida Ecosystem Assessment Project. The EPA randomly generated coordinate locations for 250 environmental monitoring sites distributed throughout the South Florida Water Management District (SFWMD) Water Conservation Area (WCA) 1, WCA 2, WCA 3, the Rotenberger/Holey Land Everglades Agricultural Area (EAA) and ENP. Vegetation communities within 1 x 1 km (1 km^2) plots centered on the EPA monitoring sites were extracted from existing Everglades vegetation databases originally created by the CRMS, the National Park Service (NPS) and the SFWMD from 1994/1995 National Aerial Photography Program (NAPP) color infrared (CIR) aerial photographs. Vegetation in areas outside of the existing databases was interpreted from U.S. Geological Survey (USGS) Digital Orthophoto Quarter Quads (DOQQ) produced from the same 1994/1995 NAPP aerial photographs. The classification system followed the Everglades Vegetation Classification System and included vegetation identified to the plant community, association and species levels.

Data analysis included the development of areal statistics for the dominant, secondary and tertiary vegetation types within each of 250 monitoring sites (1 km^2), and for combinations of the dominant/secondary vegetation classes. These summary statistics were provided to the EPA for further analysis and correlation with environmental data collected at the monitoring sites.

The cumulative distribution of four major plant communities (i.e., cattail, sawgrass, wet prairie and other) provided status and trend information on the range of vegetation types within regions and latitudinal zones distributed north to south throughout the Everglades system. A map was created depicting the proportion of vegetation cover in each 1 km^2 monitoring site as represented by a pie chart. The map also includes histogram graphs of dominant and secondary vegetation types generalized into the four major vegetation classes and summarized by region and latitudinal zone. On this map, spatial trends such as the clustering of wet prairie dominated sites within WCAs 1 and 2 can be visually correlated with man-made structures such as canals and roadways that restrict hydrologic flow. The distribution of sites containing considerable proportions of cattail, grouped within WCA 2, WCA 3 and the northeastern section of ENP, also appear to coincide with canals and may warrant further investigation of spatial correlations of cattail growth with elevated nutrient levels.

Additional data analysis included a comparison of summary statistics for vegetation distributions within 1 km^2 monitoring sites to statistics derived from existing Everglades vegetation databases in order to establish that the selected samples were indeed representative of continuous vegetation cover. The spatial interpolation of vegetation distributions between EPA monitoring sites also was demonstrated. Output products included 250 page-size vegetation maps of monitoring sites, a 1:80,000-scale overview map depicting spatial trends in major vegetation classes, digital data sets and summary statistics. This study establishes a baseline of conditions existing in 1994/1995 and documents an efficient methodology for long-term monitoring of the Everglades system.

INTRODUCTION

The Center for Remote Sensing and Mapping Science (CRMS) at The University of Georgia has cooperated with the Science and Ecosystem Support Division of the U.S. Environmental Protection Agency (EPA) to assess vegetation patterns along a north – south corridor across the Florida Everglades. This work was undertaken for Everglades National Park (Cooperative Agreement Number 5280-4-9006) in support of the EPA South Florida Ecosystem Assessment Project.

Long-term monitoring of plant community distributions as indicators of biogeochemical changes over broad areas such as the Everglades ecosystem can be implemented using remote sensing and geographic information system (GIS) techniques. The CRMS is uniquely qualified to provide the EPA with vegetative cover information for the Everglades study area. In addition to numerous studies using remote sensing/GIS to map wetlands in the southeastern United States, the CRMS has worked cooperatively with the National Park Service (NPS) since 1994 to map Everglades vegetation communities in South Florida Parks and Preserves (Welch et al., 1988, 1991 and 1992; Remillard and Welch, 1992; Welch and Madden 1999). Over a four-year period from 1994 to 1998, the CRMS and NPS developed a detailed vegetation database in Arc/Info format and produced associated 1:15,000-scale paper map products for Everglades National Park, Big Cypress National Preserve and Biscayne National Park – wetland areas covering approximately 10,000 km^2 (Welch, et al., 1995; 1999; Welch and Remillard, 1996).

The EPA South Florida Ecosystem Assessment Project study area encompasses approximately 5,600 km^2, including South Florida Water Management District (SFWMD) Water Conservation Area 1 (WCA 1), WCA 2 and WCA 3, along with the Rotenberger/Holey Land Everglades Agricultural Area (EAA) and portions of Everglades National Park (ENP) (Figure 1). The WCAs are used by the SFWMD for water storage and management with water levels controlled by a system of canals and gates. The ENP, on the other hand, is characterized by a less restricted flow of water through broad sloughs impeded only by a few roads. The Rotenberger/Holey Land EAA consists mainly of abandoned agricultural land.

The study area also was subdivided into latitudinal zones by the EPA. Depicted in Figure 1, the boundaries between latitudinal zones correspond (from north to south) to

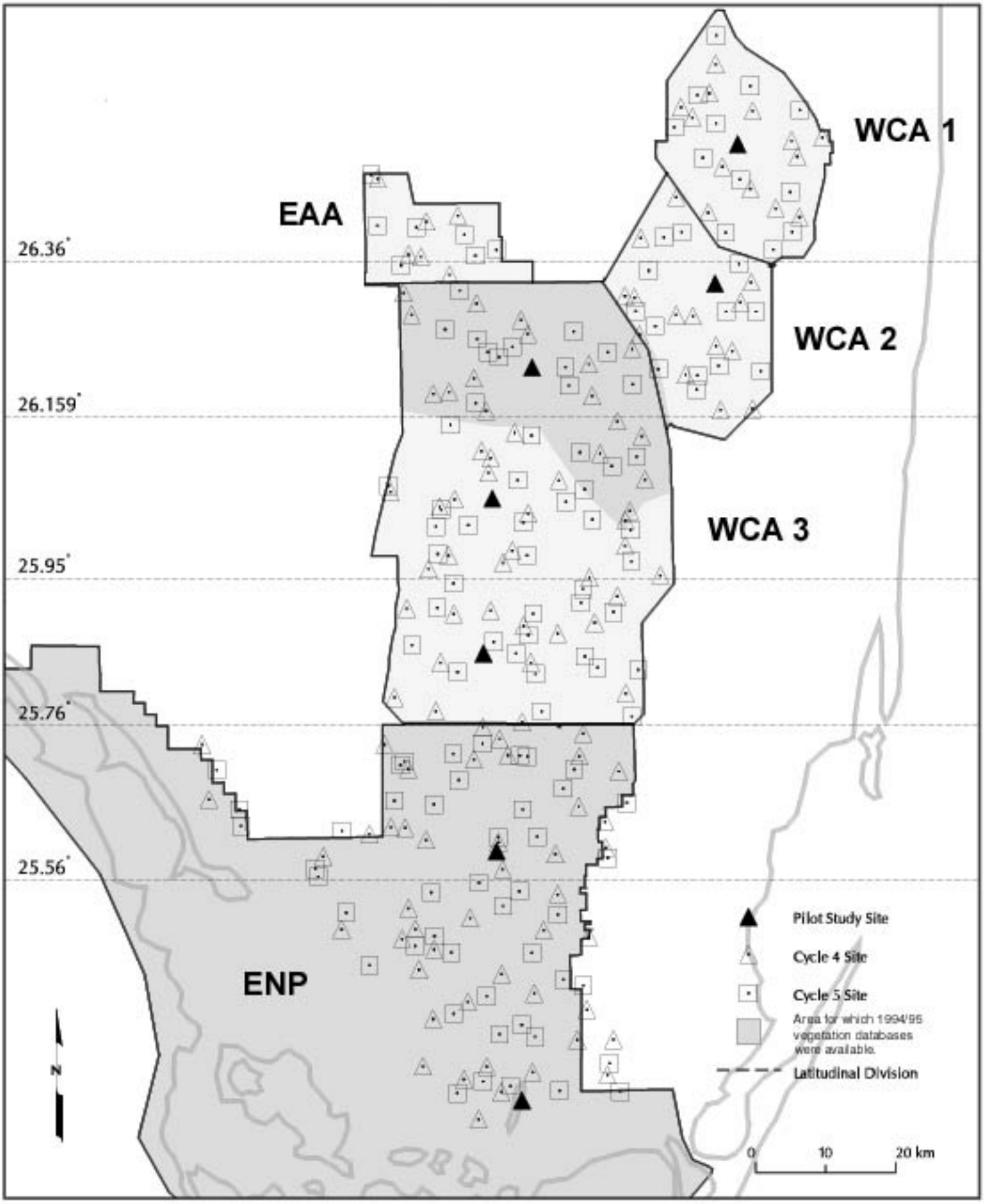

Figure 1. EPA South Florida Ecosystem Assessment Project study area and locations of pilot study, Cycle 4 and Cycle 5 monitoring sites.

26.68°, 26.36°, 26.16°, 25.95°, 25.76°, 25.56° and 25.24° north latitudes. Within these latitudinal zones, the EPA randomly located the following monitoring sites for EPA field data collection: 1) 132 stations for the Cycle 4 dry-season field survey conducted in April, 1999; and 2) 126 stations for the Cycle 5 wet-season field survey conducted in September, 1999. Eight of these monitoring sites fell outside of the EPA South Florida Ecosystem Assessment Project study area and were subsequently dropped from the analysis. The CRMS defined a 1 km^2 area around each of the remaining 250 monitoring sites for characterization of vegetation communities using remote sensing and GIS techniques.

The CRMS vegetation assessment was conducted in two parts, Phase I focused on vegetation characterization and mapping, while Phase II involved data analysis. Specific objectives for this study are outlined below.

Phase I Objectives

1) Compile remotely sensed data sets and existing GIS databases appropriate for the identification of vegetation communities within the South Florida Ecosystem Assessment Project study area.
2) Create detailed 1 km^2 vegetation maps in digital and hardcopy formats centered on each of 250 EPA monitoring sites, and provide maps/data sets to the EPA prior to the intended field survey dates.
3) Provide summary statistics of the area and percent cover of dominant, secondary and tertiary vegetation types occurring within the 1 km^2 vegetation maps. Also generate summary statistics to provide area/percent cover of dominant vegetation classes and four major vegetation classes (i.e., cattail, sawgrass, wet prairie and other).
4) Produce a map of the entire study area showing summary data for the four major vegetation classes at each monitoring station, as well as histograms characterizing vegetation cover by region and latitudinal zone.

Phase II Objectives

1) Supply summary statistics for the area and percent cover of dominant and secondary vegetation types occurring within the 1 km^2 monitoring sites.
2) Compare the proportions of vegetation types and areal coverage within a subset of the monitoring sites to the corresponding area covered by existing vegetation databases. Vegetation classes to be compared include sawgrass, wet prairie, muhly grass, cattail, mixed graminoid, non-graminoid emergent, bayhead, pine/hardwood water and other vegetation classes.
3) Interpolate the spatial distribution of major vegetation types between EPA monitoring sites to determine general trends of percent cover over the entire South Florida Ecosystem Assessment Project study area.
4) Provide a final report of the aerial photo vegetation assessment covering both development and analysis of the vegetation database (Phase I and II).

DATABASE DEVELOPMENT

Latitude and longitude values for all monitoring sites were provided to the CRMS by the EPA. These geographic coordinate values were used to create two Arc/Info coverages, one containing Cycle 4 sites, the other Cycle 5 sites (see Figure 1). Six sites from Cycle 4 and one site from Cycle 5 were selected by EPA for use in a pilot study designed to establish appropriate field techniques and statistical analysis methods before the project fieldwork began in April 1999. Eight sites provided to the CRMS fell outside both ENP and SFWMD boundaries and were disregarded, leaving 128 Cycle 4 sites and 122 Cycle 5 sites – a total of 250 monitoring sites.

Detailed vegetation databases previously compiled by the CRMS, NPS and SFWMD from 1:40,000- and 1:24,000-scale color infrared (CIR) aerial photographs recorded in 1994/1995 were the primary data sources employed in this project. In each of these databases, the vegetation was photointerpreted and vegetation boundaries rectified to the Universal Transverse Mercator (UTM) ground coordinate system tied to the North American Datum of 1983 (NAD 83) to within a root mean square error (RMSE) of approximately $\pm$ 5 to 10 m. The minimum mapping unit was one hectare. Details on the mapping procedures, ground truthing and database development can be found in Welch et al. (1999) and Rutchey and Vilchek (1999). These data sets provided consistent and detailed information on vegetation communities for 117 of the 250 EPA monitoring sites

Vegetation patterns for the remaining 133 monitoring sites were interpreted using USGS CIR Digital Orthophoto Quarter Quads (DOQQs) covering WCA 1, WCA 2, EAA and a portion of WCA 3. The DOQQs of Florida were derived from USGS NAPP aerial photographs (the same 1994/1995 NAPP photographs used in the CRMS/NPS mapping project). They are reported by the USGS to meet planimetric accuracy standards of about $\pm$ 3 m. Approximately 86 DOQQs were required to interpret the vegetation for those sites not included in the original CRMS/NPS/SFWMD databases.

For each site, a 1 km^2 plot centered on the monitoring site was created in Arc/Info coverage format. Vegetation data from the CRMS/NPS or SFWMD was clipped from the corresponding area in the vegetation databases. Where no vegetation data existed, the plot was digitally overlaid on the DOQQ and used as a template to interpret vegetation communities and create a new vegetation map centered on the monitoring site.

Vegetation classes delineated within the 1 km^2 plots followed the Everglades Vegetation Classification System developed by the CRMS, NPS and SFWMD (Madden et al., 1999; Welch et al., 1999). In this hierarchical system, 89 classes can be used to identify Everglades vegetation to the plant community, association and species levels. These classes also can be used in combination with numeric modifiers indicating factors affecting vegetation growth, (e.g., evidence of abandoned agriculture or altered drainage), information about the vegetation distribution (e.g., scattered individuals) and important environmental characteristics (e.g., abundant periphyton). Attachment A provides a description of the Everglades Vegetation Classification System.

In order to accommodate the complex vegetation patterns found in the Everglades, a three-tiered scheme was developed for attributing vegetation polygons (Welch et al., 1995; Obeysekera and Rutchey, 1997). Using this scheme, interpreters were able to annotate each polygon with a dominant vegetation class accounting for more than 50 percent of the vegetation in the polygon. Secondary and tertiary vegetation classes were added as required to describe mixed plant communities within the polygon. This three-tiered scheme, as well as the hierarchical organization of the classification system, permits classes to be collapsed and generalized as required to examine trends over space and time.

The digital data sets for 250 sites were used to create hardcopy maps and to generate summary statistics of total area and percent cover for vegetation classes. To enable the efficient production of hardcopy map products, an automated mapping interface was developed. The interface allows each 1 km^2 map to be plotted using a standardized map collar, which included the EPA monitoring station name, Cycle number, locator map, UTM grid, scale bar and legend. Detailed plant community information is included as text labels within each polygon. Tabular summary data of area and percent for each vegetation classification found in the 1 km^2 map, are automatically generated when the map is plotted and included in each map legend. The CRMS provided a total of 250 page-size (8.5 x 11 in.) paper maps to the EPA prior to the intended field survey dates that included all monitoring sites in both Cycles 4 and 5 (Table 1).

Table 1. Delivery Dates for EPA Monitoring Site Maps/Databases

Cycle	Field Survey	Date	Number
Pilot Study	Field/Mapping Procedure Test	January 1999	7
Cycle 4	Dry-Season Survey	April 1999	122
Cycle 5	Wet-Season Survey	September 1999	121
Total			250

Figure 2 shows a sample hardcopy map product for a single monitoring site as released to the EPA. The comprehensive vegetation legend providing the full Everglades Vegetation Classification name for abbreviations printed on the monitoring site maps is provided in Figure 3. Arc/Info coverages of vegetation data sets for the 250 monitoring sites were delivered to the EPA in Arc/Info Export format copied to CD-ROM.

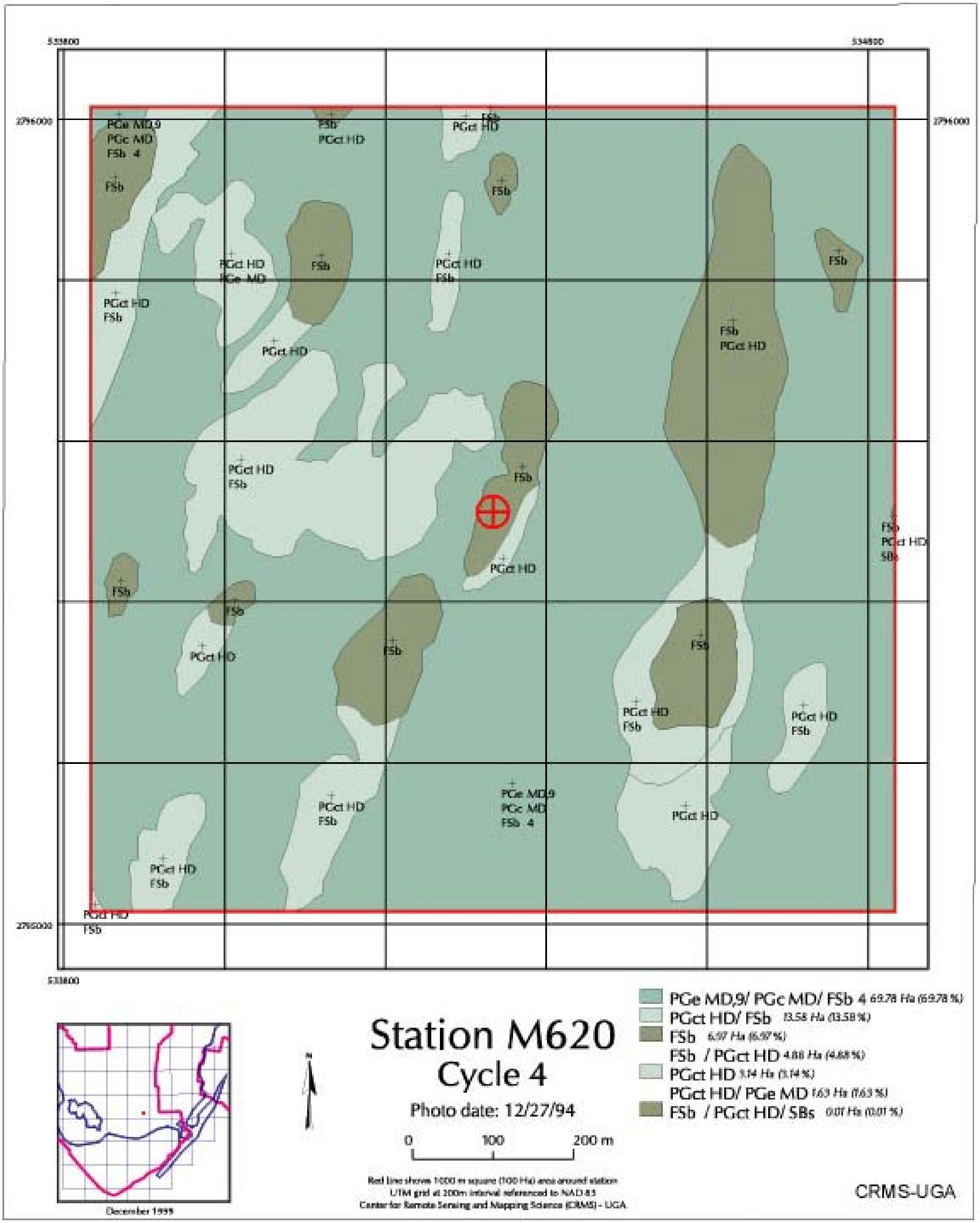

Figure 2. Sample vegetation map for a 1 km^2 plot surrounding a single EPA monitoring site.

Figure 3. Everglades Classification System legend.

VEGETATION DISTRIBUTION STATISTICS

Areal statistics were compiled for vegetation areas within the 1 km^2 maps corresponding to the 250 EPA monitoring sites. Statistics files contain the total area (in m^2), percent cover and frequency of occurrence for each unique combination of dominant, secondary and tertiary vegetation types in all monitoring sites. Table 2 illustrates a portion of the areal statistics (m^2) for two EPA monitoring sites sorted by unique combinations of dominant/secondary/tertiary vegetation classes. These statistics were collapsed in Tables 3 and 4 to illustrate further summarization of area data by unique combinations of dominant/secondary vegetation and by dominant vegetation classes, respectively. All statistical information was provided to the EPA in Microsoft Excel and text formats.

In order to assess trends in major vegetation patterns over the entire study area, the EPA identified four generalized categories as being of particular significance: cattail, sawgrass, wet prairie and "other" vegetation types. The summary statistics were therefore collapsed into these four major classes. A polygon was characterized as "cattail" or "sawgrass" when those species predominated. For example, the abbreviation for cattail in the database is "PC" (see Attachment A). Sawgrass is represented by the abbreviations "PGc", "PGct", "PGx", or "PGs". A polygon was characterized as "wet prairie" when it contained classes "PGe", "PGa", "PGw", "PE", "PEb", or "PEf". Polygons not included in one of the preceding classes were included in the "other" category.

An exception to this procedure occurred in the "wet prairie" class. Wet prairie was under-represented in the ENP vegetation database compared to the SFWMD database due to a slight difference in interpretative priorities. Since the ENP database did not separate low density sawgrass polygons in slough areas as wet prairie, a polygon was considered to be "wet prairie" if it was characterized primarily by sawgrass *and* contained open water, or if it was characterized primarily by sawgrass *and* secondarily by wet prairie species such as PGe and PGa. All maps and summary data which use the generalized, four-class system reflect this difference.

Tables 5 and 6 list the percent cover of major vegetation types (i.e., cattail, sawgrass, wet prairie and other) summarized for all 250 1 km^2 maps and organized by region and latitudinal zone, respectively. By region, cattail is most abundant in WCA 2, covering nearly 25 percent, while only 1 percent of ENP contains cattail (Figure 4). Sawgrass covers approximately 40 percent of most regions with the highest coverage (55 percent) in ENP. Wet prairie ranges between 15 and 29 percent cover in all regions except ENP where wet prairie covers only 11 percent. Other vegetation is most abundant in the EAA and ENP, covering 45 and 33 percent, respectively.

The distribution of vegetation summary statistics by latitudinal zones is shown in Table 6 and Figure 5. Ranging from north to south (left to right on the table and graph), cattail coverage decreases steadily from 12 and 17 percent in the northern most zones to 1.5 and 0.4 percent in the southern most zones. Sawgrass coverage is fairly constant among northern zones (40 to 35 percent) and peaks at 68 and 44 percent cover in the

Table 2. Sample Summary Statistics (Area in m^2) for Two EPA Monitoring Sites by Dominant/Secondary/Tertiary Vegetation Classes (Note: Sample includes a small portion of the vegetation classes)

Site	PGc	PGc PGct	PGc PGct PGw	PGc PGw	PGw	PGw PE	PGw PGc	PE PGc	SH FSb E	SH PGct
3A11	48346.5	216087.4	143097.1	163018.2			12279.6		26201.3	6062.7
3A15		8670.6		34879.3	8439.7	38892.5	1404103.0	6853.9		

Table 3. Sample Summary Statistics (Area in m^2) for Two EPA Monitoring Sites by Dominant/Secondary Vegetation Classes (Note: Sample includes a small portion of the vegetation classes)

Site	PGc/----	PGc/PGct	PGc/PGw	PGw/----	PGw/PE	PGw/PGc	PE/PGc	SH/FSb	SH/PGct
3A11	48346.5	359184.0	163018.2			12279.6		26201.3	6062.7
3A15		8670.6	34879.3	8439.7	38892.5	1404103.0	6853.9		

Table 4. Sample Summary Statistics (Area in m^2) for Two EPA Monitoring Sites by Dominant Vegetation Classes (Note: Sample includes a small portion of the vegetation classes)

Site	PGc	PGw	PE	SH
3A11	570548.7	12279.6		32264.0
3A15	43549.9	1451435.2	6853.9	

Table 5. Percent Cover of Major Vegetation Classes by Region – Cycles 4 and 5 Combined

Vegetation Class	Rotenberger/ Holey Land EAA Percent Cover	WCA 1 Percent Cover	WCA 2 Percent Cover	WCA 3 Percent Cover	ENP Percent Cover
Cattail	11.1	8.7	24.9	7.8	1.0
Sawgrass	24.7	41.7	43.3	37.0	55.1
Wet Prairie	19.4	28.8	15.6	28.0	10.9
Other	44.8	20.8	16.2	27.2	33.0

Table 6. Percent Cover of Major Vegetation Classes by Latitudinal Zone – Cycles 4 and 5 Combined

Vegetation Class	26.68° to 26.36°	26.36° to 26.16°	26.16° to 25.95°	25.95° to 25.76°	25.76° to 25.56°	25.56° to 25.24°
Cattail	11.5	16.8	7.9	5.6	1.5	0.4
Sawgrass	39.9	40.0	35.7	34.5	68.0	43.5
Wet Prairie	22.6	14.9	32.2	36.9	7.1	14.4
Other	26.0	28.3	24.2	23.0	23.4	41.7

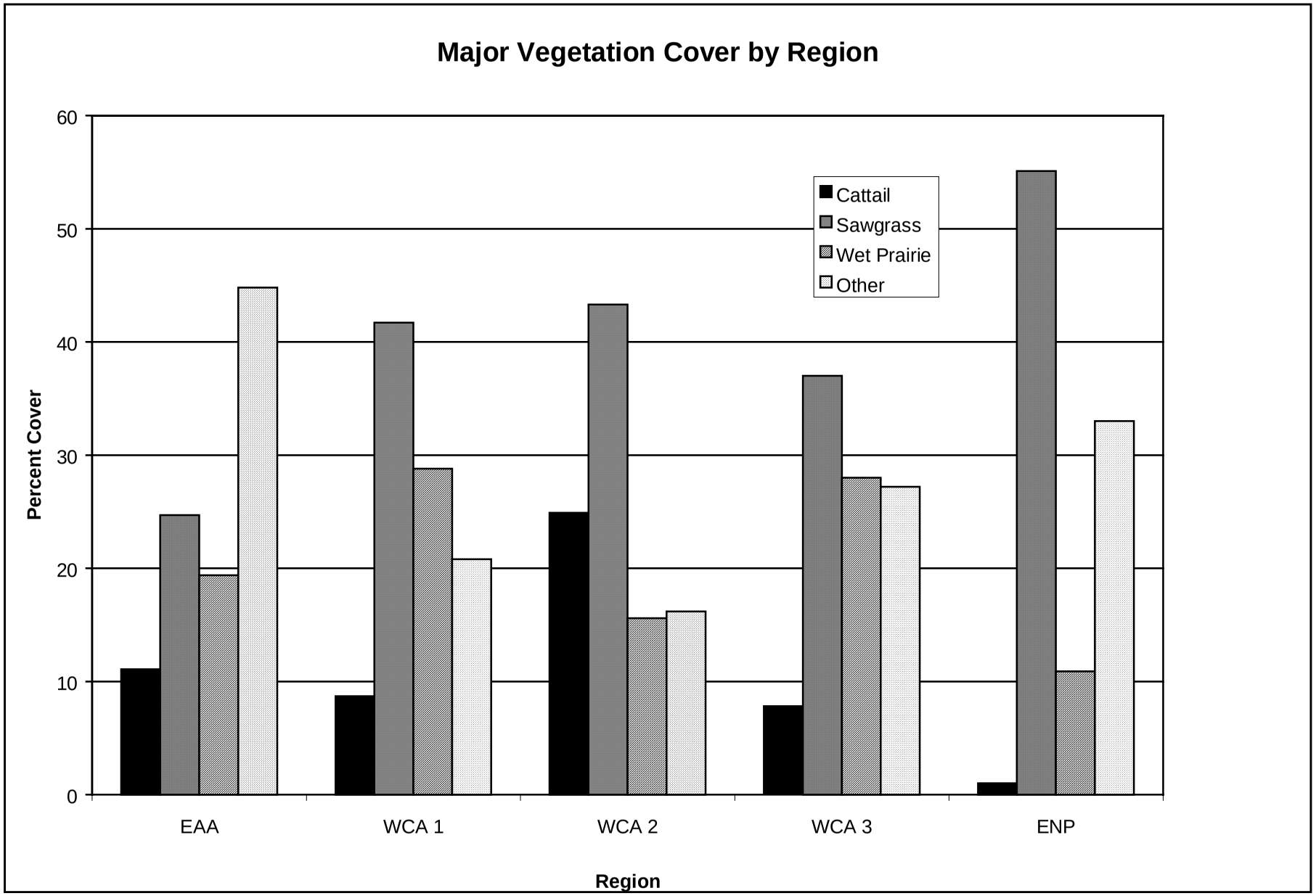

Figure 4. Major vegetation cover by region – Cycles 4 and 5 combined.

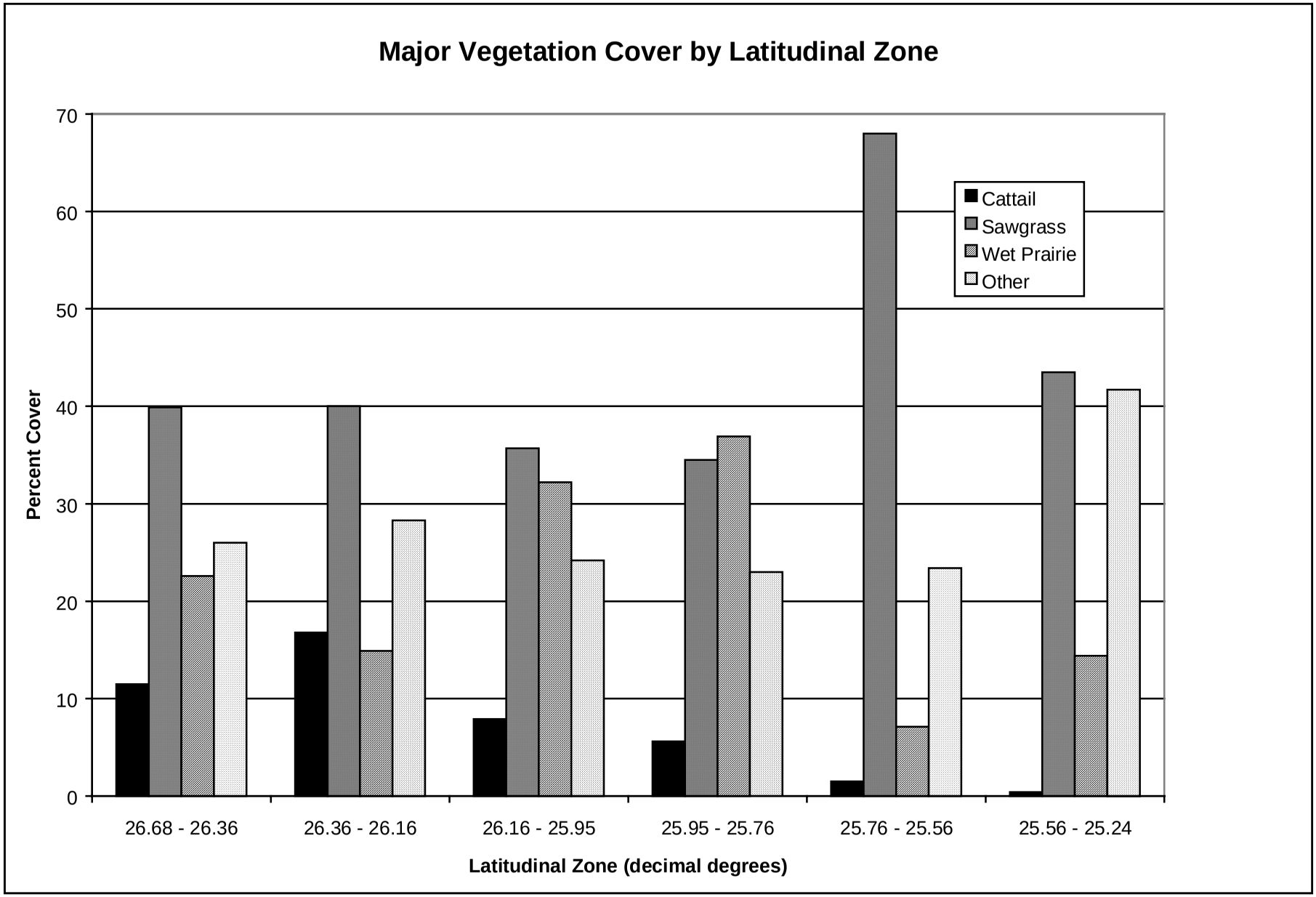

Figure 5. Major vegetation cover by latitudinal zone – Cycles 4 and 5 combined.

most southern zones. Wet prairie decreases considerably at the northern border of ENP (25.76^{o}), most likely due to the blockage of water flow by state highway 41 running east-west at this location (see Figure 1). Other vegetation cover is distributed fairly evenly across latitudinal zones with the highest coverage in the southern most zone made up mainly of mangrove scrub and forest vegetation.

DATA ANALYSIS

The spatial distribution of the four major vegetation classes was analyzed over the entire study area in a 1:180,000-scale map that was provided to the EPA. A page-size version of this map, Figure 6, shows the proportion of vegetation cover in each monitoring site represented by a pie chart. The slices of the pie chart represent the relative areas of the four major vegetation classes within the 1 km^2 plots. Pie charts representing Cycle 4 monitoring sites are outlined in blue, while those representing Cycle 5 sites are outlined in red. Sites in which periphyton existed in greater than 25% of the 1 km^2 plots are indicated with an asterisk placed at the center of the pie chart. It should be noted that given the difficulties in consistently identifying periphyton, as well as its transitory/seasonal nature, periphyton identification should not be considered definitive but rather indicative of potential areas of excessive periphyton growth.

The graphs depicted on the map represent histograms of dominant and secondary vegetation types, generalized into the four major vegetation classes. The smaller histograms summarize the total area included in each generalized class by region, namely: WCA 1, WCA 2, WCA 3, Rotenberger/Holey Land EAA and ENP. Background colors in these histograms correspond to the colors of the region that is represented. The larger histograms, with white backgrounds, summarize the total area included in each generalized class by latitudinal zone, as specified by the EPA.

In addition to representing major vegetation cover at each monitoring site, Figure 6 also provides spatial information on vegetation trends and characteristics by region and by latitudinal zone. For example, pie charts colored more than one half in dark blue and denoting monitoring sites dominated by wet prairie, are clustered within WCA 1, in the lower two-thirds of WCA 3 and within two particular areas of ENP. The distribution of predominantly wet prairie monitoring sites in the WCAs can be correlated with man-made structures such as canals and roadways that restrict hydrologic flow and tend to pool water, while the two clusters of wet prairie sites in ENP occur within natural features, namely, Shark River Slough and Taylor Slough. The distribution of sites containing considerable proportions of cattail (colored red) are also grouped within WCA 2, the north and east portions of WCA 3 and the northeastern section of ENP. These sites appear to coincide with canals and may warrant further investigation of spatial correlations with nutrient levels within the system.

In order to determine if the proportion of vegetation types and areal coverage within the monitoring sites is representative of vegetation distributions over the entire Everglades study area, a comparison was made between the percent cover of ten general vegetation classes as mapped within a subset of the monitoring sites and within the

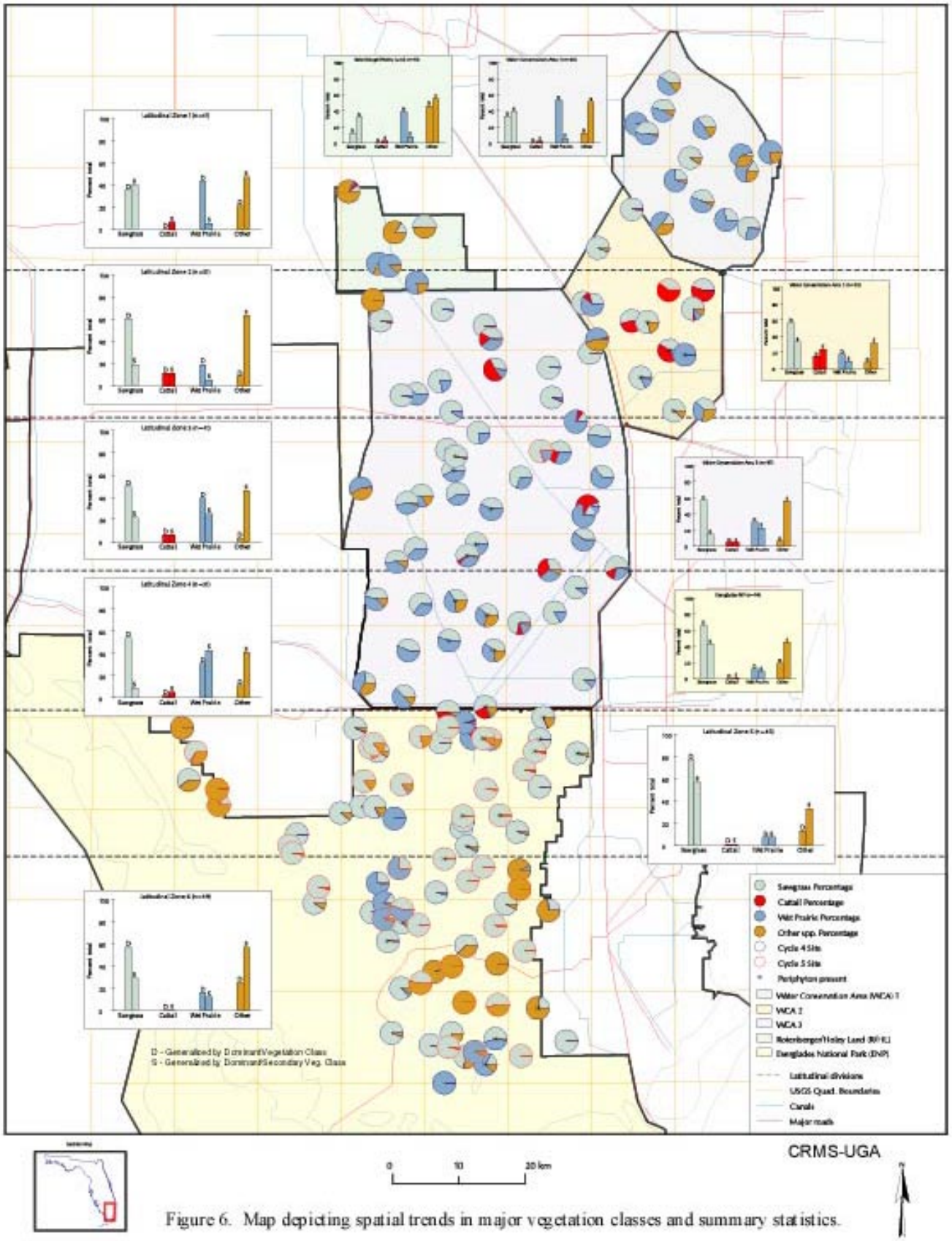

Figure 6. Map depicting spatial trends in major vegetation classes and summary statistics.

corresponding area in existing databases. Figure 7 depicts 30 monitoring sites in the northern portion of WCA3 (WCA3_N) that correspond with the existing WCA3 vegetation database (shaded in grey). Likewise, 44 monitoring sites corresponded with the northern portion of the ENP (ENP_N) vegetation database. The percent cover of vegetation was tallied for ten general classes defined by the EPA as sawgrass, wet prairie, muhly grass, cattail, mixed graminoid, non-graminoid emergent, bayhead, pine/hardwood, water and other vegetation (Table 7). Results show that there is a high degree of correspondence between the percent cover of vegetation types in the monitoring sites of both WCA3_N and ENP_N with the percent cover derived from the existing databases. The greatest difference was only 7.1 percent for sawgrass in ENP_N, and the difference for all other vegetation types was less than 4 percent. The average difference in percent cover for vegetation types in ENP_N was 1.5 percent and the average for WCA3_N was 0.4 percent.

Table 7. Percent Cover of Vegetation in Monitoring Sites and Corresponding Areas in Existing Databases

Vegetation Classes	% Cover ENP_N Existing Database	% Cover ENP_N Monitoring Sites	% Diff.	% Cover WCA3_N Existing Database	% Cover WCA3_N Monitoring Sites	% Diff.
Sawgrass	85.2	92.3	-7.1	68.7	69.6	-0.9
Wet Prairie	0.7	0.2	0.5	10.2	11.5	1.3
Muhly Grass	1.8	2.1	-0.3	0	0	0
Cattail	1.1	0.7	0.4	11.3	10.9	0.4
Mixed Graminoid	2.6	0.1	2.5	0	0	0
Non-gram. Emergent	0.1	0	0.1	2.9	2.7	0.2
Bayhead	1.7	1.6	0.1	0	0	0
Pine/ Hardwood	0	0	0	0	0	0
Other Vegetation	6.0	2.3	3.7	6.5	5.2	1.3
Water	0.8	0.7	0.1	0.4	0.1	0.3

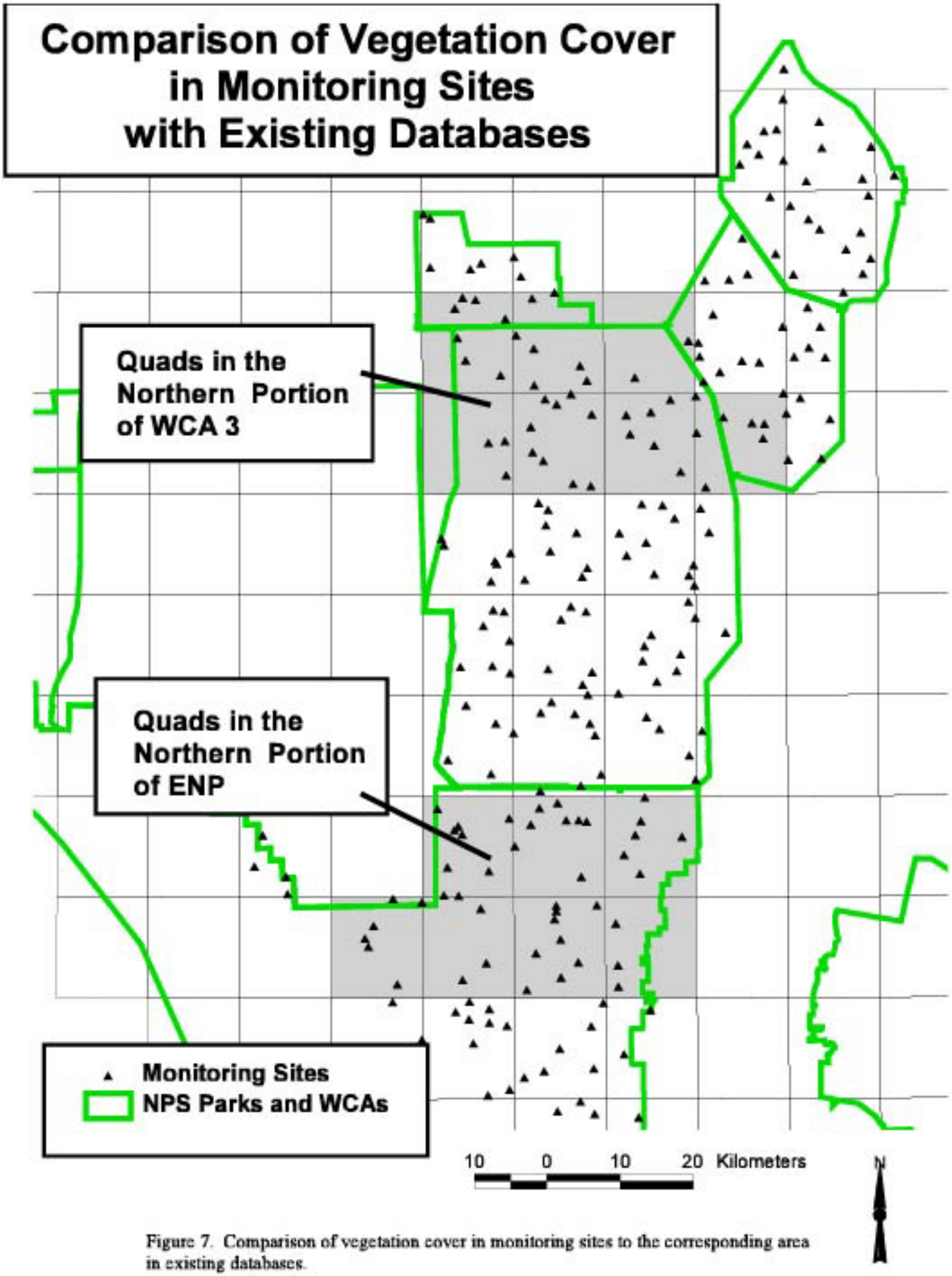

Figure 7. Comparison of vegetation cover in monitoring sites to the corresponding area in existing databases.

Figures 8 through 11 depict isolines representing predicted percentages of cover across the study area for each of the four major vegetation classes. Interpolation of percent cover data between monitoring sites was achieved using the kriging method in ArcInfo with a function to model the semi-variance of the data. Combining Cycle 4 and 5 monitoring sites, Figure 8 illustrates relatively high proportions of cattail in WCA 2, WCA 3 and the border of ENP and WCA 3. Relatively even percentages of sawgrass were interpolated throughout the study area (Figure 9), while wet prairie isolines in Figure 10 reveal higher percentages within the Water Conservation Areas and the slough areas of ENP. As expected, the highest levels of "other" vegetation are inside the Rotenberger/Holey Land EAA, largely due to abandoned agriculture in the EAA and the higher elevation pinelands area in ENP (Figure 11). These results illustrate the possibility of extrapolating information gathered within sample sites to the greater Everglades Ecosystem study area using spatial data analysis techniques such as kriging interpolation.

PRODUCTS DELIVERED TO EPA

Products delivered to the EPA include digital GIS database files, hardcopy maps, digital files for printing hardcopy maps and tabular areal summary data files. A detailed list of delivered products is provided in Table 8.

Table 8. List of Products Delivered to EPA

Data Type	Data Products
GIS Database Files (UTM NAD 83)	• AutoCAD DXF files of vegetation distributions for pilot study sites. • Digital Arc/Info coverages of vegetation distributions for each 1 km^2 map segment in Arc/Info Export format. • Point coverages of EPA monitoring sites for Cycles 4 and 5. • Ancillary Arc/Info coverages of boundaries, roads and canals in Export format.
Hardcopy Maps And Digital Files for Producing Hardcopy Maps	• 250 page-size detailed vegetation maps for 1 km^2 areas surrounding EPA monitoring sites. • 1:180,000 – scale and page-size (8.5 x 11 inches) maps of the entire study area with vegetation summaries and histograms depicting trends in cattail, sawgrass, wet prairie and other vegetation classes. • Page-size maps depicting interpolated vegetation distributions (percent cover) between monitoring sites for cattail, sawgrass, wet prairie and other vegetation classes. • EPS files for plotting maps of vegetation for each EPA monitoring station.

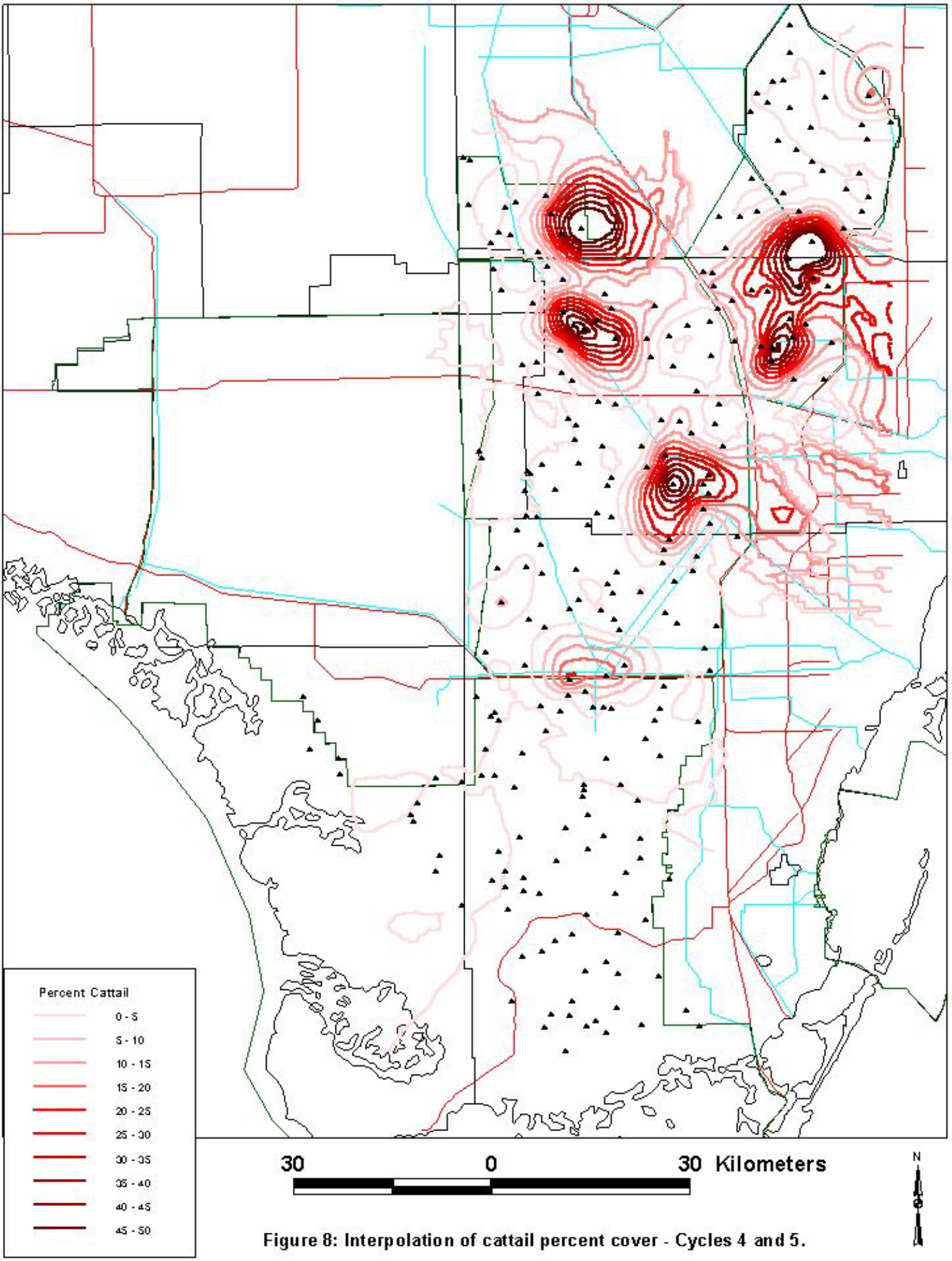

Figure 8: Interpolation of cattail percent cover - Cycles 4 and 5.

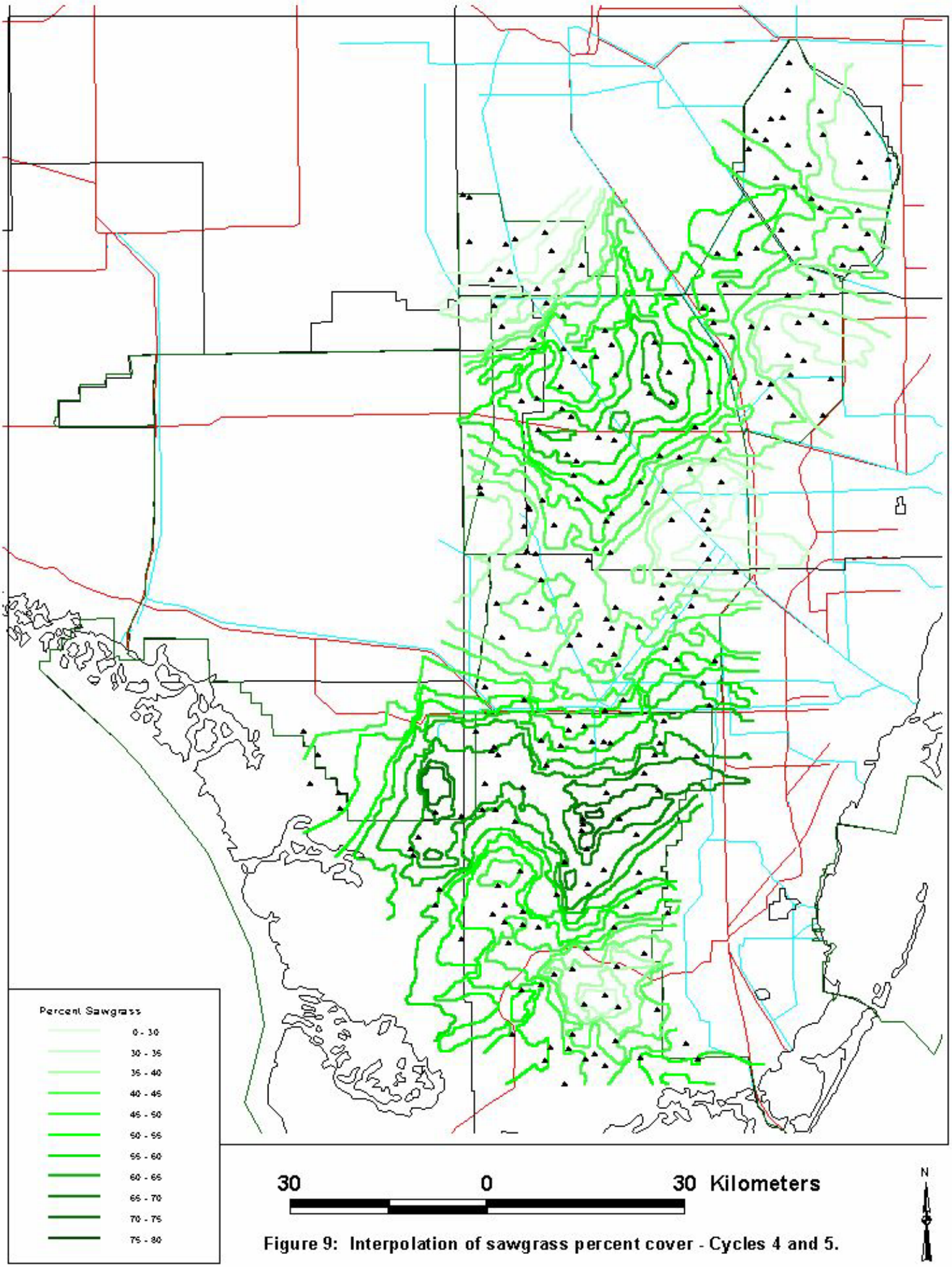

Figure 9: Interpolation of sawgrass percent cover - Cycles 4 and 5.

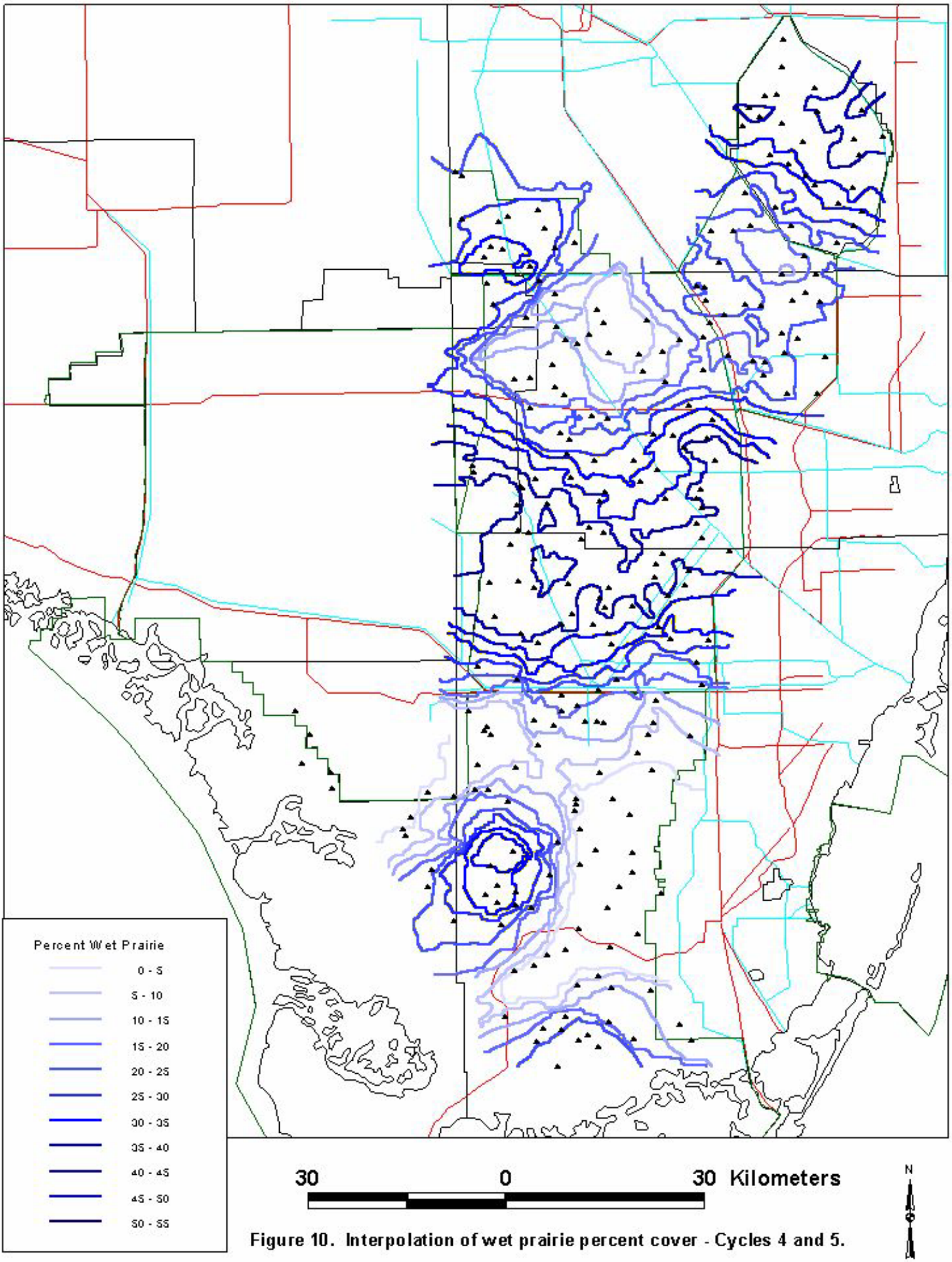

Figure 10. Interpolation of wet prairie percent cover - Cycles 4 and 5.

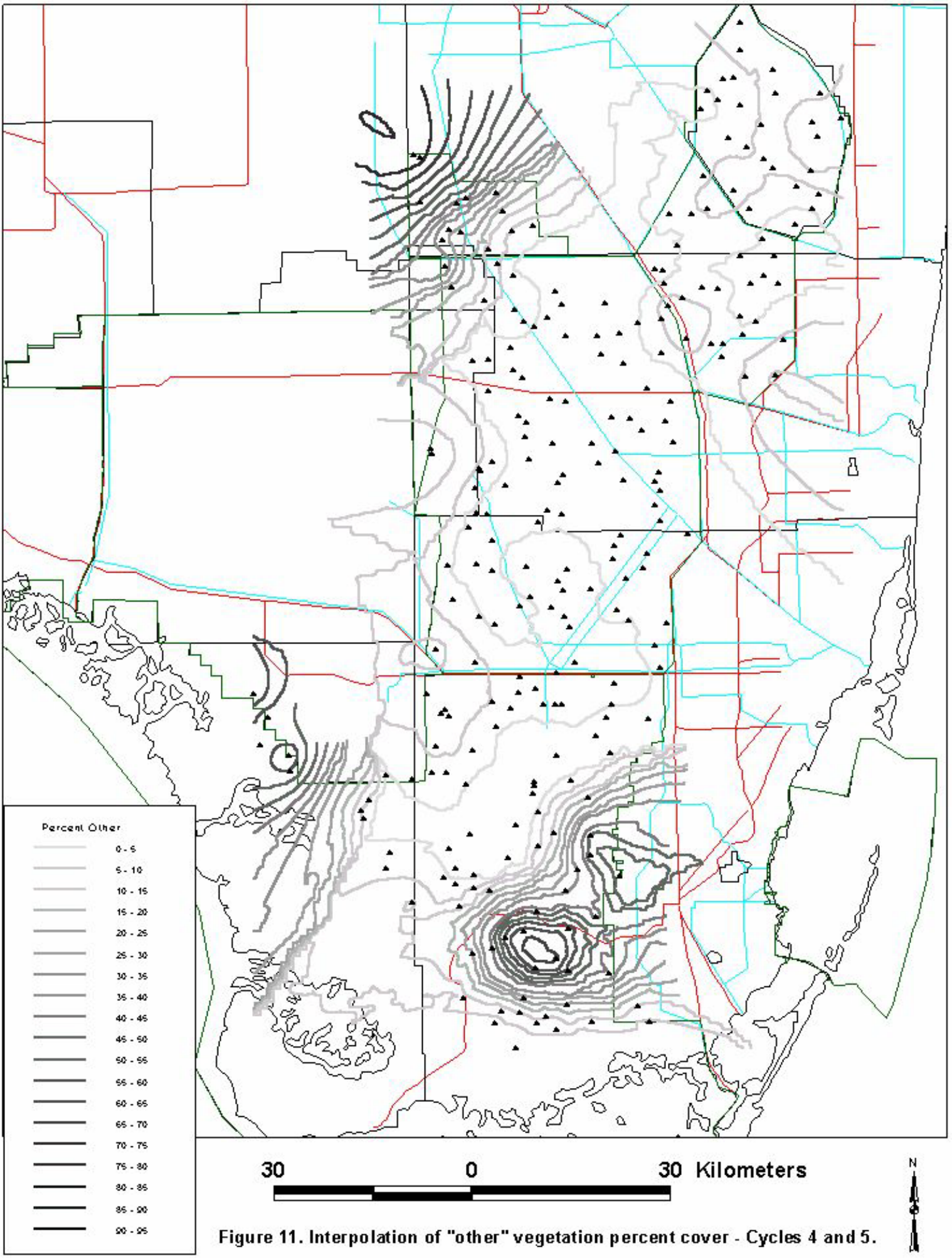

Figure 11. Interpolation of "other" vegetation percent cover - Cycles 4 and 5.

Table 8. List of Products Delivered to EPA (Continued)

Data Type	Data Products
Tabular Summary Data Files	• Unique combinations of dominant, secondary and tertiary vegetation for each monitoring station – area and percent cover – in text format. • Unique combinations of dominant and secondary vegetation for each monitoring station – area and percent cover – in Excel and text formats. • Unique combinations of dominant vegetation for each monitoring station – area and percent cover – in Excel and text formats. Four major vegetation classes summarized by region and latitudinal zone– area and percent cover – in Excel and text formats.

SUMMARY

Remote sensing and GIS techniques were successfully used to assess vegetation patterns over the Florida Everglades as part of the EPA South Florida Ecosystem Assessment Project. Vegetation communities within 1 km^2 plots centered on 250 EPA monitoring sites distributed in a north-south corridor throughout the Everglades were: 1) extracted from existing Everglades vegetation databases created by the CRMS, NPS and SFWMD from CIR aerial photographs; and 2) derived from USGS DOQQs. Areal statistics for dominant, secondary and tertiary vegetation types identified in the 250 1 km^2 plots provided EPA with spatially explicit vegetation data that can be correlated with environmental data collected at the monitoring sites.

Analysis of areal summary statistics indicated general trends over the Everglades ecosystem study area such as the diminishing coverage of cattail ranging from 12 and 17 percent in the northern most latitudinal zones to 0.4 percent in the southern most latitudinal zone. Wet prairie vegetation was found to cover greater percentages of the WCAs than the ENP and ENP contained the highest percentage of sawgrass. The EAA and ENP regions also contained the highest coverage of "other" vegetation. These patterns of major vegetation distributions over the entire study area were depicted in a map specially designed to visualize general trends in areal summary statistics. In addition, a comparison of areal statistics for monitoring sites with statistics derived from full-coverage vegetation databases confirmed randomly selected 1 km^2 plots adequately represented vegetation cover in the South Florida Ecosystem Assessment Project study area. Spatial interpolation of vegetation cover between monitoring sites also demonstrated the possibility of extrapolating sampled vegetation data to the broader landscape.

The 1994/1995 vegetation distributions documented in this study are now a baseline against which changes can be measured. It is anticipated that these

methodologies can be used to efficiently monitor future vegetation and spatially analyze change as an indicator of biogeochemical fluctuations in the Everglades Ecosystem.

ACKNOWLEDGMENTS

This study was sponsored by the U.S. Environmental Protection Agency (EPA) and the U.S. Department of Interior National Park Service (Cooperative Agreement number 5280-4-9006). The authors wish to express their appreciation to Andrew Homsey, formerly of the Center for Remote Sensing and Mapping Science (CRMS), The University of Georgia and currently with the U.S. Department of Commerce National Geodetic Survey, Bethesda, Maryland. The assistance of Ken Rutchey and Les Vilchek of the South Florida Water Management District (SFWMD), West Palm Beach, Florida is greatly appreciated.

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Attachment A

Everglades Vegetation Classification System for South Florida National Parks

By

David Jones [1], Marguerite Madden [2], Jim Snyder [3] and Ken Rutchey [4]

Draft Report of March 1998

1 Everglades National Park
2 Center for Remote Sensing and Mapping Science, The University of Georgia
3 Big Cypress National Preserve
4 South Florida Water Management District

Based on a review of several vegetation classification schemes developed by researchers of Everglades National Park and Big Cypress National Preserve, including a classification scheme devised by Craighead (1971), the following Vegetation Classification System was developed by the South Florida Natural Resources Center, Everglades National Park (ENP), the Center for Remote Sensing and Mapping Science (CRMS) at the University of Georgia, Big Cypress National Preserve (BICY) and the South Florida Water Management District (SFWMD) for use in mapping the vegetation of Everglades National Park, Big Cypress National Preserve, Biscayne National Park (BISC) and the SFWMD Water Conservation Areas.

Major Vegetation Types

I. Forest
II. Scrub
III. Savanna
IV. Prairies and Marshes
V. Shrublands
VI. Exotics
VII. Additional Categories
VIII. Special Modifiers

Under these major vegetation types are hierarchically arranged Plant Communities (classes) which are defined by typical dominant species. The species listed under these classes and subclasses were derived from South Florida Research Center Reports (1980-1983) for Everglades and Big Cypress National Parks, Craighead (1971), and Davis and Ogden (1994). The communities used in this classification system were selected from among those compiled in a summary report of all plant communities outlined by Craighead (1971) as well as those reported in vegetation studies published by the South Florida Natural Resources Center from 1980 to 1983.

Major Vegetation Types and Associated Plant Communities

I.	FOREST[1]		F
	A.	Mangrove Forest	FM
		1. Red *(Rhizophora mangle)* Mangrove	FMr
		2. Black *(Avicennia germinans)* Mangrove	FMa
		3. White *(Laguncularia racemosa)* Mangrove	FMl
		a. White Mangrove or Buttonwood Forest[2]	FMlb
		4. Mixed mangrove[3]	FMx
	B.	Buttonwood (*Conocarpus erectus*) Forest[4]	FB
	C.	Subtropical Hardwood Forest[5]	FT
	D.	Oak-Sabal Forest[6]	FO
	E.	Paurotis Palm *(Acoelorrhaphe wrightii)* Forest	FP
	F.	Cabbage Palm *(Sabal palmetto)* Forest	FC
	G.	Swamp Forest	FS
		1. Mixed Hardwood Swamp Forest[7]	FSh
		2. Cypress Strands[8]	FSc
		a. Cypress Domes/Heads[9]	FSd
		3. Cypress-Mixed Hardwoods[10]	FSx
		4. Mixed Hardwoods, Cypress and Pine[11]	FSa
		5. Cypress-Pines[12]	FSCpi
		6. Bayhead[13]	FSb

[1]High-density stands of trees with heights over 5 metres.

[2] This class signifies that it is uncertain whether vegetation is white mangrove (*Laguncularia racemosa*) or buttonwood forest (*Conocarpus erectus*), since signatures on the aerial photographs are very similar. Fieldchecking is required to correctly identify the species.

[3]Specific mixtures of mangrove species, when identified, will be distinguished as subgroups.

[4]*Conocarpus erectus* with variable mixtures of subtropical hardwoods.

[5]*Lysiloma latisiliquum, Quercus virginiana, Bursera simaruba, Mastichodendron foetidissimum, Swietenia mahagoni*, among others.

[6]*Quercus laurifolia, Q. virginiana, Sabal palmetto*.

[7]*Quercus. virginiana, Q laurifolia, Acer rubrum, Sabal palmetto, Fraxinus caroliniana.*

[8]*Taxodium ascendens, T. distichum*; cypress domes are treated as a subgroup. Cypress strands (especially in BICY) may contain an understory of species such as *Annona glabra, Chrysobalanus icaco,* and *Fraxinus caroliniana.*

[9]*Taxodium ascendens, T. distichum* ; cypress growing in a depression such that trees in the center are tallest and give the characteristic dome shape. Delineated domes may contain a fringe of short cypress (less than 5 metres).

[10]*Taxodium ascendens* and *T. distichum* with variable mixtures of subtropical and temperate hardwoods; predominantly in BICY.

[11]Mixture of various subtropical hardwoods with *Taxodium distichum* with occassional *Pinus elliottii* var. *densa.*

[12]*Taxodium distichum* with *Pinus elliottii* and a mixed hardwood scrub understory.

II.	SCRUB[14]		S
	A.	Mangrove Scrub[15]	SM
		1. Red (*Rhizophora mangle*)	SMr
		2. Black (*Avicennia germinans*)	SMa
		3. White (*Laguncularia racemosa*)	SMl
		a. White Mangrove or Buttonwood Scrub[16]	SMlb
		4. Mixed scrub[17]	SMx
	B.	Buttonwood (*Conocarpus erectus*) Scrub	SC
	C.	Saw Palmetto *(Serenoa repens)* Scrub	SP
	D.	Hardwood Scrub[18]	SH
	E.	Bay-Hardwood Scrub[19]	SS
III.	SAVANNA[20]		SV
	A.	Pine (*Pinus elliottii* var. *densa*) Savanna	SVPI
		1. Slash pine mixed with palms[21]	SVx
		2. Slash pine with hardwoods[22]	SVPIh
		3. Slash pine with cypress[23]	SVPIc
	B.	Cypress (*Taxodium distichum* and *T. ascendens*) Savanna	SVC

---

[13]*Magnolia virginiana, Annona glabra, Chrysobalanus icaco, Persea borbonia, Ilex cassine, Metopium toxiferum*, among others.

[14]Low-density areas of trees and shrubs with heights under 5 meters.

[15]The vegetation matrix in which the scrub occurs should be noted, *e.g.*, within *Eleocharis* marsh.

[16] This class signifies that it is uncertain whether vegetation is scub white mangrove (*Laguncularia racemosa*) or buttonwood scrub (*Conocarpus erectus*), since signatures on the aerial photographs are very similar. Fieldchecking is required to correctly identify the species.

[17]Sparse and high-density subgroups/modifiers can be distingished.

[18]Includes species such as *Metopium toxiferum, Persea borbonia, Myrica cerifera, Ilex cassine, Magnolia virginiana, Myrsine floridana, Conocarpus erectus, Chyrsobalanus icaco* and others. Often contains a moderate to heavy component of mixed grasses. Scrub oak (*Quercus virginiana*) is often included in areas of BICY.

[19]Mixed association of bayhead swamp species, buttonwood scrub and hardwood scrub species such as *Myrica cerifera, Chyrsobalanus icaco*, leather fern (*Acrostichum danaeifolium*), *Conocarpus erectus* and *Cladium jamaicense.* Minor species include *Metopium toxiferum, Ilex cassine, Persea borbonia, Sabal palmetto* and *Cephalanthus occidentalis.* Occurs in the transition zone between saline and fresh environments.

[20]Low-density (open canopy) trees in a matrix of graminoids.

[21]*Pinus elliottii* var. *densa, Serenoa repens, Sabal palmetto*; typical of BICY.

[22]*Pinus elliottii* var. *densa, Rhus copallina, Guettarda scabra, Bumelia salicifolia, Tetrazygia bicolor, Dodonea viscosa*, among others; typical of EVER.

[23]*Pinus elliottii* var. *densa* dominant with *Taxodium distichum* interspersed.

	1.	Dwarf cypress[24]	SVCd
	2.	Cypress with pine[25]	SVCpi
C.		Palm *(Sabal palmetto)* Savanna	SVPM
IV.		PRAIRIES AND MARSHES	P
A.		Graminoid Prairie/Marsh[26]	PG
	1.	Black rush *(Juncus roemerianus)*	PGj
	2.	Sawgrass *(Cladium jamaicense)*[27]	PGc
	3.	Muhly grass *(Muhlenbergia filipes)*	PGm
	4.	Cordgrass *(Spartina* spp.*)*	PGs
	5.	Spike rush *(Eleocharis cellulosa)*	PGe
	6.	Common reed (*Phragmites* spp.)	PGp
	7.	Maidencane (*Panicum hemitomon*)	PGa
		a. Maidencane-Spike rush[28]	PGw
	8.	Mixed graminoids[29]	PGx
B.		Non-graminoid Emergent Marsh[30]	PE
	1.	Broadleaf Emergents	PEb
	2.	Floating/Floating Attached Emergents	PEf
C.		Cattail (*Typha* spp.) Marsh	PC
D.		Halophytic Herbaceous Prairie	PH
	1.	Graminoid[31]	PHg
	2.	Succulent[32]	PHs
E.		Prairie with Scattered Pines[33]	PPI

[24]Cypress of stunted growth less than 5 metres in height.

[25]*Taxodium distichum* and *T.ascendens* dominant with mixed *Pinus elliottii* var. *densa.*

[26]Contains grasses, sedges and rushes. The extent of periphyton cover is expressed as a modifier for all appropriate subclasses.

[27]The modifier 't' is used to distinguish tall sawgrass, *e.g.*, PGct.

[28] Mix of shallow open water, *Eleocharis* spp. and *Panicum hemitomon* which can include sparse associations of low stature *Cladium jamaicense*, *Typha* spp., *Sagittaria lancifolia*, *Pontedaria lanceolata*, *Nymphaea* spp., etc. typical of SFWMD impounded conservation areas.

[29]Specific mixtures of graminoids, when identified, will be distinguished as subgroups.

[30]*Pontederia lanceolata*, *Sagittaria* spp., *Nymphaea odorata*, *Typha* spp., with *Ludwigia repens* and *Utricularia* spp. as possible submergents.

[31] Saltgrass (*Distichlis spicata*), smutgrass (*Sporobolus* spp.) and keys grass (*Monanthocloe littoralis*).

[32]Very salt tolerant species such as saltwort (*Batis maritima*), glasswort (*Salicornia* spp.) and sea purslane (*Sesuvium* spp.).

[33]Sparsely distributed *Pinus elliottii* var. *densa* in a matrix of graminoids, at the pinelands-glades ecotone.

V.	SHRUBLANDS	SB
A.	Willow (*Salix caroliniana*)	SBs
B.	Pop Ash (*Fraxinus caroliniana*)	SBf
C.	Wax myrtle (*Myrica cerifera*)	SBm
D.	Groundsel bush (*Baccharis* spp.)	SBb
E.	Buttonbush (*Cephalanthus occidentalis*)	SBc
F.	Primrose (*Ludwigia* spp.)	SBl
G.	Cocoplum (*Chrysobalanus icaco*)	SBy
VI.	EXOTICS[34]	E
A.	Cajeput (*Melaleuca quinquenervia*)	EM
B.	Australian Pine (*Casuarina* spp.)	EC
C.	Lather Leaf (*Colubrina asiatica*)	EO
D.	Brazilian Pepper (*Schinus terebinthifolius*)	ES
E.	Shoebutton Ardisia (*Ardisia elliptica*)	EA
F.	Tropical Soda Apple (*Solanum viarum*)	EL
G.	Java Plum (*Syzygium cumini*)	EJ
VII.	ADDITIONAL CATEGORIES	
A. Open Water		W
B. Beaches		BCH
C. Mud		MUD
D. Cultural Areal Features		
1.	Structures and Cultivated Lawns	HI[35]
	a. Pumping Stations	HIp
	b. Disturbed Fish Camp Site	HId[36]
2.	Major Roads (greater than 30 m wide)	RD
3.	Major Canals (greater than 30 m wide)	C
4.	ORV Trails	ORV
E. Cultural Linear Features		
1. Secondary roads (less than 30 m)		(Dash)
2. Secondary canals (less than 30 m)		(Dash-Dot)
3. ORV trails (less than 15 m wide)		(brown)
a. Primary		
b. Secondary		

[34]For sparse to low-density stands, modifiers are used to indicate (1) the vegetation matrix in which the exotic occurs, and (2) the original vegetation replaced by the exotic, when applicable.

[35]Human Influence includes structures (e.g., buildings, fishing and hunting camps), parking lots and cultivated lawns.

[36] Human influence site common in SFWMD that has been disturbed by former fishing/hunting camp. Although buildings are no longer present, an unusual mix of introduced and exotic species persist.

c. Tertiary

F. Spoil Areas	SA
1. Artificial Deer Islands	SAd

VIII. SPECIAL MODIFIERS

A. Hurricane Damage Classes	
1. Low to medium (0% to 50% damage)	- 1
2. High (51% to 75% damage)	- 2
3. Extreme (> 75% damage)	-3
B. Low Density (Scattered Individuals)	- 4
C. Human Influence[37]	- 5
1. Abandoned agriculture	- 6
2. Altered drainage	- 7
3. High density ORV trails	- 8
D. Periphyton	- 9
E. Treatment Damage (e.g., herbicide treatment)	-10
F. Other Damage (e.g., freeze damage)	-11
G. Ponds	-12
H. Exposed Rock (i.e., pinnacle rock)	-13

[37]The Human Influence modifier can be added to a vegetation class to indicate evidence of human disturbance.

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APPENDIX B: Quality Assurance Project Plan

EVERGLADES ECOSYSTEM ASSESSMENT
(PHASE II REMAP)

QUALITY ASSURANCE PROJECT PLAN

Prepared for

United States Environmental Protection Agency
Science and Ecosystem Support Division
Region 4 and Office of Research and Development

Prepared by

FTN Associates, Ltd.
3 Innwood Circle, Suite 220
Little Rock, AR 72211

July 31, 2000

FORWARD

This document is the Quality Assurance Project Plan (QAPP) for environmental data operations performed by the US Environmental Protection Agency (EPA) and a consortium of groups as part of the Investigation of Mercury Contamination in the Everglades Ecosystem and Everglades Ecosystem Assessment (Phase II REMAP) Project. This document generally follows on *Requirements for QA Project Plans for Environmental Data Operations* (EPA QA/R-5).

The project will be conducted in three phases: planning, implementation, and assessment. The first phase involved the development of Data Quality Objectives (DQOs), which provided statements about the expectations and requirements of the various data users. In the second phase, the QAPP and its associated documentation translates these requirements into measurement performance specifications and quality assurance/quality control (QA/QC) procedures for the data suppliers to provide the information needed to satisfy the data user's needs. Once the data have been collected and validated in accordance with the elements of the QAPP, the data will be evaluated to determine whether the DQOs have been satisfied. In this assessment phase, the data will be analyzed to determine whether they meet the assumptions made during planning and whether the total error in the data is small enough to support decisions within tolerable decision error rates expressed by the data users. Plans for data validation and assessment of the data are discussed in the final sections of the QAPP.

Although there is no agency-wide template for QAPP format, this QAPP follows organizational consistency and content of the current EPA guidance for such documents. In addition, this document has been prepared under the EPA Region IV jurisdiction and will be reviewed and approved following pilot-scale testing of project protocols (currently scheduled for early 1999) and prior to implementation of the wet and dry season sampling elements of the project.

This QAPP documents how QA/QC activities will be planned and implemented. Overall, the QAPP provides sufficient detail to demonstrate the following:

- The project's technical and quality objectives are identified and agreed upon.
- The intended measurements or data acquisition methods are consistent with project objectives.
- The assessment procedures are sufficient for determining if data of the type and quality needed and expected are obtained.
- Limitations on the use of the data can be identified and documented.

Project documents that have been prepared prior to the QAPP (e.g., standard operating procedures [SOPs], test plans, and sampling plans) are appended or, in some cases, incorporated by reference.

The elements of this QAPP are categorized into "groups" according to their function and include the following.

Group A: Project Management

This group of QAPP elements covers the general areas of project management, project history and objectives, and roles and responsibilities of the participants. The following elements ensure that the project's goals are clearly stated, that all participants understand the goals and the approach to be used, and that project planning is documented:

- Title and Approval Sheet,
- Table of Contents and Document Control Format,
- Distribution List,
- Project/Task Organization and Schedule,
- Problem Definition/Background,
- Project/Task Description,
- Quality Objectives and Criteria for Measurement Data,
- Special Training Requirements/Certification, and
- Documentation and Records.

Group B: Measurement/Data Acquisition

This group of QAPP elements covers the aspects of measurement system design and implementation so that appropriate methods for sampling, analysis, data handling, and QC are employed and will be documented. These elements are primarily contained in attachments to the QAPP:

- Sampling Process Design (Experimental Design);

- Sampling Methods Requirements;
- Sample Handling and Custody Requirements;
- Analytical Methods Requirements;
- Quality Control Requirements;
- Instrument/Equipment Testing, Inspection, and Maintenance Requirements;
- Instrument Calibration and Frequency;
- Inspection/Acceptance Requirements for Supplies and Consumables; and
- Data Management.

Group C: Assessment/Oversight

The purpose of assessment is to ensure that the QAPP is implemented as prescribed. This group of QAPP elements addresses the activities for assessing the effectiveness of the implementation of the project and the associated QA/QC activities:

- Assessments and Response Actions, and
- Reports to Management.

Group D: Data Validation and Usability

Implementation of Group D elements ensures that the individual data elements conform to the specified criteria, thus enabling reconciliation with the project's objectives. This group of elements covers the QA activities that occur after the data collection phase of the project has been completed:

- Data Review, Validation, and Verification Requirements;
- Validation and Verification; and
- Reconciliation with Data Quality Objectives.

The organizational group performing the work is also responsible for implementing the approved QAPP. This responsibility includes ensuring that all personnel involved in the work have copies of or access to the approved QAPP along with all other necessary planning documents. In addition, the group must ensure that these personnel understand their requirements prior to the start of data generation activities.

Moreover, these organizations are responsible for keeping the QAPP current when changes to technical aspects of the project change. QAPPs must be revised to incorporate such changes and must be re-examined to determine the impact of the changes. Any revisions to the QAPP must be re-approved and distributed to all participants in the project.

EVERGLADES AND ECOSYSTEM ASSESSMENT

(PHASE II REMAP)

QUALITY ASSURANCE PROJECT PLAN

APPROVAL SHEET

	Date of the Revision	Reviewer Signature	Date
Technical Project Manager, Jerry Stober, PhD	6/1/99		
EPA QA Officer, Gary Bennett	6/1/99		
Laboratory Reviewers			
SERP Director, Ron Jones, PhD	6/1/99		
SESD Representative, Jenny Scifres	6/1/99		
Battelle Representative, Brenda Lasorsa	6/1/99		

EVERGLADES AND ECOSYSTEM ASSESSMENT

(PHASE II REMAP)

QUALITY ASSURANCE PROJECT PLAN

DISTRIBUTION LIST

NAME	DESCRIPTION	QAPP VERSION
Jerry Stober, PhD	EPA Region IV SESD Project Manager	6/1/99 [(1)]
Mike Birch	EPA Region IV Office of Quality Assurance	6/1/99 [(1)]
Gary Bennett	EPA Region IV Office of Quality Assurance	6/1/99 [(1)]
Ron Jones, PhD	Director, Southeast Environmental Research Program, Florida International University	6/1/99 [(1)]
Jenny Scifres	EPA Region IV SESD Laboratory	6/1/99 [(1)]
Brenda Lasorsa	Battelle Marine Science Laboratory, Sequim, WA	6/1/99 [(1)]
Stuart Ponder	Integrated Laboratory Systems, Inc.	6/1/99 [(1)]
Kent Thornton, PhD	FTN Associates, Ltd.	6/1/99 [(1)]

[(1)] with 9/9/99, 9/23/99, and 7/31/00 updates

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PROJECT/TASK ORGANIZATION

A1 PROBLEM DEFINITION/BACKGROUND

A1.1 Purpose/Background

The purpose of this project is to assess the risks to fish and wildlife from mercury contamination in the South Florida Everglades ecosystem. It is Phase II of the South Florida Ecosystem Assessment being conducted by US Environmental Protection Agency (EPA) Region IV SESD as their contribution to the South Florida Mercury Science Program and the Everglades restoration activities. The project organizational structure is provided as Figure A1.

A1.2 Problem Statement and Background

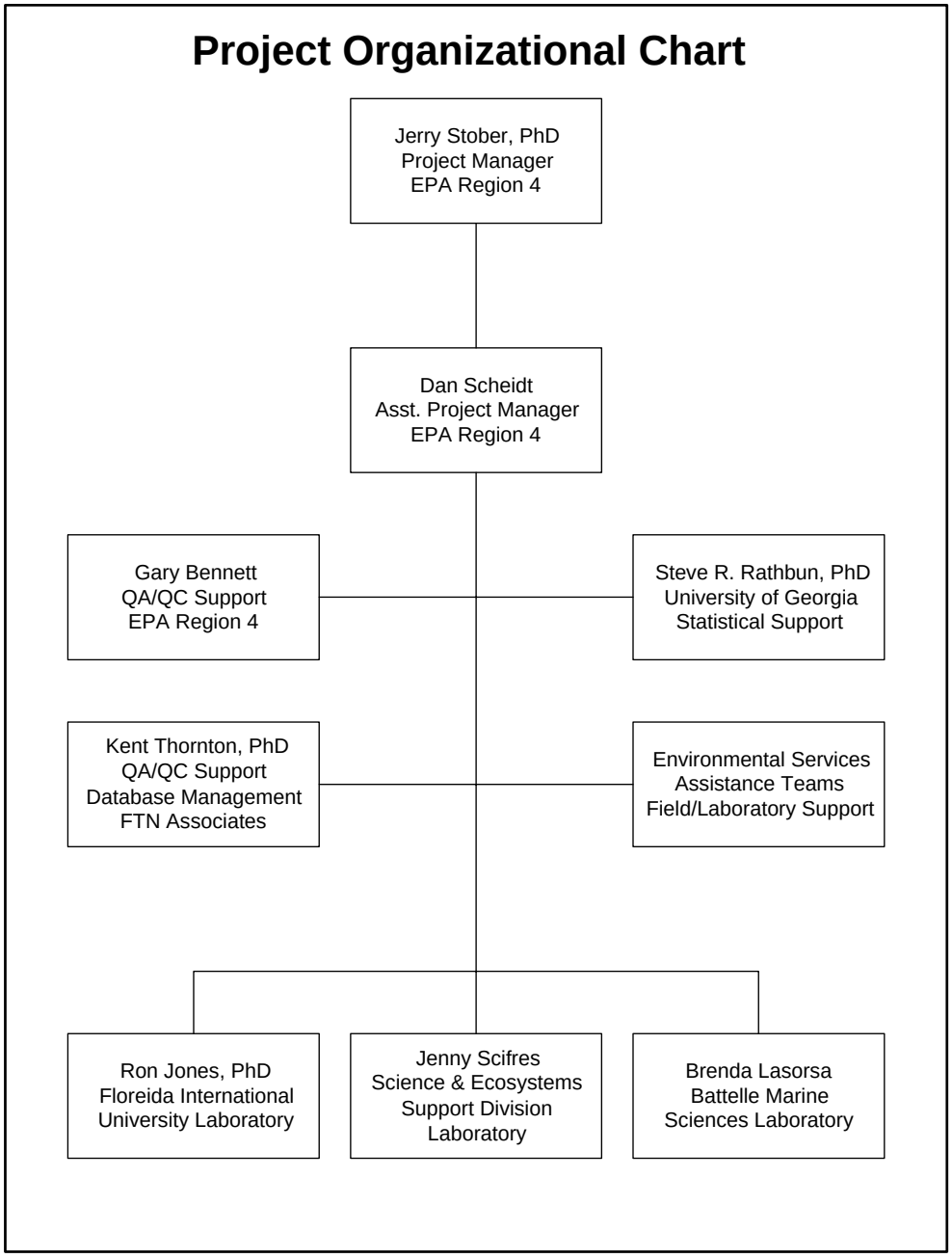
Over 2 million acres in South Florida are currently under fish consumption advisories because of mercury contamination. The risks to fish and wildlife, particularly the threatened and endangered species (e.g., Florida panther, woodstork), from mercury contamination are currently unknown. This risk assessment is being conducted as part of the larger Everglades ecosystem restoration program so that the risks from mercury contamination can be compared with the risks from hydroperiod modification, habitat alteration, nutrient enrichment, and introduction of exotic species.

A2 PROJECT/TASK DESCRIPTION AND SCHEDULE

A2.1 Purpose/Background

The purpose of Phase II is to provide decision makers with answers to 7 policy-relevant questions so that improved environmental decisions can be made on the multiple environmental issues and restoration efforts being conducted in South Florida. Phase II is an extension of the Phase I Interim Assessment conducted from 1994 through 1997.

Project Organizational Chart



A2.2 Description of the Work to be Performed

The Phase II REMAP statement of work (September 1998) (Attachment 1) provides the following information:

1) Measurements that are expected during the course of the project;

2) Applicable technical quality standards or criteria;

3) Any special personnel and equipment requirements that may indicate the complexity of the project;

4) The assessment techniques needed for the project;

5) A schedule for the work performed; and

6) Project and quality records required, including various reports needed.

A3 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

A3.1 Purpose/Background

The purpose of this element is to document the data quality objectives (DQOs) of the project and to establish performance criteria for the mandatory systematic planning process and measurement system that will be employed in generating the data.

A3.2 Specifying Quality Objectives

DQOs were prepared during the Phase I Interim Assessment (*South Florida Ecosystem Assessment Project Decision-Based Data Quality Objectives, March 1997*). These DQOs have been reviewed and updated to address Phase II. A copy of the project DQOs is included as Attachment 2.

A3.3 Specifying Measurement Performance Criteria

The DQO measurement performance criteria were established following Phase I and are listed in Table A1A of the Project DQO document (Attachment 2). Sampling and analytical

methods criteria specified under the elements contained in Section B are designed to meet the applicable criteria described in the DQO document.

A4 SPECIAL TRAINING REQUIREMENTS/CERTIFICATION

Not Applicable for this project.

A5 DOCUMENTATION AND RECORDS

A5.1 Purpose/Background

This element defines which records are critical to the project and what information needs to be included in reports, as well as the data reporting format and the document control procedures to be used. Required report formats are also discussed in Section D. Specification of the proper reporting format, compatible with data validation, will facilitate clear, direct communication of the project.

A5.2 Project Information Requirements

A5.2.1 Field Operation Records

- *Sample Collection Record* - To document that the proper sampling protocols were followed in the field. At a minimum, this documentation will include the names of the persons conducting the activity, sample number, sample collection points, maps and diagrams, equipment/method used, climatic conditions, and unusual observations as applicable. Field notebooks are used to record raw data and make references to prescribed procedures and changes in planned activities.

- *Chain-of-Custody Records* - To document the progression of samples as they travel from the original sampling location to the laboratory and finally to their disposal area, if applicable. Chain-of-custody forms will be required for all environmental samples.

- *QC Sample Records* - To document the generation of quality control (QC) samples such as field, (equipment) blank, and duplicate samples. Documentation of sample integrity and preservation along with calibration and standards traceability documentation capable of providing a reproducible reference point will be required for appropriate QC records. Quality control sample records will

contain information on the frequency, conditions, level of standards, and instrument calibration history.

- *General Field Procedures* - To document general field conditions and actions and outline potential areas of difficulty in gathering specimens. Field logs will be completed to address this documentation.

- *Corrective Action Reports* - Corrective action reports to show what methods were used in cases where general field or laboratory practices or other standard procedures were not followed and include the methods to resolve the issue.

A5.2.2 Laboratory Records

- *Sample Data* - Documentation of the times that samples were analyzed to verify that they met the holding times prescribed in the analytical methods. Included will be the overall number of samples, sample location information, any deviations from the SOPs, time of day, and date. Corrective action procedures to replace samples violating the protocol also will be documented.

- *Sample Management Records* - Sample management records document sample receipt, handling and storage, and scheduling of analyses. The records verify that the chain-of-custody and proper preservation were maintained, reflect any anomalies in the samples (such as receipt of damaged samples), note proper log-in of samples into the laboratory, and address procedures used to ensure that holding time requirements were met.

- *Test Methods* - Analyses to be performed are described in the Phase II Scope of Work (Attachment 1) and in Table A1. Attachments 4 through 6 describe how the analyses will be carried out in the project laboratories, including sample preparation and analysis, instrument standardization, detection and reporting limits, and test-specific QC criteria. Documentation demonstrating laboratory proficiency with each method used is included or is available for inspection.

- *QA/QC Reports* - These reports will include the general QC records, such as initial demonstration of capability, instrument calibration, routine monitoring of analytical performance, calibration verification, etc. Project-specific information from the quality assurance/quality control (QA/QC) checks such as blanks, spikes, calibration check samples, etc. will be included in these reports to facilitate data quality analysis.

Table A1. Measurement and analytical methods for Phase II laboratories.

Media/Parameter	SERP	SESD/ESAT	Battelle
Surface Water			
Dissolved Oxygen	--	EPA 360.1	--
pH	--	EPA 150.1	--
Temperature	--	EPA 170.1	--
Conductivity	--	EPA 120.1	--
Redox Potential	--	Voltage Meter	--
Water Depth	--	Calibrated Extensive Rod	--
Turbidity	--	EPA 180.1	--
Total Phosphorus	EPA 365.1(modified)	EPA 365.1	--
Total Nitrogen	Antek 7000N Analyzer	EPA 351.1 + (EPA 300 or 353.2) (1)	--
Ammonium-N (filtered-0.8)*	EPA 350.1	EPA 350.1	--
Nitrite-N (filtered)*	EPA 353.2	EPA 353.2 or EPA 300	--
Nitrate-N (filtered)*	EPA 353.2	EPA 353.2 or EPA 300	--
Soluble Reactive Phosphate*	EPA 365.1	EPA 365.1 or EPA 300	--
Total Organic Carbon	EPA 415.1 (modified)	EPA 415.2	--
Sulfate	--	EPA 300.0	--
Sulfate (filtered - 0.8)*	EPA 300.0	EPA 300.0	
Sulfide*	--	Hach	--
Alkaline Phosphatase	Experimental Methodology	--	--
Total Mercury	CVAF	CVAF	CVAF
Methyl Mercury	CVAF	--	CVAF
Pore Water*			
Total Phosphorus*	EPA 365.1	--	--
Total Nitrogen*	Antek 7000N Analyzer	--	--
Ammonium-N (filtered)*	EPA 350.1	--	--
Nitrite-N (filtered)*	EPA 353.2	--	--
Nitrate-N (filtered)*	EPA 353.2	--	--
Soluble Reactive Phosphate*	EPA 365.1	--	--
Bromide*	--	EPA 300.0	--
Chloride*	--	EPA 300.0	--
Fluoride*	--	EPA 300.0	--
Sulfate (ion)*	--	EPA 300.0	--
Sulfide*	--	Hach	--
Soil/Sediment			
Type	--	Visual Classification	--
Thickness	--	Visual Classification	--
Redox Potential (in situ)	--	Voltage Meter	--
Total Mercury	CVAF	CVAF	CVAF
Methyl Mercury	CVAF	--	CVAF
Sulfate	--	EPA 300.0	--
Total Phosphorus	EPA 365.1	--	--

(1) Sum of TKN + NO_2/NO_3

* = Parameter added for the Phase II analysis

Table A1. (Continued)

Media/Parameter	SERP	SESD/ESAT	Battelle
Ash Free Dry Weight	ASTM D2974-87	--	--
Bulk Density	ASTM D4531-86	--	--
Mineral Content	ASTM D 2974-87	--	--
Methane*	ASTM D 2974-87	--	--
Carbon Dioxide*	ASTM D 2974-87	--	--
Alkaline Phosphatase	Experimental Analytical Methodology	--	--
Periphyton - Utricularia			
Total Mercury	CVAF	CVAF	CVAF
Methyl Mercury	CVAF	--	CVAF
Diatoms	ASTM D 2974-87	--	--
Pigments	ASTM D 2974-87	--	--
Periphyton - Floating			
Total Mercury	CVAF	CVAF	CVAF
Methyl Mercury	CVAF	--	CVAF
Biomass*		–	--
Diatoms*	ASTM D 2974-87	--	--
Pigments	ASTM D 2974-87	--	--
Media: Periphyton - Soil			
Total Mercury	CVAF	CVAF	CVAF
Methyl Mercury	CVAF	--	CVAF
Biomass*		--	--
Diatoms*	ASTM D 2974-87	--	--
Pigments	ASTM D 2974-87	--	--
Media: Sawgrass			
Total Mercury	CVAF	CVAF	CVAF
Media: Cattails			
Total Mercury	CVAF	CVAF	CVAF
Media: Mosquitofish			
Total Mercury	CVAF	CVAF	CVAF
Length	Measurement	--	--
Weight	Measurement	--	--
Sex	Visual	--	--
Gut Contents	Visual	--	--
Habitat Evaluation			
Food Habits Analysis*	Visual	--	--
Periphyton*	Experimental	--	--
Microphyton*	Experimental	Experimental	--
Aerial Photo Interpretation*		Experimental (UGA)	--

(1) Sum of TKN + NO_2/NO_3

* = Parameter added for the Phase II analysis

A5.2.3 Data Handling Records Documentation

The protocols and actions used in data reduction, verification, and validation are provided below and in Section D of this QAPP. Data reduction addresses data transformation operations such as converting raw data into reportable quantities and units, use of significant figures, recording of extreme values, blank corrections, etc. Data verification ensures the accuracy of data transcription and calculations, if necessary, by checking a set of computer calculations manually. Data validation ensures that QC criteria have been met.

A5.3 Data Reporting Package Format and Documentation Control

The format of data reporting packages will be consistent with the requirements and procedures used for data validation and data assessment described in Sections B, C, and D of this QAPP. Individual records that represent actions taken to achieve the objective of the data operation and the performance of specific QA functions are potential components of the final data reporting package.

A5.4 Data Reporting Package Archiving and Retrieval

Data reporting packages will be stored at the offices of FTN Associates, Ltd., in Little Rock, Arkansas, until the end of the data analysis and QA/QC checks. Upon completion of these activities, the data reporting packages will be transferred to the EPA Region IV offices in Athens, GA. The laboratories will keep all documentation related to the data reporting package and preparation and analysis of samples on file for a minimum of 5 years. If the laboratory desires to dispose of these records after 5 years they will first contact the EPA quality assurance officer. The EPA quality assurance officer may request that the documents be forwarded to EPA.

MEASUREMENT/DATA ACQUISITION

Bl SAMPLING PROCESS DESIGN (EXPERIMENTAL DESIGN)

B1.1 Purpose/Background

This section provides information to describe how and why the samples will be collected. The Phase II REMAP Statement of Work (Attachment 1) presents a detailed discussion of the sampling strategies including station location selection and sampling protocols (specific sampling protocols are also included in ESAT [1996] - Attachments 3). This was also fully documented in South Florida Ecosystem Assessment Final Technical Report - Phase I, EPA 904-R-98-002. Included in these documents are

- a schedule for project sampling activities,
- a rationale for the design (in terms of meeting DQOs),
- the sampling design assumptions, and
- the procedures for locating and selecting environmental samples.

B1.2 Classification of Measurements as Critical or Noncritical

Classification of measurements as being critical versus noncritical was performed at a Technical Team meeting held at EPA Region IV offices on October 15, 1998. A listing of critical and noncritical measurements is included as Tables B1 and B2. The basis of selection of critical vs. noncritical measurements was that measurements thought to have regulatory implications or usage for setting regulatory criteria/standards were considered “critical” measurements. All other measurements collected during the project are considered noncritical and useable for research purposes. These tables also designate Project Laboratory responsibilities, desired method detection limits (MDLs), and the anticipated sample numbers.

Table B1. REMAP Phase II critical parameters by cycle.

Parameter	Primary Lab	Primary QA/QC	Secondary QA/QC	Primary Lab MDL	Holding times	Site No. Per Cycle	Samp No.
SURFACE WATER							
DO	SESD	SESD-SOP		0.2 mg/L	in situ	129	129
pH	SESD	SESD-SOP		0.1 s.u.	in situ	129	129
Conductivity	SESD	SESD-SOP		1.0 uS	in situ	129	129
Turbidity	SESD	SESD		0.1 NTU	48 hrs	129	155
Total Phosphorus	FIU	SESD		0.6 ug/L	28 days (1)	129	155
Total Nitrogen	FIU	SESD		0.03 mg/L	14 days (1)	129	155
Total Organic Carbon	FIU	SESD		0.12 mg/L	28 days	129	155
Sulfate	SESD	SESD		0.05 mg/L	28 days	129	155
Total Mercury	FIU	Battelle	SESD	0.3 ng/L	28 days	129	187
Methyl Mercury	FIU	Battelle		0.02 ng/L	28 days	129	187
SOIL/SEDIMENT							
Total Mercury	FIU	SESD	Battelle	4.3 μg/kg	28 days	129	155
Methyl Mercury	FIU	Battelle		0.2 μg/kg	28 days	129	155
Total Phosphorus	FIU	SESD		0.06 mg/kg	28 days	129	155
Ash Free Dry Weight	FIU			0.02 mg/kg		129	155
Bulk Density	FIU			0.001 g/cc		129	155
MOSQUITO-FISH							
Total Mercury	FIU	SESD	Battelle	3.2 μg/kg	28 days	129	1043
Length	FIU			0.1 mm	14 days (1)	129	993
Weight	FIU			0.05 g	14 days (1)	129	993

THg in water = 129 sites, 16 field blanks, 13 duplicates, 16 equip. blanks, 13 splits = 187
Porewater (nutrients/anions) = 129 sites, 13 dups, 16 equip blanks, 13 splits = 171
THg in soil = 129 sites, 13 dups, 13 splits = 155
THg in fish = 129 sites @ 7 fish/site = 903, 90 dups, 50 stand. tissue = 1,043

(1) Holding time goals

Table B2. REMAP Phase II noncritical parameters by cycle.

Parameter	Primary Lab	Primary QA/QC	Secondary QA/QC	Primary Lab MDL	Holding Times	Site No. Per Cycle	Samp No.
SURFACE WATER							
(Eh) Redox Potential	SESD	SESD-SOP		1 m V	in-situ	129	129
Depth	SESD	SESD-SOP		1 cm	in-situ	129	129
Sulfide	SESD	SESD		0.01 mg/L	7 days (1)	129	155
(APA) Alkaline Phosphate	SESD	FIU		0.01 uM/h	24 hrs (1)	129	155
Temperature	SESD	SESD-SOP		0.15 C	in-situ	129	129
Chlorophyll a	FIU	FIU		0.1 ug/L	14 days (1)	30	33
Sulfate (filtered-0.8)*	SESD	SESD		0.5 mg/l	28 days	129	155
Filtered (0.8) Nutrients (NH_4,NO_2, NO_3, PO_4)*	FIU	SESD		NO3-0.7 ug/L NO2-0.3 ug/L NH4-0.8 ug/L SRP-0.6 ug/L	48 hrs (1)	129	155
SOIL/SEDIMENT							
Type	SESD				14 days (1)	129	129
Thickness	SESD			1 cm	14 days (1)	129	129
pH	SESD				in-situ	129	129
(Eh in situ) Redox Potential	SESD			1 m V	in-situ	129	129
(Eh lab) Redox Potential	SESD			1 m V	48 hrs (1)	129	129
Sulfate	SESD			0.05 ug/kg	28 days (1)	129	155
Mineral Content	FIU			3%	14 days (1)	129	155
(CH_4) Methane*	FIU				48 hrs (1)	129	155
(CO_2) Carbon Dioxide*	FIU				48 hrs (1)	129	155
(APA) Alkaline Phosphate	FIU					129	155
MOSQUITO-FISH							
Sex	FIU				14 days (1)	129	993
Food Habits Analysis	FIU					129	993
PORE WATER*							
Total Phosphorus*	FIU			0.6 ug/L	28 days (1)	129	171
Total Nitrogen*	FIU			0.3 mg/L	14 days (1)	129	155
Filtered (0.8) Nutrients (NH_4, NO_2, NO_3, PO_4)*	FIU			NO3-0.7 ug/L NO2-0.3 ug/L NH4-0.8 ug/L SRP-0.6 ug/L	48 hrs (1)	129	155
Anions (Br, Cl, Fl, NO_2, NO_3, SRP, SO_4)*	SESD			ion chrom.	14 days (1)	129	155
Sulfate*	SESD			0.05 mg/L	28 days (1)	129	171

Table B2. (Continued).

Parameter	Primary Lab	Primary QA/QC	Secondary QA/QC	Primary Lab MDL	Holding Times	Site No. Per Cycle	Samp No.
Sulfide*	SESD			0.01 mg/L	7 days (1)	129	171
PERIPHYTON - Utricularia							
Total Mercury	FIU	Battelle		4.3 ug/kg	28 days (1)	100	110
Methyl Mercury	FIU	Battelle		0.2 ug/kg	28 days (1)	100	110
Diatoms*	FIU				14 days (1)	30	33
Pigments	FIU				14 days (1)	30	33
PERIPHYTON - Soil							
Total Mercury	FIU	Battelle		4.3 ug/kg	28 days (1)	100	110
Methyl Mercury	FIU	Battelle		0.2 ug/kg	28 days (1)	100	110
Biomass*	SESD			1 g	14 days (1)	100	110
Diatoms*	FIU				14 days (1)	30	33
Pigments	FIU				14 days (1)	30	33
PERIPHYTON - Floating							
Total Mercury	FIU	Battelle		4.3 ug/kg	28 days (1)	100	110
Methyl Mercury	FIU	Battelle		0.2 ug/kg	28 days (1)	100	110
Biomass*	SESD			1 g	14 days (1)	100	110
Diatoms*	FIU				14 days (1)	30	33
Pigments	FIU				14 days (1)	30	33
SAWGRASS							
Total Mercury	FIU	Battelle		4.3 ug/ku	28 days (1)	65	72
Surface Area (% cover)	UGA					65	
CATTAILS							
Total Mercury	FIU	Battelle		4.3 ug/ku	28 days (1)	40	44
Surface Area (% cover)	UGA					40	
Habitat Evaluation							
Food Habits Analysis*	FIU					129	129
Periphyton*	FIU					129	129
Microphyton*	FIU					129	129
Aerial Photo Interpretation*	UGA					129	129

* = Parameter added for the Phase II analysis
** = minimum reportable quantities
THg in water = 129 sites, 16 field blanks, 13 duplicates, 16 equip. blanks, 13 splits = 187
Porewater (nutrients/anions) = 129 sites, 13 dups, 16 equip blanks, 13 splits = 171
THg in soil = 129 sites, 13 dups, 13 splits = 155
THg in fish = 129 sites @ 7 fish/site = 903, 90 dups, 50 stand. tissue = 1,043

(1) Holding time goals

B1.3 Validation of Any Nonstandard Methods

Nonstandard sampling/measurement methods will be validated by either comparisons with standard sampling/measurement methods or by review of the associated QC and QA samples generated versus QAPP requirements.

B2 SAMPLING METHODS REQUIREMENTS

B2.1 Sample Collection, Preparation, and Decontamination Procedures

Project sampling, preservation, preparation, and documentation protocols are included in the *ESAT SOP XXXII Standard Operating Procedures for Sampling Water Sediment and Biota in Expansive Wetlands* (ESAT, 1996), (Attachment 3). The project DQOs were considered in choosing these methods to ensure that (1) the sample accurately represents the portion of the environment to be characterized, (2) the sample is of sufficient volume to support the planned chemical analysis, and (3) the sample remains stable during shipping and handling. EPA and Southeast Environmental Research Program (SERP) personnel will provide technical support for sampling activities associated with the project.

B2.2 Sampling/Measurement System Response and Corrective Action Process

Corrective actions for field activities will be documented and submitted with data reports to FTN Associates for review during validation. When deviations from approved standard operating procedures (SOPs) occur or in situations when sample integrity is compromised or questionable, it is the responsibility of the staff member who identified the problem to bring it to the attention of the Laboratory Manager or Field Team Leader immediately for resolution. In the event of an instrument problem, it is the responsibility of the operator to attempt to correct the problem (e.g., recalibrate the instrument). If the problem persists or cannot be identified, the issue should be brought to the attention of the Laboratory Manager or Field Team Leader for resolution. Such issues will be documented by the Laboratory Manager or Field Team Leader and submitted to Science and Ecosystems Support Division Office of Quality Assurance (SESD OQA) and FTN Associates.

B2.3 Sampling Equipment, Preservation, and Holding Time Requirements

Sampling equipment, preservation, and holding time requirements for the study parameters are addressed in Tables B1 and B2 and the SESD, SERP, and Battelle QA Plans in Attachments 4 through 6.

B3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

Sample handling and shipping requirements are found in the ESAT, 1996 (Attachment 3). Chain-of-custody tracking/management for the project is performed using SESD's FORMS (field operations record management system) software.

These procedures insure that

- samples are collected, transferred, stored, and analyzed by authorized personnel;
- sample integrity is maintained during all phases of sample handling and analyses; and
- an accurate written record is maintained of sample handling and treatment from the time of its collection through laboratory procedures to disposal.

A sample is in custody if it is in actual physical possession or it is in a secured area that is restricted to authorized personnel. Custody for this project is primarily concerned with the tracking of sample collection, handling, and analysis.

An outline of the scope of sample custody starting from the planning of sample collection progressing through field sampling and sample analysis to sample disposal is included in Attachment 3. Samples will be numbered using the format X_1X_2-YYY-AAB, where X_1 is the sampling event (P = pilot; W = wet season; D = dry season) and X_2 is the replicate designation (A, B, or C). YYY is the sampling site designation and AA indicates sample media. The sample media codes are as follows:

SW - surface water
SG - sawgrass
CT - cattail
FS - fish
PM - periphyton, floating mat
PS - periphyton, soil mat (not floating)
PW - pore water
SD - soil, sediment
PU - periphyton, Utricularia
FC - Floc

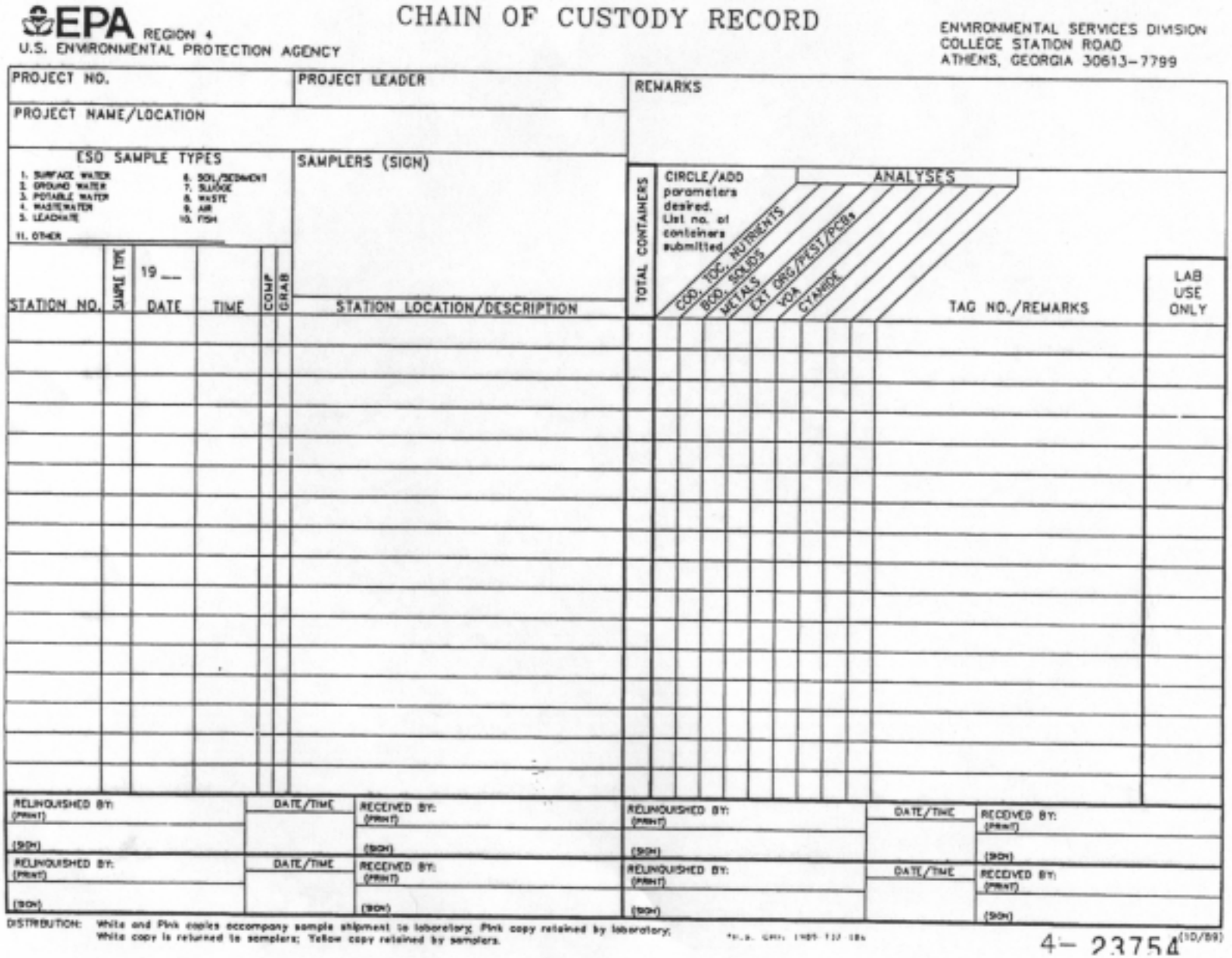
"B" is laboratory designation (B - Battelle, S - SESD, F - FIU). Examples of forms and labels that will be utilized during the project are included in Attachment 3. An example of the chain-of-custody forms that could be utilized is found in Figure B1.

B4 ANALYTICAL METHODS

B4.1 Purpose/Background

Specific monitoring methods and requirements to demonstrate compliance traditionally have been specified in the applicable regulations and/or permits. However, this approach is being replaced by the Performance-Based Measurement System (PBMS). PBMS is a process in which data quality needs, mandates, or limitations of a program or project are specified and serve as criterion for selecting appropriate methods. Under the PBMS framework, the performance of the method employed is emphasized rather than the specific technique or procedure used in the analysis. Equally stressed in this system is the requirement that the performance of the method be documented and that appropriate QA/QC procedures have been conducted to verify the performance. PBMS applies to physical, chemical, and biological techniques of analysis performed in the field as well as in the laboratory. PBMS does not apply to the method-defined parameters.

The listing of analyses anticipated during the project are included in Table A1 of this QAPP. Details of the analytical methods and equipment required for each of the methods are addressed in the SESD, SERP, and Battelle QA Plans in Attachments 4 through 6. These references include any subsampling and/or extraction/preparation methods, laboratory decontamination procedures and materials, and waste disposal requirements (if any).



EPA REGION 4
U.S. ENVIRONMENTAL PROTECTION AGENCY

CHAIN OF CUSTODY RECORD

ENVIRONMENTAL SERVICES DIVISION
COLLEGE STATION ROAD
ATHENS, GEORGIA 30613-7799

PROJECT NO.

PROJECT LEADER

REMARKS

PROJECT NAME/LOCATION

ESD SAMPLE TYPES

1. SURFACE WATER
2. GROUND WATER
3. POTABLE WATER
4. WASTEWATER
5. LEACHATE
6. SOIL/SEDIMENT
7. SLUDGE
8. WASTE
9. AIR
10. FISH
11. OTHER

SAMPLERS (SIGN)

STATION NO.	SAMPLE TYPE	19 __ DATE	TIME	COMP	GRAB	STATION LOCATION/DESCRIPTION	TOTAL CONTAINERS	ANALYSES: COD, TOC, NUTRIENTS	BOD, SOLIDS	METALS	EXT ORG/PEST/PCBs	VOA	CYANIDE	TAG NO./REMARKS	LAB USE ONLY

CIRCLE/ADD parameters desired. List no. of containers submitted.

RELINQUISHED BY: (PRINT) / (SIGN)	DATE/TIME	RECEIVED BY: (PRINT) / (SIGN)	RELINQUISHED BY: (PRINT) / (SIGN)	DATE/TIME	RECEIVED BY: (PRINT) / (SIGN)
RELINQUISHED BY: (PRINT) / (SIGN)	DATE/TIME	RECEIVED BY: (PRINT) / (SIGN)	RELINQUISHED BY: (PRINT) / (SIGN)	DATE/TIME	RECEIVED BY: (PRINT) / (SIGN)

DISTRIBUTION: White and Pink copies accompany sample shipment to laboratory; Pink copy retained by laboratory; White copy is returned to samplers; Yellow copy retained by samplers.

4- 23754 (10/88)

Figure B1. Sample Chain-of-Custody Form.

For noncritical analyses (Table B2), method performance study information will be developed to document performance of the method for the particular matrix.

B5 QUALITY CONTROL REQUIREMENTS

QC requirements are discussed as part of the validation section (Section D). Sampling process design, which identifies the planned field QC samples as well as procedures for QC sample preparation and handling will be finalized following the pilot study in early 1999. In general, measurement performance assessment follows the Phase II REMAP Statement of Work.

B6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS

Equipment testing, inspection and maintenance procedures are addressed in the SESD, SERP, and Battelle QA Plans in Attachments 4 through 6. The purpose of this testing is to ensure that all instruments and equipment are maintained in sound operating condition and are capable of operating at acceptable performance levels.

B7 INSTRUMENT CALIBRATION AND FREQUENCY

B7.1 Purpose/Background

Calibration here refers to checking instrument measurements against standards with known valid relationships to nationally recognized performance standards.

B7.2 Instrumentation Requiring Calibration

Field and laboratory equipment associated with this project that are calibrated are listed in the laboratory QA Plans, Attachments 4 through 6.

B7.3 Calibration Methods

All field and laboratory instruments are calibrated and checked for proper function prior to all analyses. Documentation of calibration for analytical instruments will be maintained by each laboratory and SESD for field instruments. Procedures for calibrating field equipment are included in ESAT, 1996 (Attachment 3). Calibration procedures for laboratory equipment are included in the individual analytical methods.

B7.4 Calibration Apparatus

This section is not applicable. All instruments are calibrated using standard materials.

B7.5 Calibration Standards

Primary standards are purchased from reliable scientific supply firms. The standards received by the Project Laboratories and Field Team will be inspected, dated, initialed, and stored in the appropriate storage area for that standard (desiccator, refrigerator, or freezer). Once opened, the standards will be dated and initialed again. The manufacturer's certificates for standards received will be kept on file at the Project Laboratories.

Primary standards are prepared by dissolving the source standard into the appropriate solvent. Secondary and working standards are prepared by diluting the primary standards in the appropriate solvent. Standard preparation methods are detailed in the individual laboratory SOPs. The date, concentration, chemical vendor lot number, and technician's initials for all standards made will be recorded and maintained by the Project Laboratories. Primary standards are produced at least quarterly, while working standards are produced daily.

B7.6 Calibration Frequency

Frequency of calibration of field instruments is provided in Attachment 3 (ESAT, 1996). Calibration frequency for laboratory instruments basically occurs prior to each use at least daily. After instrument calibration, an initial calibration verification sample is run at the start of each

analytical batch (a batch equals approximately 20 samples), and continuing calibration verification checks are run after approximately every 10 samples and/or the end of the batch.

B8 SUPPLIES AND CONSUMABLES

The purpose of this element is to document that a system for receiving, inspecting, and accepting supplies and consumables that may directly or indirectly affect the quality of the project or task is in place in the analytical laboratories. The on-site performance evaluation audit (Section D) will include inspection of laboratory protocols and documentation for proper receipt, inspection, cleaning, labeling, decontamination, etc. of supplies and consumables as necessary.

B9 DATA ACQUISITION REQUIREMENTS (NONDIRECT MEASUREMENTS)

This section is not applicable to this project.

B10 DATA MANAGEMENT

B10.1 Purpose/Background

This element is an overview of operations and analyses performed on raw ("as-collected") data to change their form of expression, location, quantity, or dimensionality. These operations include data recording, validation, transformation, transmittal, reduction, analysis, management, storage, and retrieval. Selected field measurements and analytical results and associated information will be transferred to electronic files. These files can be created in any spreadsheet program that is compatible with QuattroPro version 6 (or the spreadsheet and version that is currently standard at EPA).

B10.2 Data Recording and Reduction

Data recording shall be accomplished using established techniques. The calculations required to perform the reduction of data may be performed manually or with the aid of automated data processing systems. In either case, the SOPs for the testing/analysis of samples will specify the calculations and the mode for raw data processing. To reduce the potential of

errors in data transcription, the manual transfer of data will be minimized. All calculations performed manually will be checked for accuracy by someone other than the person performing the original calculation. Checking shall be documented, by signature and date in the raw data. Separate documentation is acceptable, provided traceable records are maintained. For automated data processing or recording, the accuracy of values will be verified through the use of standards or raw data inputs with known results.

B10.3 Data Transformation

Data transformation is discussed in the Phase II Scope of Work (Attachment 1) and relates to specific requirements of data users. Data analysis results will be provided in a comprehensive report that will be prepared following field and laboratory tasks.

B10.4 Data Transmittal

All collected data that will be used for analysis will be entered into electronic files in either Excel, QuattroPro, or dBase IV. Lists of the data that could be included in the electronic files for each analyst (EPA/SESD, EPA/ESAT, FIU/SERP, and Battelle) are included as Tables B3 through B6 (the tables may not reflect final decisions regarding analyses to be performed). These electronic files will be sent to FTN Associates on diskettes or via e-mail. If necessary, the data files may be compressed using PKZIP or WINZIP. These provided data will be imported into the statistics and graphing package SYSTAT 7.0 (SPSS Inc., 1997, Chicago, IL). Data for analyses will be extracted from these files and combined as needed.

B10.5 Data Analysis

Data analyses will include using analytical results in the EPA ORD NERL-Athens mercury screening model and the BASS model. Summary statistics will be calculated and compared for a number of regional groupings. Analytical results will also be used to create

spatial isoconcentration maps. Additional statistical analyses of analytical results will likely include cumulative distributions, ANOVAs, regressions, and trend analyses. These analyses will use the data for the entire study area grouped together, or split by geographic regions. Most of these statistical analyses will be performed using SYSTAT. QuattroPro will be used to develop the cumulative distributions.

B10.6 Data Storage and Retrieval

Data received by FTN from the field data collectors and the laboratories will be imported into SYSTAT files that will not be modified. These files will serve as storage for these data. Any data files needed for data analyses will be created using data extracted from these storage files. For the duration of this project, these files will be stored at the office of FTN Associates in Little Rock, AR. Upon completion of this phase of the project, these files will be transferred to the EPA Region IV offices in Athens, GA in a spreadsheet format (Excel, QuattroPro, or dBase IV).

Table . . Analytical data from A S S lab.

		Surface ater	ore ater	Soil	Floc	eriphyton	Cattail	Sawgrass	Fish
Sample I	Field sample name								
eriphyton Type	mat or epiphytic								
Total Hg	collection date								
	lab batch id for total Hg analysis								
	sample result for total Hg								
	units of total Hg								
	dilution								
	analysis date								
	analysis time								
	duplicate and or replicate total Hg sample results								
	total Hg spi e and or standard percent reco erys								
	total Hg sample minimum detection le el								
T C	collection date								
	lab batch id for T C analysis								
	sample result for T C								
	units of T C								
	analysis date								
	analysis time								
	duplicate and or replicate T C sample results								
	T C spi e and or standard percent reco erys								
	T C sample minimum detection le el								
Total	collection date								
	lab batch id for Total analysis								
	sample result for Total								
	units of Total								
	dilution								
	analysis date								
	analysis time								
	duplicate and or replicate Total sample results								
	Total spi e and or standard percent reco erys								
	Total sample minimum detection le el								
Filtered Ammonia	collection date								
	lab batch id for filtered NH4 analysis								
	sample result for filtered NH4								
	units of filtered NH4								
	dilution								
	analysis date								
	analysis time								

Table . . Continued.

	duplicate and or replicate filtered NH4 sample results
	filtered NH4 spi e and or standard percent reco erys
	filtered NH4 sample minimum detection le el
Filtered	collection date
Nitrite	lab batch id for filtered N analysis
	sample result for filtered N
	units of filtered N
	dilution
	analysis date
	analysis time
	duplicate and or replicate filtered N sample results
	filtered N spi e and or standard percent reco erys
	filtered N sample minimum detection le el
Filtered	collection date
Nitrate	lab batch id for filtered N analysis
	sample result for filtered N
	units of filtered N
	dilution
	analysis date
	analysis time
	duplicate and or replicate filtered N sample results
	filtered N spi e and or standard percent reco erys
	filtered N sample minimum detection le el
Filtered	collection date
hosphate	lab batch id for filtered 4 analysis
(S)	sample result for filtered 4
	units of filtered 4
	dilution
	analysis date
	analysis time
	duplicate and or replicate filtered 4 sample results
	filtered 4 spi e and or standard percent reco erys
	filtered 4 sample minimum detection le el
Filtered	collection date
Sulfate	lab batch id for filtered S 4 analysis
	sample result for filtered S 4
	units of filtered S 4
	dilution
	analysis date
	analysis time

Table . . Continued.

	duplicate and or replicate filtered S 4 sample results
	filtered S 4 spi e and or standard percent reco erys
	filtered S 4 sample minimum detection le el
T N	collection date
	lab batch id for T N analysis
	sample result for T N
	units of T N
	dilution
	analysis date
	analysis time
	duplicate and or replicate T N sample results
	T N spi e and or standard percent reco erys
	T N sample minimum detection le el
Unfiltered Nitrite	collection date
	lab batch id for unfiltered N analysis
	sample result for unfiltered N
	units of unfiltered N
	dilution
	analysis date
	analysis time
	duplicate and or replicate unfiltered N sample results
	unfiltered N spi e and or standard percent reco erys
	unfiltered N sample minimum detection le el
Unfiltered Nitrate	collection date
	lab batch id for unfiltered N analysis
	sample result for unfiltered N
	units of unfiltered N
	dilution
	analysis date
	analysis time
	duplicate and or replicate unfiltered N sample results
	unfiltered N spi e and or standard percent reco erys
	unfiltered N sample minimum detection le el
Sulfate anion	collection date
	lab batch id for S 4 anion analysis
	sample result for S 4 anion
	units of S 4 anion
	dilution
	analysis date
	analysis time

Table . . Continued.

	duplicate and or replicate S 4 anion sample results
	S 4 anion spi e and or standard percent reco erys
	S 4 anion sample minimum detection le el
Chloride anion	collection date
	lab batch id for Cl anion analysis
	sample result for Cl anion
	units of Cl anion
	dilution
	analysis date
	analysis time
	duplicate and or replicate Cl anion sample results
	Cl anion spi e and or standard percent reco erys
	Cl anion sample minimum detection le el
romide anion	collection date
	lab batch id for r anion analysis
	sample result for r anion
	units of r anion
	dilution
	analysis date
	analysis time
	duplicate and or replicate r anion sample results
	r anion spi e and or standard percent reco erys
	r anion sample minimum detection le el
Fluoride anion	collection date
	lab batch id for F anion analysis
	sample result for F anion
	units of F anion
	dilution
	analysis date
	analysis time
	duplicate and or replicate F anion sample results
	F anion spi e and or standard percent reco erys
	F anion sample minimum detection le el
Unfiltered ortho-	collection date
	lab batch id for unfiltered ortho- analysis
	sample result for unfiltered ortho-
	units of unfiltered ortho-
	dilution
	analysis date
	analysis time

Table . . Continued.

duplicate and or replicate unfiltered ortho-	sample results
unfiltered ortho-	spi e and or standard percent reco erys
unfiltered ortho-	sample minimum detection le el

Table B.4. Analytical data from EPA/ESAT lab.

		Surface Water	Pore Water	Soil	Floc	Periphyton	Cattail	Sawgrass	Fish
Sample ID	Field sample name	X	X						
Periphyton Type	mat or epiphytic								
Hydrogen	collection date	X	X						
Sulfide	lab batch id for H2S analysis	X	X						
	sample result for H2S	X	X						
	units of H2S	X	X						
	% dilution	X	X						
	analysis date	X	X						
	analysis time	X	X						
	duplicate and/or replicate H2S sample results	X	X						
	H2S spike and/or standard percent recoverys	X	X						
	H2S sample minimum detection level	X	X						
Turbidity	collection date	X							
	lab batch id for turbidity analysis	X							
	sample result for turbidity	X							
	units of turbidity	X							
	% dilution	X							
	analysis date	X							
	analysis time	X							
	duplicate and/or replicate turbidity sample results	X							
	turbidity spike and/or standard percent recoverys	X							
	turbidity sample minimum detection level	X							
Alkaline	collection date	X							
Phosphatase	lab batch id for alkaline phosphatase analysis	X							
	sample result for alkaline phosphatase	X							
	units of alkaline phosphatase	X							
	% dilution	X							
	analysis date	X							
	analysis time	X							
	duplicate and/or replicate alkaline phosphatase sample results	X							
	alkaline phosphatase spike and/or standard percent recoverys	X							
	alkaline phosphatase sample minimum detection level	X							

Table .5. Analytical data from FIU S lab.

		Surface ater	ore ater	Soil	Floc	eriphyton	Cattail	Sawgrass	Fish
Sample I	Field sample name								
eriphyton Type	mat or epiphytic								
Total Hg	collection date								
	lab batch id for total Hg analysis								
	sample result for total Hg								
	units of total Hg								
	dilution								
	analysis date								
	analysis time								
	duplicate and or replicate total Hg sample results								
	total Hg spi e and or standard percent reco erys								
	total Hg sample minimum detection le el								
ethyl Hg	collection date								
	lab batch id for methyl Hg analysis								
	sample result for methyl Hg								
	units of methyl Hg								
	dilution								
	analysis date								
	analysis time								
	duplicate and or replicate methyl Hg sample results								
	methyl Hg spi e and or standard percent reco erys								
	methyl Hg sample minimum detection le el								
thyl Hg	collection date								
	lab batch id for ethyl Hg analysis								
	sample result for ethyl Hg								
	units of ethyl Hg								
	dilution								
	analysis date								
	analysis time								
	duplicate and or replicate ethyl Hg sample results								
	ethyl Hg spi e and or standard percent reco erys								
	ethyl Hg sample minimum detection le el								
T C	collection date								
	lab batch id for T C analysis								
	sample result for T C								
	units of T C								
	analysis date								
	analysis time								

Table .5. (Continued).

	duplicate and or replicate T C sample results
	T C spi e and or standard percent reco erys
	T C sample minimum detection le el
Total	collection date
	lab batch id for Total analysis
	sample result for Total
	units of Total
	dilution
	analysis date
	analysis time
	duplicate and or replicate Total sample results
	Total spi e and or standard percent reco erys
	Total sample minimum detection le el
Total N	collection date
	lab batch id for total N analysis
	sample result for total N
	units of total N
	analysis date
	analysis time
	duplicate and or replicate total N sample results
	total N spi e and or standard percent reco erys
	total N sample minimum detection le el
Filtered	collection date
Ammonia	lab batch id for filtered NH4 analysis
	sample result for filtered NH4
	units of filtered NH4
	dilution
	analysis date
	analysis time
	duplicate and or replicate filtered NH4 sample results
	filtered NH4 spi e and or standard percent reco erys
	filtered NH4 sample minimum detection le el
Filtered	collection date
Nitrite	lab batch id for filtered N analysis
	sample result for filtered N
	units of filtered N
	dilution
	analysis date
	analysis time
	duplicate and or replicate filtered N sample results

Table .5. (Continued).

	filtered N spi e and or standard percent reco erys
	filtered N sample minimum detection le el
Filtered Nitrate	collection date
	lab batch id for filtered N analysis
	sample result for filtered N
	units of filtered N
	dilution
	analysis date
	analysis time
	duplicate and or replicate filtered N sample results
	filtered N spi e and or standard percent reco erys
	filtered N sample minimum detection le el
Filtered hosphate (S)	collection date
	lab batch id for filtered 4 analysis
	sample result for filtered 4
	units of filtered 4
	dilution
	analysis date
	analysis time
	duplicate and or replicate filtered 4 sample results
	filtered 4 spi e and or standard percent reco erys
	filtered 4 sample minimum detection le el
Unfiltered Nitrite	collection date
	lab batch id for unfiltered N analysis
	sample result for unfiltered N
	units of unfiltered N
	dilution
	analysis date
	analysis time
	duplicate and or replicate unfiltered N sample results
	unfiltered N spi e and or standard percent reco erys
	unfiltered N sample minimum detection le el
Unfiltered Nitrate	collection date
	lab batch id for unfiltered N analysis
	sample result for unfiltered N
	units of unfiltered N
	dilution
	analysis date
	analysis time
	duplicate and or replicate unfiltered N sample results

Table .5. (Continued).

	unfiltered N spi e and or standard percent reco erys
	unfiltered N sample minimum detection le el
Unfiltered rtho-	collection date
	lab batch id for unfiltered ortho- analysis
	sample result for unfiltered ortho-
	units of unfiltered ortho-
	dilution
	analysis date
	analysis time
	duplicate and or replicate unfiltered ortho- sample results
	unfiltered ortho- spi e and or standard percent reco erys
	unfiltered ortho- sample minimum detection le el
Sulfate anion	collection date
	lab batch id for S 4 anion analysis
	sample result for S 4 anion
	units of S 4 anion
	dilution
	analysis date
	analysis time
	duplicate and or replicate S 4 anion sample results
	S 4 anion spi e and or standard percent reco erys
	S 4 anion sample minimum detection le el
Chloride anion	collection date
	lab batch id for Cl anion analysis
	sample result for Cl anion
	units of Cl anion
	dilution
	analysis date
	analysis time
	duplicate and or replicate Cl anion sample results
	Cl anion spi e and or standard percent reco erys
	Cl anion sample minimum detection le el
romide anion	collection date
	lab batch id for r anion analysis
	sample result for r anion
	units of r anion
	dilution
	analysis date
	analysis time
	duplicate and or replicate r anion sample results

Table .5. (Continued).

	r anion spi e and or standard percent reco erys
	r anion sample minimum detection le el
Fluoride anion	collection date
	lab batch id for F anion analysis
	sample result for F anion
	units of F anion
	dilution
	analysis date
	analysis time
	duplicate and or replicate F anion sample results
	F anion spi e and or standard percent reco erys
	F anion sample minimum detection le el
Ash Free	collection date
ry eight	lab batch id for AF analysis
	sample result for AF
	units of AF
	analysis date
	analysis time
	duplicate and or replicate AF sample results
ul ensity	collection date
	lab batch id for bul density analysis
	sample result for bul density
	units of bul density
	analysis date
	analysis time
	duplicate and or replicate bul density sample results
ineral	collection date
Content	sample result for mineral content
	units of mineral content
	analysis date
	analysis time
	duplicate and or replicate mineral content sample results
ethane	collection date
	lab batch id for CH4 analysis
	sample result for CH4
	units of CH4
	analysis date
	analysis time
	duplicate and or replicate CH4 sample results
	CH4 sample minimum detection le el

Table .5. (Continued).

Carbon io ide	collection date
	lab batch id for C analysis
	sample result for C
	units of C
	analysis date
	analysis time
	duplicate and or replicate C sample results
	C sample minimum detection le el
iatom Composition	collection date
	lab batch id for diatoms analysis
	sample result for diatoms
	units of diatoms
	analysis date
	analysis time
	duplicate and or replicate diatoms sample results
igment	collection date
	lab batch id for pigment analysis
	sample result for pigment
	units of pigment
	analysis date
	analysis time
	duplicate and or replicate pigment sample results
Al aline hosphatase	collection date
	lab batch id for al aline phosphatase analysis
	sample result for al aline phosphatase
	units of al aline phosphatase
	dilution
	analysis date
	analysis time
	duplicate and or replicate al aline phosphatase sample results
	al aline phosphatase spi e and or standard percent reco erys
	al aline phosphatase sample minimum detection le el
olume eight atio	collection date
	lab batch id for olume weight ratio
	sample result for olume weight ratio
	units of olume weight ratio
	analysis date
	analysis time
	duplicate and or replicate olume weight ratio results

Table B.6. Analytical data from Battelle lab.

		Surface Water	Pore Water	Soil	Floc	Periphyton	Cattail	Sawgrass	Fish
Sample ID	Field sample name	X		X	X	X			
Periphyton Type	mat or epiphytic					X			
Total Hg	collection date	X							
	lab batch id for total Hg analysis	X							
	sample result for total Hg	X							
	units of total Hg	X							
	% dilution	X							
	analysis date	X							
	analysis time	X							
	duplicate and/or replicate total Hg sample results	X							
	total Hg spike and/or standard percent recoverys	X							
	total Hg sample minimum detection level	X							
Methyl Hg	collection date	X		X	X	X			
	lab batch id for methyl Hg analysis	X		X	X	X			
	sample result for methyl Hg	X		X	X	X			
	units of methyl Hg	X		X	X	X			
	% dilution	X		X	X	X			
	analysis date	X		X	X	X			
	analysis time	X		X	X	X			
	duplicate and/or replicate methyl Hg sample results	X		X	X	X			
	methyl Hg spike and/or standard percent recoverys	X		X	X	X			
	methyl Hg sample minimum detection level	X		X	X	X			

ASSESSMENT/OVERSIGHT

C1 ASSESSMENTS AND RESPONSE ACTIONS

C1.l Purpose/Background

This element of the QAPP describes the internal and external checks necessary to ensure that

- all elements of the QAPP are correctly implemented as prescribed;
- the quality of the data generated by implementation of the QAPP is adequate; and
- corrective actions, when needed, are implemented in a timely manner and their effectiveness is confirmed.

External assessments that are planned are described in the QAPP although the most important part of this element is documenting all planned internal assessments. Generally, internal assessments are initiated or performed by the laboratory QA Officers.

C1.2 Assessment of Project Activities

The following assessments are planned as part of the overall QA/QC associated with the project.

A) *Technical Systems Audit (TSA).* A TSA is a onsite qualitative audit, where facilities, equipment, personnel, training, procedures, and record keeping are examined for conformance to the QAPP. One TSA is planned during the project to review field sampling and analytical activities and the FIU contract laboratories. The TSA will be utilized with broad coverage to evaluate the management structure, policy, practices, or procedures. The TSA will be conducted during the second seasonal sampling.

B) *Performance Evaluation (PE).* Use of “blind” PE samples will indicate accuracy and precision of the measurement system. The constituents to be measured will include all “critical” parameters for aqueous samples. PE samples will be utilized

during the seasonal sampling and analyses. Historical PE data from the analytical laboratories will also be evaluated. Successful accomplishment of PEs will be based on criteria presented in Section D.

C) *Data Quality Assessment (DQA). A* DQA will be performed to ensure data collected during the project meet the assumptions that the DQOs and data collection design were developed under and whether the total error in the data is tolerable.

A combination of SESD OQA and FTN personnel will perform the TSAs during the project. Results of audits and other assessments that reveal findings of practices or procedures that do not conform to the written QAPP will be reported to the Project Technical Director in writing within 1 week of the audit. The written summary will provide recommendations for corrective actions. Upon approval of the corrective actions by the Project Technical Director, the field sampling group or analytical laboratory that is the subject of the recommendations will be notified of the finding and the required corrective actions. Written documentation of implementation of the corrective actions will be required to be returned to Mr. Mike Birch, SESD OQA and Dr. Kent Thornton, FTN Associates.

C2 REPORTS TO MANAGEMENT

Effective communication between all personnel is an integral part of a quality system. Written reports provide a structure for apprising management of the project schedule, the deviations from approved QA and test plans, the impact of these deviations on data quality, and the potential uncertainties in decisions based on the data. Verbal communication on deviations from QA plans should be noted in summary form.

Management reports are anticipated on a routine frequency of once per week during sampling and analytical activities associated with the two seasonal sampling events. The anticipated benefits of these reports include alerting the management of data quality problems, proposing viable solutions, and procuring additional resources. If program assessment (including the evaluation of the technical systems, the measurement of performance, and the assessment of data) is not conducted on a continual basis, the integrity of the data generated in the program may

not meet the quality requirements. These audit reports, submitted in a timely manner, will provide an opportunity to implement corrective actions when most appropriate.

The reports to management will originate from three groups: (1) the field sampling/activities group, (2) the analytical laboratories, and (3) the data validation/management group. Reports will be directed to Dr. Jerry Stober, the Project Technical Director.

Contents of the reports will include (1) status of the project each group is associated with, (2) anticipated activities for the next period, (3) problems or delays encountered and associated resolutions, (4) additional needs, and (5) general comments.

DATA VALIDATION

This section presents validation activities that occur before, during, and after the data collection phases of the project. QA/QC sampling, analytical, and validation requirements described in this QAPP will generally apply to both the pilot and two seasonal sampling periods during the Phase II assessment. However, various nonstandard or developmental sampling protocols and analytical methods/protocols utilized during the pilot sampling in early 1999 may not be continued in subsequent seasonal sampling phases. These pilot study protocols will be closely evaluated based on a number of criteria including problems encountered, volume and applicability of data collected compared to the sampling effort, cost of data collection, data needs to address sampling design parameters, etc. Based on this evaluation, sampling parameters and protocols, as well as analytical methods (and to some degree, validation requirements) will be refined as necessary and included in the wet and dry season sampling efforts. This QAPP will be revised following the pilot study sampling and analysis. It is anticipated, however, that many data produced from the pilot study will meet validation requirements and will be available for various uses including design of subsequent phases, future analyses and system characterization.

The SERP of Florida International University will be the primary analytical laboratory during the project. The Florida Department of Environmental Protection (DEP) has reviewed and approved the SERP QA Plans and methods manuals for analytical services and mercury laboratories. The EPA Region IV SESD analytical laboratory and Battelle's Marine Science Laboratory in Sequim, WA are also providing extensive analytical services for the Phase II Assessment. Laboratory-specific listing of analyses for the project are included as Tables B1 and B2.

Section D1 of this QAPP provides criteria that will be used to review and "validate" (i.e., accept, reject, or qualify) data produced during this project by the contract laboratories. The process to be used during validation is discussed in Section D2. Sections D2 and D3 describe how limitations on the use of the data will be reported to the data users.

D1 VALIDATION CRITERIA

The USEPA Region IV SESD OQA QA/QC attachment to the September, 1998 Statement of Work (in Attachment 1) was followed to prepare this section of the QAPP. SERP's current DEP contract for analytical and sampling support services (Contract No. SP419) was also followed during development of the QAPP and the specific validation criteria as well as Battelle's Quality Assurance Management Plan (Attachment 5). Actual validation of the data associated with the project will be achieved with development and review (verification) of documentation to show that the required QA/QC procedures are followed. As stated in the QA/QC attachment to the Work Plan, the QA/QC documentation developed during the project will allow evaluation of the following indicators of data quality:

- Integrity and stability of the samples,
- Instrument performance during analysis,
- Sample contamination,
- Identification and quantitation of analytes,
- Analytical precision, and
- Analytical accuracy.

The following sections provide criteria that must be met to evaluate and validate data generated during the project. Specific exceptions (i.e., certain sample and analytical methods) to these validation criteria are discussed in subsections below. In addition, certain corrective actions to resolve QC problems are presented in these following sections.

General QA/QC requirements for the project include the following:

- Field sampling activities will follow the Phase II REMAP Statement of Work (Attachment 1) and protocols described in ESAT (1996) (Attachment 3).

- Analytical laboratories involved with the project will establish and implement comprehensive QA programs to define the reliability of the analytical results produced for this project. The QA programs will be documented in a written QA plans that will be submitted, along with this QAPP, for approval by SESD OQA.

- Analytical laboratories utilized will comply with the EPA approved laboratory QA plans submitted as required during this project. Any proposed modifications to the

laboratory QA plans must be reviewed and approved by EPA prior to implementing the modification. If there is a discrepancy between this QAPP and Attachments 4-6, this QAPP will supercede individual laboratory QA Plans.

- Sample containers, blank water, and equipment - Field and laboratory personnel will prepare and use containers and equipment that do not contribute contamination to samples detectable as critical constituents. Field equipment blanks will be utilized to verify this requirement by comparing analyte concentrations in the wash water before and after it contacts the equipment. Blank requirements specifically apply to surface water (media) samples only at a level of 1 blank prepared (field or equipment) per batch or for approximately every 20 samples collected.

- Sample custody and tracking - Field and laboratory custody will utilize SESD's "FORMS" software and will follow SOPs in ESAT (1996) (Attachment 3). Chain-of-custody will be maintained throughout sampling, transport, and analysis.

- Documentation - Contract laboratories will follow document control procedures to assure all documents including but not limited to logbooks, chain-of-custody records, sample work sheets, sample run logs, instrument raw data, bench sheets, sample preparation records, and data deliverable reports are prepared.

- Sample Data Reports - Contract laboratories will complete and submit data summaries (spreadsheets) hard copy and electronic copy. Laboratory MDLs for each parameter are required with these reports, calculated according to 40 CFR Part 136, Appendix B, or other approved method.

- QC Data Reports - Along with sample results from each batch of environmental samples, the contract laboratories will submit results of all field generated QC samples including equipment blanks, field duplicates (colocated samples), and field blanks. Contract laboratories will compile and submit QC data for these sample types. The laboratory will also compile and submit results of laboratory QC samples for replicates and spikes including the parameter and matrix. Relative percent difference (RPD) for duplicates or relative standard deviation (RSD) will be required for precision evaluation utilizing laboratory split samples. Percent recovery (%R) or percent difference (PD) for standard reference materials (SRMs) will be required for accuracy evaluation.

- Data entry - The analytical laboratories or FTN Associates will enter data following standard procedures for manual entry. Accuracy of transcription for the data will be checked by\ another person. Data plots and descriptive statistics will be used to screen accuracy of data entry where historical data exist.

Specific QA/QC criteria for validation and verification of data associated with the project include the following, these analytical data will be available for inspection as necessary.

- Documentation packages for data submittals.
- Narrative description of the data report packages (including range of samples analyzed, analytical methods, sample holding times summary, descriptions of problems encountered, and explanation for any QA/QC samples that do fall outside project acceptance criteria - see Attachment 2 for Laboratory Acceptance criteria for project parameters); applicable comments relating to sample integrity or data quality.
- Chain-of-custody documentation and summary (including completed forms that match all data submitted with package).
- Summary of results (including data tables and statement regarding achievement of MDLs specified in the project statement of work–Attachment 1).
- Field and/or laboratory data for approximately 10 percent of analyses of "critical" parameters for the batch (Critical parameters for each media are listed in Table B1). This includes
    - Sample log in documentation.
    - Manual calculations including raw data, formulae utilized, any conversion constants, and an example calculation. Verification of one of each type of calculation will be necessary.
    - Instrument printouts, bench sheets, digestion worksheets, sample preparation logs, and other sample analysis and preparation documentation/calculations.
    - Sample date and times of collection, digestion, and analysis along with sample volumes and digestion volume (as appropriate).
- QC Sample Documentation
    - Instrument calibration documentation - An instrument calibration curve will be prepared at minimum at the beginning of each day of analysis utilizing at least three standards plus one blank (four standards and one blank for methylmercury).

- Laboratory Method Blanks - A laboratory method blank will be analyzed at the start of each analytical batch.

- Internal calibration data (initial and CCV–continuing calibration verification data) - Documentation of initial calibration and mid-level CCV at the first of each batch and one per 10 samples analyzed. CCV will be prepared from standard reference material from source(s) which attest to the concentration of the standard source.

• QC Sample Data - for each batch of 20 samples or fewer, the analytical laboratory will provide data for the following QC samples:

  - One laboratory method blank that will be included with every step in the analytical procedure.

  - One laboratory replicate.

  - One matrix spike - For water, the matrix spike will be designed to result in a sample analysis concentration that does not exceed 2 times the PQL or 2 times the expected sample concentration, whichever is larger. For solids, the matrix spike will be designed to result in a sample analysis concentration that does not exceed 2 times the unspiked sample.

  - One SRM for the matrix in an appropriate concentration that will not exceed the concentration of the most concentrated standard.

Data will be available for inspection.

D2 VALIDATION METHODS

Validation methods to assess the following general QA/QC requirements for the project are presented in this section. Any nonconformance issues for this section will result in implementation of corrective actions to address the issue, documentation of the corrective action, and a preparation of narrative description to describe potential impacts to data quality due to the problem.

• Conformance of field sampling activities to the Phase II REMAP (September 1998) Statement of Work (Attachment 1) and sample/data management protocols described in SERP's Comprehensive QA Plans (Attachment 6) verified by

conducting on-site field and laboratory PE audits during either the wet or dry season sampling period.

- QA program and written QA plan preparation and acceptance - validated during pre-sampling review by EPA Region IV SESD OQA.

- Compliance with the EPA approved laboratory QA plans will be validated by (1) performing an on-site laboratory audit during either the wet or dry season sampling/analysis activities and (2) on-going review of data deliverable packages submitted with analytical results packages. Verification of supporting functions such as sample custody, reagent and standards preparation, sample preparation, equipment and container cleaning, calibration, etc. will also be performed via on-site PE audit of the analytical laboratory.

- Appropriateness of sample containers, blank water, and equipment will be validated by analysis of blanks (field and equipment) during the project as well as review of laboratory operations during a PE audit described above. Successful performance for blank usage and analysis is defined as no differences ($\leq$3 times the MDL) in analytical results between blanks and source water utilized for preparation of blanks.

- Sample custody and tracking conformance will be validated by review of documentation submitted with data report packages as well as by direct observance during a PE audit described above. Conformance to this requirement will be met with custody documented for all samples. Non-conformance may result in limiting the usability of the data.

- Preparation and storage of appropriate project documentation will be validated by means of reviewing data deliverable report packages and on-site PE audits.

- Completeness and accuracy of reports will be validated by reviewing and verifying data entry QA/QC results and during data analysis and outlier identification.

Specific QA/QC targets and validation methods of data associated with the project include:

- Documentation packages for data submittals - validation by verifying necessary components included with each package submitted to FTN Associates. Recalculation of approximately 10% of the test results for critical parameters for each analytical batch, parameter group, and matrix as applicable.

- QC Sample Documentation

  - Initial instrument calibration documentation - A correlation coefficient of 0.995 or better using least squares fit unless the approved calibration method permits verification of the initial calibration using fewer standards. Documentation of low and mid-range CCV checks at the first of and during analyses will be required as well as one SRM. The laboratories will maintain this documentation.

  - Laboratory Method Blanks - If the difference between results from the laboratory method blank and the source water used to prepare the blank exceeds the action limit of >3 times the MDL, documentation of corrective actions taken to reduce it to below the action limit prior to any analysis. Documentation of such corrective actions will be prepared and maintained at each Project Laboratory.

  - Internal calibration data (continuing calibration verification data) - If results differ by >15% from the known value or the initial check, whichever is appropriate, the laboratory will take corrective action(s) to reduce the difference to below 15% and document the problem and action(s) taken. Any samples analyzed after the last passing CCV and prior to the failing CCV will be reanalyzed after corrective action(s) are taken and a passing CCV is analyzed.

- QC Samples

  - Laboratory Method Blank - Difference between blank results and source water must be $\leq$3 times the MDL. Action to determine the cause of the contaminant, correct the problem, and document such actions must be taken and documented when results are >3 times the MDL.

  - Equipment (field) Blank - Differences between blank results and the source water $\geq$3 times the MDL will result in the samples collected with the field equipment used to produce the blank on the same day of sampling to be qualified to alert data users to potential cleaning or sampling problems.

  - Replicated Samples - Where replicates producing two samples from one are performed by the lab, precision (RPD) within limits presented in Table A1A of the project DQO document (Attachment 2), or within 20% of laboratory replicate samples for parameters not named in Table A1A. These criteria relate to analytes >5 times the MDL.

- Replicated Samples - Where triplicates or more are performed by the lab, RSD (coefficient of variation) must fall within limits presented in Table A1A of the project DQO Document (Attachment 2) or within 20% for parameters not named in Table A1A. These criteria apply to analytes >5 times the MDL.

- Replicated Samples - Where samples are "split" in the field, RPD should be $\leq$20%. Field split samples with RPDs >20% will be qualified to alert data users to potential sampling problems. These criteria apply to analytes >5 times the MDL.

- Colocated Samples - If RPDs are greater than precision guidelines in Table A1A, these data will be qualified only to alert data users to potential sampling variability. These guidelines were based on Phase I colocated sample results. These criteria apply to analytes >5 times the MDL.

- Laboratory Standards and CCV - Percent difference from initial calibration check should be $\leq$15%.

- Matrix Spikes - Percent recovery (%R) for matrix spikes should fall within the range of 75 to 125% of the spiked concentration for all media. However, matrix spike recovery outside this range will not by itself result in a "reject" qualifier. Rather, the data will be qualified as having a matrix effect to alert data users.

- SRMs, Blank Spikes, PE Samples - Accuracy as %R and precision of replicates as RPD or RSD for those samples must meet Project DQO requirements (Table A1A, Attachment 2).

All reported data will be validated according to Section D of this QAPP. When reporting data to EPA, the following data qualifiers are anticipated for use with this project:

"U" Analyte not detected at or above the MDL.

"J" Concentration reported should be considered an estimate. The data are acceptable for use as determined by specific data users but certain QC criteria were not met; e.g.,

- data were above or below appropriate linear calibration range,
- holding times were exceeded,
- certain QC documentation was not prepared as required or

- the analyte was detected below the MDL

"A" Analyte was analyzed as a replicate and the value reprinted is the mean of the replicates.

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range. Data are usable.

"D" Analyte concentration reported as the result of a secondary dilution. Discrepancies between two runs may be due to dilution errors. Data are usable provided other criteria are met.

"B" Analyte concentration in the associated blank was >3 times the MDL.

D3 RECONCILIATION WITH DATA QUALITY OBJECTIVES

The purpose of element D3 is to outline and specify, if possible, the acceptable methods for evaluating the results obtained from the project. This element includes scientific and statistical evaluations of data to determine if the data are of the right type, quantity, and quality to support their intended use.

D3.1 Reconciling Results with DQOs

There will be two phases of reconciliation of the results with the DQOs. In Phase I, statistical analyses will be performed to compare computed estimates (recovery, precision, PE sample variance, etc.) with DQOs specified in this QAPP. This information will be provided to the Project Manager and QA Officer. In Phase II, the user will determine if the data results meet their needs and objectives. Phase II supersedes any and all Phase I QA/QC analyses and results because the purpose of any QA/QC program is to provide information of known quality so that the user can determine if the data meets their needs and objectives.

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Attachment 1

Phase II REMAP Statement of Work

Investigation of Mercury Contamination in the Everglades Ecosystem
and
Everglades Ecosystem Assessment (Phase II REMAP)

Statement of Work
for
USEPA/USNPS IAG
September 1998

Introduction

The interim assessment (Stober et al. 1996) and the results of the final technical assessment for Phase I (USEPA 1998) indicate the importance of hydropattern, nutrient, habitat, vegetation and food web information for ecosystem management and restoration efforts. Continued monitoring of water, soil/sediment, periphyton, and fish is critical both for better understanding of mercury cycling in the ecosystem and to evaluate the effectiveness of ecosystem restoration activities and natural hydropattern changes which are occurring over time. This work is an extension of the USEPA REMAP research and monitoring conducted from 1993-96 and is consistent with the objectives of the South Florida Mercury Science Program (SFMSP) and the Everglades restoration activities. The studies in Phase II are designed to fill existing data gaps in the ecological baseline assessment (habitat assessment), initiate trend monitoring, provide additional input for models of mercury cycling, landscape, and water management and to determine systemwide responses to management actions.

Objectives

The USEPA South Florida ecosystem assessment project is an innovative, large-scale monitoring and assessment program designed to measure the current and changing conditions of ecological resources in South Florida using an integrated, holistic approach. The ultimate goal of this program is to provide decision makers with sound ecological data to improve environmental management decisions on multiple environmental issues and restoration efforts in the Everglades. The South Florida ecosystem assessment project provides a foundation for addressing the multiple issues that are critical to the restoration of the Everglades ecosystem and contributing to the Interagency Task Force on Ecosystem Restoration efforts. The South Florida ecosystem assessment project uses the EPA ecological risk assessment framework (USEPA 1992) as a foundation for providing decision makers with critical information. The program is guided by seven policy relevant assessment questions:

1) **Magnitude** - What is the magnitude of the problem(s) in the Everglades?
2) **Extent** - What is the extent of the problem(s)?
3) **Trend** - Is the problem(s) getting better, worse, or staying the same?
4) **Cause** - What factors are associated with or causing the problem(s)?
5) **Source** - What are the sources contributing to the causes and what is the importance of different sources to the problem(s)?
6) **Risk** - What are the risks to different ecological systems and species from the stressors of factors causing the problem(s)?
7) **Solutions** - What management alternatives are available to ameliorate or

eliminate the problem(s)?

The seven questions listed are equally applicable to each issue impacting the Everglades ecosystem, such as, hydropattern modification, Hg contamination, eutrophication, habitat alteration, and endangered and exotic species.

The USEPA South Florida ecosystem assessment project is a long-term research, monitoring and assessment program. Initial conceptual models and testable hypotheses have been developed. A number of studies will be required to test all of the hypotheses and to refine the conceptual models and complete the ecological risk assessment in the Everglades. Initially, the South Florida ecosystem assessment project has focused on a subset of hypotheses which are directly related to the first four policy-relevant assessment questions identified above. Additional coordinated studies directed at addressing other high priority elements of the interagency program will be conducted and merged with this program.

Multiagency Ecosystem Restoration Efforts

A series of efforts by many agencies are underway to protect and restore the Everglades ecosystem. In 1994, the Florida Governor established the Governor's Commission for a Sustainable South Florida to make recommendations for achieving a healthy Everglades ecosystem that can coexist with and be mutually supportive of a sustainable South Florida economy and quality communities. This Commission has adopted five guiding principles (1) restore key ecosystems, (2) achieve a cleaner environment, (3) limit urban sprawl, (4) protect wildlife and natural areas, and (5) create quality communities and jobs. The Commission has also concluded that, on its present course, South Florida is not sustainable (Governor's Commission for a Sustainable South Florida 1995). The US Army Corps of Engineers (USACE) is currently conducting a restudy of the Central and Southern Florida Project to evaluate the feasibility of structural or operational modifications to the project, and identify those modifications that are essential to restoration of the Everglades and Florida Bay ecosystems while providing for other water-related needs (USACE 1994). The federal Water Resources Development Act of 1996 established the South Florida Ecosystem Restoration Task Force, composed of representatives of federal agencies, state agencies, Indian Tribes and local governments, to coordinate the development of consistent strategies for restoration, protection and preservation of the South Florida ecosystem (US Congress 1996). The Science Subgroup of this Task Force has developed integrated scientific information needs for the ecosystem restoration effort (Science Subgroup 1996) along with success criteria for South Florida ecosystem restoration (Science Subgroup 1997). In addition, the State of Florida has many other ecosystem restoration efforts underway (SFWMD 1997a) including a comprehensive plan to address Everglades eutrophication through land acquisition, construction projects, research, and regulation, as required by Florida's 1994 Everglades Forever Act (SFWMD 1997b). Phase I of the phosphorus control program is using a combination of agricultural best management practices and 174 km^2 (43,000 acres) of constructed wetlands (i.e., stormwater treatment areas) to achieve phosphorus removal. The goal of Phase I of the phosphorus control program is to decrease total phosphorus (TP) concentrations in the water discharged to the public Everglades to at least 50 ug/L.

Many other federal and state agencies and universities, including the US Environmental

Protection Agency (USEPA), the US Geological Survey (USGS), US National Park Service (NPS), Florida Department of Environmental Protection (FDEP), and South Florida Water Management District (SFWMD), US Army Corps of Engineers (USACE), Loxahatchee National Wildlife Refuge (LNWR), Florida Game and Freshwater Fish Commission (FGFFC), Indian Tribes, and Industry currently are conducting monitoring, modeling or restoration programs within the Florida Everglades to evaluate the condition of Everglades resources and restoration alternatives. The research and monitoring by USEPA is unique in its system-wide multi-media survey sampling design.

Project Participants: The principals actively involved in this team effort include Dr. Jerry Stober, Project Manager, USEPA, Region 4, SESD, EAB, Athens, Ga.; Dan Scheidt, South Florida Coordinator, USEPA, Region 4, Water Management Division, Athens, Ga.; Dr. Ron Jones, Director, Florida International University, Southeast Environmental Research Program, Miami, Fl; Drs. Kent Thornton and Lisa Gandy, FTN Associates, Ltd, Little Rock, AR; Dr. Don Stevens, Dynamac, Inc., Corvallis, OR; Joel Trexler, Florida International University, Southeast Environmental Research Program, Miami, Fl; Dr. Roy Welch, University of Georgia, Center for Remote Sensing & Mapping Science, Athen, Ga; Steve Rathbun, University of Georgia, Statistics, Athens, Ga; Bob Ambrose, Dr. Craig Barber and Dr. Rochelle Araujo, USEPA, NERL–Athens, Ga.; Brenda Lasorsa, Battelle Marine Science Laboratory, Sequim, WA; Dr. Carol Kendall, USGS, Menlo Park, CA; Mike Birch, USEPA, SESD, OQA, Athens, Ga.; Jenny Scifres, USEPA, SESD, ASB, Athens, Ga.

Project Responsibilities: A USEPA Region 4 SESD EAB senior scientist will be responsible for overall project management. An EAB sampling team will be responsible for the pilot study and synoptic field sampling during the dry season (April) and wet season (September). A three laboratory design including FIU-SERP as the primary analytical laboratory with Battelle MSL and EPA SESD ASB as two secondary laboratories will be used. Distribution of the analytical work load among the three laboratories is required for QA/QC intercomparisons and to physically complete the analyses within the required holding times for the large volume of samples generated during 8-9 days of sampling alotted for each cycle. In addition, utilization of the same analytical laboratories which developed the methodologies and produced the REMAP Phase I baseline results will ensure continuity of the database enhancing the ability to detect change over time. Region 4 SESD OQA will have final responsibility for the QA/QC evaluations of the laboratories, however, FTN Associates will be used to conduct the initial reviews of the data. SESD EAB will be responsible for database management, data analysis, interpretation, and presentation with support from FTN Associates, FIU-SERP, UGA-Statistics, and ORD-EMAP**.**

Projected Timeline– Three field events are planned including a pilot study (methods development and interlaboratory calibration) in January 1999, a dry season survey in April 1999 and a wet season survey in September 1999.

Milestones and Products in FY99-00

September 1998–Peer Reviews ORD EMAP and Science Coordination Team of the South Florida Ecosystem Restoration Task Force

October 1998–Initiate Aerial Photo Habitat Assessment with CRMS at UGA and design detailed pilot study.

November 1998–Design and build Sampling Devices to Be Tested in Pilot Study

January 1999–Conduct pilot study testing field and analytical methods in the Everglades

February 1999–Evaluate Interlaboratory Calibration and Pilot study results.

March 1999–Develop final field and laboratory protocols. Report habitat sampling results in peer review journal. Train crew and prepare for the Spatial REMAP sampling

April 1999–Conduct Dry Season Sampling

August 1999–Complete Dry Cycle Sample Analyses

September 1999–Conduct Wet Season Sampling

January 2000–Complete Wet Cycle Sample Analyses

June 2000–Bring data analysis to conclusion with a final report

Task 1. University of Georgia, Center for Remote Sensing and Mapping Science (CRMS) Aerial Photo Habitat Assessment (Roy Welch): A Phase I probability assessment of habitat was conducted by visually determining the major habitat types at each sampling location and documenting the sites with 35 mm photographs. These procedures permitted qualitative estimates of presence and dominance of selected emergent plant species and floating periphyton at each site. However, quantitative estimates are needed in Phase II to provide plant biomass and mercury concentrations for input to Everglades mercury cycling models. Estimates of plant biomass along the system are also needed to document baseline responses to the nutrient gradient. The Center for Remote Sensing and Mapping Science (CRMS) at the University of Georgia is developing detailed vegetation maps and digital databases for the Federal park lands in south Florida using aerial photo intrepretation techniques. These techniques will be applied in this study, however, they will be adapted to the USEPA probability sampling design used for assessment and monitoring of the Everglades ecosystem. CRMS has the necessary experience and tools to accomplish this task in a minimum time frame while ensuring systemwide data comparability. The steps involved in building the vegetation database for the random sample points are listed below.

A. CRMS will obtain U.S. Geological Survey (USGS) National Aerial Photography Program (NAPP) color infrared aerial photo transparancies for the study area (WCA-1,2,3, ENP

and Rottenberger).

B. USEPA will provide CRMS with the UTM map coordinates (NAD 83) for the approximately 260 random sample points to be used in the survey. Map interpretation will be conducted in the following order to facilitate the pilot study (January 1999), dry season survey (April 1999) and the wet season survey (September 1999).

1. Pilot study – six points
2. Dry season survey – 130 points
3. Wet season survey – 130 points

The pilot study and each survey will have a unique set of randomized spatially distributed sampling points which will be identified with a unique numbering system. The survey points will be ordered by latitude from north to south.

The aerial photo interpretation will provide the detailed information for each site on which the field sampling will be based, therefore completion of the digitized habitat maps must preceed the field sampling by atleast two months.

C. The CRMS will plot the sample site locations on the NAPP color infrared aerial photographs, interpret the vegetation density of all plant species or communities which can be identified consistently from the photographs. While particular attention will be focused on sawgrass, cattails and/or periphyton at each location with subsequent biomass sampling by USEPA, interpretation of the photos to evaluate all plant species/communities which can be consistently identified in the photos for changes in presence/absence, abundance and/or density will maximize the information generated. Interpretation will focus on 1 x 1 km square plots centered at the GPS coordinates for each sample point. A vegetation map in digital format will be prepared for each 1 x 1 km plot.

D. The pilot study digital vegetation maps will be provided to ORD EMAP (Corvallis) for development of an algorithm to weight (to the center point) the selection of random sampling points for plant species biomass determination. This will be tested on the six pilot study stations. Following development of the algorithm it will be tested on the dry season survey points to evaluate the logistical requirements of the systemwide sampling effort. With development of the final working algorithm it will be provided to CRMS for point location on the remaining digital vegetation maps.

E. The USEPA Region 4 field sampling team will load each site map with associated plant type polygons into Field Notes on a laptop and the field sampling crew will ground truth the plant type communities. CRMS experts will assist EPA habitat assessment teams in the appropriate field observations most appropriate to air photo interpretation and accompany the habitat assessment team during the pilot study.

F. CRMS will provide USEPA Region 4 with spreadsheets of the surface areas of each plant species or community type identified from the aerial photo interpretation for each station to which EPA will add the sample biomass estimates. CRMS will also undertake spatial interpolations of the station point data to establish variations or trends in plant distributions and

to provide a basis for future comparisons.

G. A final report of this work will be provided to USEPA Region 4 Project Leader by December 31, 1999.

Task 2. University of Georgia, Statistics (Steve Rathbun): General statistical support to this study will be provided on an as need basis during the analysis of the data. Numerous opportunities exist to develop both design- and model-based statistical analyses, requiring the development of new statistical methods. Design based analyses require methods for assessing the uncertainty of statistical summaries such as provided by cumulative distribution functions. In addition, methods are required for evaluating the current sampling designs to ensure that adequate power is achieved to answer the objectives of the respective monitoring initiatives. Model based analyses require the development of models that mimic the complex processes that occur in nature. Environmental processes are complex, involving interactions of numerous biotic and abiotic factors over different spatial and temporal scales.

The specific objectives of the required research are as follows:

A. Develop spatio-temporal models for the data from the Everglades ecosystem. These models shall take into consideration processes occurring at all spatial and temporal scales including habitat, mercury and water quality indicators.

B. Develop methods for combining data collected at different spatial and temporal scales and trophic levels.

C. Develop methods required for analyzing spatially and temporally correlated data when some observations are left-censored by the detection limits of instruments used to measure contaminants.

Task 3. Florida International University (FIU), Southeast Environmental Research Program (SERP). This task will utilize the three laboratories (FIU-SERP, Battelle MSL and EPA-SESD) involved in Phase I to analyze the comprehensive array of samples of water, soil, and tissue (plants and fish) and to conduct the routine QA/QC requirements.

A. Primary Analytical Laboratory (Ron Jones): FIU-SERP (only laboratory addressed in this IAG) will be the primary analytical laboratory for this project and the facility from which the USEPA field sampling team will stage field activities. The methods previously developed by FIU-SERP for Phase I will be utilized in Phase II to maintain continuity of results. FIU-SERP will assist USEPA in the testing and development of new field sampling and analytical methods during the pilot study in January 1999. New methods for phase II include development of porewater sampling, dissolved nutrients and selected anions, sulfate/sulfide ratios, diatom species composition and periphyton pigment analyses and macrophyte mercury analyses. All sampling and analyses to be carried out during the next cycles of the study will be tested and proven during the pilot study. The pilot study analytes will include HgT, MeHg, TP, TN, dissolved nutrients (NH4, NO2, NO3, PO4), TOC, sulfate, sulfide in surface water; TP, TN, dissolved nutrients, selected anions (Br, Cl, F, NO2, NO3, O-p, SO4), sulfide in porewater; HgT, MeHg, sulfate, sulfide, TP, CH4 and CO2 in soil; HgT, MeHg and EtHg in floating and soil periphyton; HgT, MeHg, and EtHg in sawgrass and cattails; and HgT in mosquitofish. All of the

media will be composited and split with equal amounts of water, soil or tissue going to each laboratory. The mosquitofish will be analyzed two ways as individual fish (7 per sample) as well as a homogenate for QA/QC purposes. Each laboratory will analyze three replicates of each sample for each station to provide a statistically valid data set on which to conduct an analysis of the interlaboratory calibration. USEPA SESD EAB field sampling team will be responsible for "clean" sample collection, splits will be conducted in the FIU-SERP laboratory and the EPA/ESAT field team will be responsible for ensuring chain of custody, sample tracking, shipping of blind, split, duplicate and replicate samples to each laboratory. The data will be returned to FTN Associates who will be responsible for statistical analysis of the data and report preparation and presentation to EPA Region 4 SESD OQA for final review to ensure the QA/QC requirements have been fullfilled.

FIU-SERP will assist USEPA in the development of a biomass sampling method for macrophytes which is quantitative, efficient and practically deployed from a helicopter. The pilot study will determine the wet/dry ratios of various volumes of biomass from 0.1, 0.5 and 1.0 m^2 clip plots. An effort will be made to determine if this large volume of biomass can be weighed in the field thus optimizing logistics.

Phase I floating and soil periphyton samples were collected at each station when present for mercury analyses, however, biomass was not measured. FIU-SERP will assist EPA in phase II methods development which will include quantitative biomass estimates of soil, epiphytic and floating periphyton. Each type of periphyton will be collected from a known surface area (i.e., surface of soil cores for soil periphyton; standard grid for floating and epiphytic periphyton) and placed into a volumetric cylinder to estimate volume. Periphyton samples will continue to be collected for analysis of total, methyl- and ethyl-mercury, diatom species composition and pigments which will be the responsibility of FIU-SERP.

Following the pilot study a standard protocol will be developed which optimizes the biomass estimation methods. These protocols will be presented in a report for peer review by EPA Region 4 SESD. The protocol will subsequently be validated during each sampling cycle with spatially distributed duplicates from 10% of the sampling stations.

Mosquitofish sampling sites are spatially distributed across the marsh at the same randomly selected water quality monitoring sites. Seven individual fish will be analyzed for total mercury concentration at each site which will allow detection of a 10% change in mercury concentrations (among vs within sites) across the system.

A list of the pilot study (interlaboratory calibration) samples indicating the analyte, subarea, laboratory analyzing and the number of samples to be analyzed by each laboratory is presented in Table 1. A complete list of the analytical parameters by laboratory, MDL, and number of samples to be analyzed per cycle are listed in Table 2.

Mosquitofish Food Habits Analysis (Joel Trexler): A strong north to south gradient in the bioaccumulation factor calculated for mercury uptake in mosquitofish was found during Phase I research and monitoring of the Everglades ecosystem. This discovery indicates a series of important interactions are occurring in the system primarily affected by phosphorus loading from the north which impacts the food chain dynamics in the system. One means of assessing these impacts is to analyze the food habits of the omnivorous mosquitofish across the system. This

was done once during the September 1996 marsh survey and will be repeated again in the pilot study, and both dry and wet sampling cycles in 1999. Twelve individual fish will be analyzed at each site for stomach contents. These data will be used in a comparative study with the 1996 food habits analysis to develop an understanding of how changes in the food chain may affect the habits and uptake of this ubiquitous fish species across the system.

Table 1. Everglades Jan '98 Pilot Study and Laboratory Intercalibration (triplicate analysis)

Sites	LOX	AA-N	WCA3-C	WCA3-S	ENP-N	ENP-S
Parameter						
Surf-Water						
Turbidity	1,3	1,3	1,3	1,3	1,3	1,3
Alk-Phosp	1	1	1	1	1	1
HgT	1,2	1,2	1,2	1,2	1,2	1,2
MeHg	1,2,	1,2	1,2	1,2	1,2	1,2
TP	1,3	1,3	1,3	1,3	1,3	1,3
TN	1,3	1,3	1,3	1,3	1,3	1,3
Diss. Nut-NH4,NO2, NO3, PO4	1,3	1,3	1,3	1,3	1,3	1,3
TOC	1,3	1,3	1,3	1,3	1,3	1,3
SO4	1,3	1,3	1,3	1,3	1,3	1,3
H2S	1,3	1,3	1,3	1,3	1,3	1,3
Porewater						
TP	1,3	1,3	1,3	1,3	1,3	1,3
TN	1,3	1,3	1,3	1,3	1,3	1,3
Diss.Nut-NH4,NO2, NO3,PO4	1,3	1,3	1,3	1,3	1,3	1,3
Selected Anions	1,3	1,3	1,3	1,3	1,3	1,

SO4	1,3	1,3	1,3	1,3	1,3	1,3
H2S	1,3	1,3	1,3	1,3	1,3	1,3
Soil						
HgT	1,2,3	1,2,3	1,2,3	1,2,3	1,2,3	1,2,3
MeHg	1,2	1,2	1,2	1,2	1,2	1,2
SO4	1,3	1,3	1,3	1,3	1,3	1,3
H2S	1,3	1,3	1,3	1,3	1,3	1,3
Alk-Phos	1	1	1	1	1	1
AFDW	1	1	1	1	1	1
Bulk Den.	1	1	1	1	1	1
Min. Conc.	1	1	1	1	1	1
TP	1,3	1,3	1,3	1,3	1,3	1,3
CH4&CO2	1,3	1,3	1,3	1,3	1,3	1,3
Peri-F						
HgT	1,2,3	1,2,3	1,2,3	1,2,3	1,2,3	1,2,3
MeHg	1,2	1,2	1,2	1,2	1,2	1,2
EtHg	1	1	1	1	1	1
Diatom comp.	1	1	1	1	1	1
Pigment	1,3	1,3	1,3	1,3	1,3	1,3
Peri-S						
HgT	1,2,3	1,2,3	1,2,3	1,2,3	1,2,3	1,2,3
MeHg	1,2	1,2	1,2	1,2	1,2	1,2
EtHg	1	1	1	1	1	1
Diatom Comp.	1	1	1	1	1	1
Pigment	1	1	1	1	1	1

Sawgrass						
HgT	1,2,3	1,2,3	1,2,3	1,2,3	1,2,3	1,2,3
MeHg	1,2	1,2	1,2	1,2	1,2	1,2
EtHg	1	1	1	1	1	1
Cattails						
HgT	1,2,3	1,2,3	1,2,3	1,2,3	1,2,3	1,2,3
MeHg	1,2	1,2	1,2	1,2	1,2	1,2
EtHg	1	1	1	1	1	1
Fish						
HgT-indiv.	1,2,3	1,2,3	1,2,3	1,2,3	1,2,3	1,2,3
HgT-homo	1,2,3	1,2,3	1,2,3	1,2,3	1,2,3	1,2,3

1-FIU-SERP; 2-BATTELLE; 3-EPA-SESD

Table 2. Analytical Parameters by laboratory, MDL, and sample number for each sampling cycle. Revised August 6, 1998

PARAMETER	PRIMARY LAB	PRIMARY QA/QC	SECOND-ARY QA/QC	MDL	Site No. per cycle	Samp. No.
SURFACE WATER						
DO	SESD	SESD-SOP		0.2 mg/L	129	129
pH	SESD	SESD-SOP		0.1 s.u.	129	129
Temp	SESD	SESD-SOP		0.15 C	129	129
Conductance	SESD	SESD-SOP		1.0 uS	129	129
Redox	SESD	SESD-SOP		1 mV	129	129
Depth	SESD	SESD-SOP		1 cm	129	129
Turbidity	FIU	SESD		0.1 NTU	129	155
Total Phosphorus	FIU	SESD		0.6 ug/L	129	155
Total Nitrogen	FIU	SESD		0.03 mg/L	129	155
Dissolved Nutrients* (NH4,NO2, NO3, PO4)	FIU	SESD		NO3-0.4ug/L NO2-0.1ug/L NH4-0.7ug/L SRP-0.3ug/L	129	155
Total Organic Carbon	FIU	SESD		0.12 ug/L	129	155
Sulfate	SESD	SESD		0.01 mg/L	129	155
Sulfide*	SESD	SESD		0.01 ug/L	129	155
Alk_Phos	FIU	FIU		0.01uM/h	129	155
Chlorophyll a	FIU	FIU		0.1 ug/L	30	33
Total Mercury	FIU	Battelle	SESD	0.3 ng/L	129	187
Methyl Mercury	Battelle	FIU		0.02 ng/L	129	187
PORE WATER						
Total Phosphorus*	FIU	SESD		0.6 ug/L	129	171

Total Nitrogen*	FIU	SESD		0.03 mg/L	129	155
Dissolved Nutrients* (NH4, NO2, NO3, PO4)	FIU	SESD		NO3-0.4ug/L NO2-0.1ug/L NH4-0.7ug/L SRP-0.3ug/L	129	155
Anions* (Br,Cl,Fl,NO_2,NO_3,O-p,SO_4)	FIU	SESD		ion chrom.	129	155
Sulfate	SESD			0.01 mg/L	129	171
Sulfide*	SESD	SESD		0.01 ug/L	129	171
SOIL/SEDIMENT						
Type	SESD				129	129
Thickness	SESD			1 cm	129	129
pH	SESD				129	129
Redox (in situ)	SESD			1 mV	129	129
Redox (lab)*	SESD			1 mV	129	129
Total Mercury	SESD	FIU	Battelle	3 ug/kg	129	155
Methyl Mercury	FIU	Battelle		0.2 ug/kg	129	155
Ethyl Mercury	FIU			0.2 ug/kg	129	155
Sulfate	SESD			0.01 ug/kg	129	155
Sulfide*	FIU			0.01 ug/kg	129	155
Total Phosphorus	FIU	SESD		0.06 mg/kg**	129	155
Ash Free Dry Wt	FIU			0.02 mg/kg**	129	155
Bulk Density	FIU			0.001 g/cc**	129	155
Mineral Content	FIU			3%	129	155
CH4*	FIU	SESD			129	155
CO2*	FIU	SESD			129	155
Alk_Phos	FIU	FIU			129	155

PERIPHYTON--floating						
Total Mercury	FIU	SESD	Battelle	3 ug/kg	100	110
Methyl Mercury	FIU	Battelle		0.2 ug/kg	100	110
Ethyl Mercury	FIU			0.2 ug/kg	100	110
Biomass*	SESD			1 g	100	110
Surface Area* (%cover)	UGA				50	
Diatoms*	FIU				30	33
Pigments*	FIU				30	33
PERIPHYTON--soil						
Total Mercury	FIU	SESD	Battelle	3 ug/kg	100	110
Methyl Mercury	FIU	Battelle		0.2 ug/kg	100	110
Ethyl Mercury	FIU			0.2 ug/kg	100	110
Biomass*	SESD			1 g	100	110
Diatoms*	FIU				30	33
Pigments*	FIU				30	33
SAWGRASS						
Total Mercury*	FIU	SESD	Battelle	3 ug/kg	65	72
Methyl Mercury*	FIU	Battelle		0.2 ug/kg	65	72
Ethyl Mercury*	FIU			0.2 ug/kg	65	72
Biomass*	SESD			10 g	65	72
Surface Area* (% cover)	UGA				65	
CATTAILS						
Total Mercury*	FIU	SESD	Battelle	3 ug/kg	40	44
Methyl Mercury*	FIU	Battelle		0.2 ug/kg	40	44

Ethyl Mercury*	FIU			0.2 ug/kg	40	44
Biomass*	SESD			10 g	40	44
Surface Area* (% cover)	UGA				40	
HABITAT EVALUATION * (% cover, pres/absence)	UGA				129	129
MOSQUITO-FISH						
Total Mercury	FIU	SESD	Battelle	1 ug/kg	129	1043
Length	FIU			0.1 mm	129	993
Weight	FIU			0.05 g	129	993
Sex	FIU				129	993
STABLE ISOTOPE ANALYSIS	USGS				129	993
FOOD HABITS ANALYSIS	FIU				129	993

*= new parameter
**= minimum reportable quantities
HgT in water = 129 sites, 16 field blanks, 13 duplicates, 16 equip. blanks, 13 splits = 187
Porewater (nutrients/anions) = 129 sites, 13 dups, 16 equip blanks, 13 splits = 171
HgT in soil = 129 sites, 13 dups, 13 splits = 155
HgT in fish = 129 sites @ 7 fish/site = 903, 90 dups, 50 stand. tissue = 1,043

B. QA/QC Requirements: Data package requirements for USEPA Region 4, Science and Ecosystem Support Division (SESD).

1. Data Quality Requirements and Validation: In all data collection activities, data quality requirements will be specified in five areas: accuracy and bias, precision, comparability, completeness and representativeness (Stanley and Verner, 1985; Smith et al., 1988). Method detection limits have been specified based on the phase I REMAP monitoring and some have been lowered where lower detection levels are needed. The validation process will consider each of the following components using a statistically appropriate method.

Accuracy and Bias: Accuracy is the degree to which a measured value or property agrees with an accepted "true" value (Taylor 1988). Accuracy is estimated by measuring a sample with a know reference value. Bias is the systematic error inherent in a method or caused by some artifact or idiosyncrasy of the measurement system. One-way bias is estimated by interlaboratory comparison of performance evaluation samples among laboratories.

Precision: Precision is a measure of the scatter among independent repeated observations or measures of the same property made under prescribed conditions (Taylor 1988). Precision can be estimated at several points in the data collection process in order to estimate the effects of different sources of error. Precision can be partitioned into analytical and measurement system precision. Analytical precision refers to precision of the analysis performed by analytical instruments; it is estimated by laboratory replication, including replicates of performance audit samples. Measurement system percision refers to the precision of the sampling process, including sample collection, storage, transport, preparation and analysis. Collocated field duplicated are used to estimate precision of the entire measurement system, and laboratory splits are used to estimate the precision of sample processing after the sample has been received at the laboratory.

Precision and bias are estimates of random and systematic error in a measurement process (Kirchner 1983, Hunt and Wilson 1986). Collectively, they provide an estimate of the total error or uncertainty associated with an individual measurement, or set of measurements. Estimates of the various error components will be determined primarily by replicate sampling. The statistical design and sampling plan will minimize systematic errors in all components except measurement error by using documented methodologies and standardized procedures. If new more sensitive methods must be developed or analytical modifications made documentation will be provided as the process moves toward standardization. In addition, standard samples will be included in the field and subjected to the entire collection and measurement process. Variance components of the collection and measurement process (e.g., among analytical laboratories) will be estimated after the pilot study and at the completion of each cycle so the QA efforts can be allocated to control major sources of error.

Comparability: Comparability is defined as "the confidence with which one data set can be compared to another" (Stanley and Verner 1985, Smith et al. 1988). Comparability studies will be conducted with cooperating laboratories and agencies through round robin analyses. Identical field collection and laboratory procedures will be used when possible.

Completeness: Completeness requirements for this monitoring effort will be that 90 percent of all proposed samples are collected and analyzed.

Representativeness: Representativeness is defined as "the degree to which the data accurately and precisely represent a characteristic of a population parameter, a variation of a property, a process characteristic, or an operation condition" (Stanley and Verner 1985, Smith et al., 1988). The statistical survey, sampling periods and sample locations were selected to ensure representative samples.

Tolerable Background Levels: Background is operationally defined as the amount of contamination due to collection, handling, processing and measurement. It is particularly relevant to the

measurement of trace concentrations of mercury species. Background levels will not be tolerated due to the use of "clean sampling and analytical techniques" and if detected the source will be isolated and eliminated. Field and laboratory blank samples will be added to each day's samples and used to control and eliminate background contamination.

Data Quality Objectives: The assessment of Data Quality Objectives will follow the guidance provided in EPA QA/G-4 (EPA 1994) or a revision intended for research projects which is currently under development. This assessment of the data will be compared after the pilot study and each cycle of spatial sampling for comformance to the Phase I results. Deviations with Phase I results will be investigated and the most probable explanation developed. The overall goal of maintaining consistency in the database between Phase I and Phase II is most important to provide the most accurate basis for trend assessments.

2. Specific Data Package Requirements: The specific requirements for laboratories which submit results and data packages to the USEPA Region 4, SESD for validation are contained in the attached document entitled **"Laboratory Documentation and Quality Control Requirements for Data Validation, August 1998**. These requirements must be addressed in the laboratory's QA plan which must be approved by the SESD Office of Quality Assurance prior to the initiation of sample analysis. All data reported from each analytical laboratory for Phase II will be transmitted in electronic format (variable by numeric station ID indicating analytical batch order and all other required QA information) in either Excel, QuattroPro or dBase IV. Any additional format requirements will be specified by EPA prior to initiation of the data collection. FTN will be the initial repository for the data who will compile the database and conduct the initial QA/QC review of the data.

Task 4. QA/QC Data Review/Data Analysis/Comparative Ecological Risk Assessment/Final Reports (FTN Assoc.–Kent Thornton)

A. **Independent QA/QC Data Review:** FTN will independently review the QA/QC data forwarded from each of the three laboratories in Phase II of the Everglades Ecosystem Assessment Project to verify adherence to stated QA objectives and criteria. DQO requirements will be met through the following approaches:

1. Accuracy and Bias-- Comparisons of performance evaluation samples will be used to estimate accuracy and bias of the laboratory results. In addition to the PE samples, internal standards developed by the laboratory will be used to assess accuracy (bias) and matrix spikes will be evaluated to assess matrix interferences with the analytical procedure.

2. Precision -- Field and laboratory replicates (duplicate and split samples) will be used to assess precision of the sampling and analytical methods. Replicates of performance audit samples will be performed in addition to field samples. Percent relative standard deviation estimates represent one of the statistics to be calculated for precision.

3. Comparability -- Comparability will be verified by using results of round robin analyses among the analytical laboratories. Typically, standard methods are used to assist with comparability, but there are no standard methods for mercury and this is a program to develop and refine analytical methods.

4. Completeness -- Completeness will be assessed by comparing the results of the field sampling effort to the goal of having 90% of all proposed samples collected and analyzed. This goal does not include sites where no samples can be obtained because the site was dry or located on private land.

5. Representativeness – Representativeness will be achieved by following the statistical survey design which ensures probability samples will be collected. By definition, a probability sample is representative of a specific, known proportion of the population.

6. Tolerable background levels – Field and laboratory blank samples will be used to assess background levels and establish minimum detection limits and quantitation limits.

7. Data Quality Objectives developed during Phase I will be used for comparison with QA results. Upon receipt of the data files range checks will be conducted for each constituent. Data will be plotted on control charts to ensure data are within the DQO specifications (e.g., +/- 3 standard deviations, etc.). The data will be flagged, as appropriate, if QC checks do not satisfy QA requirements. Additional QC analyses will be conducted as part of the statistical analysis of the data.

The QA/QC data review package with the associated evaluations by FTN will be presented to Region 4 SESD OQA for final review.

B. Data Analysis: The accumulation of data from the Phase II monitoring effort requires continued analytical support. Statistical analysis in cooperation with Region IV, SESD, EAB is needed to assist in evaluation, interpretation and integration of the data to achieve sophisticated assessment results from the database. Phase II data will be analyzed in series with Phase I data to begin tend analysis where possible.

C. **Comparative Ecological Risk Assessment:** The comparative ecological risk assessment of the effects of mercury and other interacting variables on south Florida ecosystems will be guided by the following outline. The initial ecorisk assessment has been deferred to allow additional information to accumulate from other sources and to follow Phase II monitoring. Multiple iterations of this ecorisk assessment are necessary to include more information which is becoming available from the South Florida Mercury Science Program and other south Florida reasearch over time.

A Visual Basic program was formulated around the EPA Ecological Risk Assessment paradigm and was used to help identify the important assessment questions, develop logic paths and decision trees for addressing these questions, and information needs to conduct a comparative ecological risk assessment of the effects of mercury on South Florida ecosystems. Since this program was developed, several changes have occurred within the EPA EMAP and REMAP program that require modification of the original plan for conducting this comparative assessment. This describes the modified approach proposed for each of the phases of the risk assessment.

Phase I: Problem Formulation

1. Stressor Characterization
   a. Literature review of mercury characteristics and information on mercury in AQUIRE, IRIS and similar data bases.
   b. Literature review and brief summarization on other stressors including total phosphorus, habitat alteration, hydroperiod modification and exotic species introductions.
   c. Discussion of primary and secondary stressors and the interactions among stressors.
2. Ecosystems at Risk - Previously Identified in EcoRisk Model
   a. Description of ecosystems, including geographic location, unique habitats and species.

b. Tabulated format for information, with satellite images and pictures similar to D. Scheidt's briefing book.

3. Ecological Effects
   a. Literature review, building on reviews of Weiner, Loftus and Spalding.
   b. Assess chronic and acute effects of mercury, including secondary effects such as higher susceptibility to DNA damage from radiation at higher Hg concentrations.
   c. Re-evaluate the ecological effects listed in the EcoRisk model such as reproductive failure, tetralogies, decreased feeding efficiency. While these impacts or effects might be true, little, if any, evidence that these changes are occurring.
   d. Human health issues are secondary to this assessment, but there are potential impacts to the human population.
4. Ecological Endpoints
   a. Three areas valued by society are T&E species, wildlife and habitat protection, and aesthetics.
   b. Ecological or assessment endpoints have been selected for each of these and are incorporated in the EcoRisk Model - Florida panther, alligator, wading birds, and fish.
   c. Measures of effects (measurement endpoints) such as Gambusia, hair and feather mercury concentrations also have been identified.
5. Conceptual Model
   a. Model has been developed based on REMAP data. This model will be expanded to incorporate the risk hypotheses developed during the February 1996 workshop (See Attachment).
   b. The conceptual models developed as part of the original program will be integrated into this model.
   c. The conceptual model will continue to evolve as additional information and additional hypotheses are generated.
6. Preliminary Uncertainty Analysis
   a. This analysis has already been initiated as part of the development of the conceptual model.
   b. This analysis will be refined as additional analyses are conducted.

Phase II. Analysis

1. Stressor Characterization, Ecosystem Characterization and Relevant Effects Data
   a. REMAP data analysis will be conducted to assess seasonal mercury and associated constituent dynamics and the potential effects of seasonal hydrology on these dynamics.
   b. Literature review to assess seasonal differences in species distributions and behaviors (feeding, breeding, nursery areas,) will be correlated with seasonal patterns in mercury dynamics.
   c. Deposition information from the FAMS network will be obtained and evaluated along with SOFAMS (if the analyses are available in time).
   d. Other subtropical studies will be evaluated, particularly those presented at the 2nd, 3rd, and 4th International Mercury conferences, for relevance to South Florida Everglades.

2. Exposure Analysis
   a. Literature review of other systems to identify other exposure pathways or

methylation/demethylation processes.

b. Statistical analyses relating possible stressors to exposure concentrations will be conducted, including the unique set of environmental conditions that must occur for mercury methylation.

c. Order of magnitude source apportionment calculations will be made to assess the relative contributions of different sources and subsequently used to evaluate possible management scenarios for risk management.

d. ORD Screening Model results will be used to assess possible pathways and factors contributing to mercury methylation and subsequent methyl mercury concentrations.

e. The availability of mercury cycling models, (e.g., ORD Screening Model, MERC5) and mass balance models (e.g., SFWMD ENRP model, Multimedia **fate models)** will be utilized to the extent of their availability.

f. Sensitivity analysis will be an integral part of these analyses. In addition, if time permits, Monte Carlo or similar "stochastic" approach (e.g., Regional Sensitivity Analysis) will be used to determine the range of possible exposures that might occur.

3. Ecological Response Analysis

a. Literature review of laboratory studies that have been conducted on dose-response relationships for different organisms.

1) These typically will be acute doses.

2) Limited information on chronic toxicity and even less information on dose-behavior relationships.

b. Search on-line data bases (AQUIRE, CCRIS, CESARS, ENVIROFATE, IRIS, etc) for information on dose-response relationships for mercury.

c. Information from on-going studies will be solicited for inclusion in the risk assessment - ATLAS, ELM, BASS.

d. A simple food chain model can be developed for critical path analysis that provides information similar to BASS, this is done using a spreadsheet model. This will be done when information from cooperators becomes available.

e. Sensitivity analysis will be an integral part of these analyses. In addition, if time permits, Monte Carlo or similar "stochastic" approach (e.g., Regional Sensitivity Analysis) will be used to determine the range of possible effects that might occur. (This may have to be deferred following development of a food chain model).

4. Uncertainty Analysis

a. The assumptions made for each of the analyses, and the potential impact of these assumptions on the conclusions, will be documented.

b. Assumptions inherent in the REMAP design and other field, laboratory studies, and the potential impact on decisions, also will be documented.

Phase III. Risk Characterization

1. Integration

a. The Quotient Method will be used as a Tier 1 screen of possible impacts by comparing (Exposure Conc)/(Effects Conc).

b. ORD Screening Model and MERC5 results will be used as input to the simple food chain model or other food chain models available.

c. A Markov chain or similar probability chain can be developed to assess the likelihood of effects given different stressor input concentrations and different seasonal scenarios. This is not currently planned, but could be developed in future assessments. (It will be deferred in this assessment).

d. Various management scenarios will be identified and evaluated using the integrated models to assess the consequences of different management strategies.

2. Uncertainty Analyses
   a. Results from Phase I and II uncertainty analyses will be integrated with uncertainty arising from integrating various models or anecdotal information.
   b. These uncertainty analyses will be qualitatively used to evaluate the certainty of conclusions made in Risk Summary and Risk Significance Sections.
   c. The uncertainty associated with different management strateties also will be qualitatively estimated.
3. Ecological Risk Assessment Summary
   a. Process of Elimination approaches will be used to assess possible ecological effects that might be occurring because of other stressors and assess the likelihood that the effect observed can be attributed directly to mercury.
   b. Weight of Evidence approaches will be used to corroborate possible effects identified through the process of elimination. This must be a joint effort of all the collaborators on the REMAP and ORD projects so that we have consensus among investigators on these conclusions.
   c. The potential risk associated with different management scenarios will be incorporated in the risk assessment summary so the potential consequences of management strategies can be incorporated into the Risk Management Analyses.
   d. A qualitative uncertainty ranking system will be used to score the certainty of various conclusions.
4. Ecological Significance
   a. Ecological significance will use the flow chart approach developed by Thornton and Gentile and incorporated in the Gentile et al. Issue Paper and included in the revised EcoRisk model.
   b. This includes considerations of temporal and spatial scales, reversibility of the effects, and magnitude of the response.
   c. A qualitative approach will be used to rate the ecological significance of mercury compared to other stressors.
5. Presentation of Results
   a. The risk assessment will be documented in a report and published in the scientific literature.
   b. The risk assessment information will be packaged separately for different audiences and different managers based on their information needs and the most effective approach for providing them with this information.
   c. Communication specialists will be consulted at this stage.

Assessment is a process, not a product. Multiple iterations of this assessment are needed to include new information becoming available each year of the South Florida Mercury Science Program in an on-going process of evaluating the effects of mercury on South Florida ecosystems.

ATTACHMENT

1. Risk Hypotheses
   a. Canals

1) Total phosphorus stimulates primary productivity, which contributes to anaerobic conditions, and an increase in microbial activity and methylation of Hg

or

Total phosphorus stimulates microbial decomposition, which contributes to anaerobic conditions, and an increase in microbial methylation of Hg.

2) Canals receiving drainage from the EAA have increased methylation in the canals and subsequent uptake through the food chain.

or

Canals receiving drainage from the EAA receive increased methyl mercury that is produced in the EAA and discharged into the canals with subsequent uptake through the food chain.

or

Canals receiving drainage from marshes have higher methyl mercury concentrations because of increased methylation in the marshes. The methyl mercury concentration in the canals is directly proportional to the surface area of the marsh draining into the canals.

3) The northern canals serve as a source of mercury while the southern canals serve as a mercury sink because of transport and sedimentation.

4) The northern canals have an incomplete, low diversity food web with few steps in the food chain while the southern canals have a more diverse food web with more links in the food chain and, therefore, greater biomagnification of mercury through the food chain.

5) The conceptual model for mercury in fish is: Northern Canals [Hi TOC,SO_4,S-,Lo THgF] => Alligator Alley-Tamiami Trail Canal Sector [Mod TOC, SO_4, Lo S-,TP, HiTHgF] => Southern Canals [Lo TOC, SO_4, TP,THgF].

6) The proximity to air sources results in elevated THgF concentrations. Canals closest to the eastern shore of FL have the highest THgF concentrations. Canals in the EAA have the highest THgF concentrations because of the burning of sugar cane.

7) Mercury containing agricultural chemicals contribute to elevated THgF concentrations.

8) Differences in the mercury regime are due to the high energy regime in the canals versus the low energy regime in the marshes (e.g., sedimentation and burial).

b. Marshes

1) Increased total phorphorus concentrations result in increased decomposition of peat with the greatest methylation occurring at the transition between the aerobic and anaerobic phase.

2) The processes in canals that result in elevated THgF are the same processes that result in elevated THgF in the marshes.

C_o: The processes are similar but of different importance.

C_o: The processes resulting in elevated THgF concentrations are different in canals and marshes.

3) Marsh areas proximal to the canals have higher THgF concentrations.

C_o: The high energy regime of canals results in lower methylation rates because there is lower sedimentation in canals than marshes.

C_o: Marshes are high filtration systems compared to the canals, which is why the short transect gradients in TP and THgF concentrations are observed.

4) Long hydroperiod peat marshes have higher THgF concentrations.
5) The THgF concentrations in marshes are controlled more by soils than canal THgF concentrations are controlled by sediments.
6) Periphyton/emergent vegetation control biomagnification in marshes.
7) The proximity to air sources results in elevated THgF concentrations. Marshes closest to the eastern shore of FL have the highest THgF concentrations. Marshes in the EAA have the highest THgF concentrations because of the burning of sugar cane.
8) Peat marshes have higher THgF concentrations than marl marshes.
9) The difference between peat and marl marsh THgF concentrations are controlled by burial and removal of mercury.
10) Vegetation removes elemental mercury from the air and methylates/ethylates it, resulting in increased methyl mercury concentrations in water.

C_o: Vegetation pumps mercury from the sediment/water and releases it to the atmosphere.

11) Food web differences (incomplete versus diverse food chain links) account for the elevated THgF (e.g., enriched marshes => incomplete food webs).
12) Geologic history (i.e., characteristics) control processes influencing methylation and uptake**.**

c. These hypotheses need to be included in the report, along with any other hypotheses that were considered during the analysis; not just the hypotheses that were retained. It is important that the reader understand multiple hypotheses were considered during these analyses. Additional hypothese will be added, revised and reviewed during the analytical process.

Deliverables

Task 1 – Vegetation maps (digital and hard copy), aerial coverages by plant type for each station in a spreadsheet database, and a final project report including interpolation maps of the system.

Task 2 – Spatial-temporal statistical model(s) for assessment of habitat, mercury and water quality indicators in the Everglades ecosystem.

Task 3 – Assistance to USEPA on field and laboratory methods development.
The analytical data with all associated QA/QC requirements specified in the SOW submitted in a timely manner as completed by the laboratory through FTN Associates.

Task 4 – QA/QC data review packages with initial assessments for each of the seven DQO requirements conducted by FTN Associates.
Assistance to FIU and USEPA in data evaluation, interpretation, analysis and integration in preparation for presentation in final reports.

Continue preparation for a comparative ecological risk assessment and initiate early stages of the analysis.

Note: A final project report of Phase II results will not be required until after the wet season survey has been completed and all the 1999 data can be analyzed in conjunction with the 1993-96 data.

QA/QC ATTACHMENT

LABORATORY DOCUMENTATION AND QUALITY CONTROL REQUIREMENTS FOR DATA VALIDATION, Ecological Risk Assessment, Everglades Ecosystem, Phase Two, August 1998. Office of Quality Assurance, Science and Ecosystem Support Division, USEPA Region 4, 980 College Station Road, Athens, Ga 30605-2720

INTRODUCTION

In all environmental projects, it is essential to know the quality of the data used for decision-making purposes. The process of generating data of known quality begins in the planning stages when data quality objectives (DQOs) are established (EPA 1993 and 1994), continues during sample collection and laboratory analysis, is evaluated when validating the analytical data (EPA 1994a, 1994b), and is finalized as part of the data quality assessment process (EPA 1996). The purpose of this document is to identify the specific laboratory quality assurance and documentation requirements that are generally necessary as part of the data validation process.

The quality assurance and documentation requirements described in this document are similar to those defined in recent versions of EPA's contract laboratory program (CLP) inorganic and organic statements of work (SOW) [EPA 1992, 1994c]. However, the requirements are not exclusive to CLP work and will apply whenever EPA Region 4 projects require environmental data which is of known quality and legally defensible. As noted in various parts of this document, it is desirable from the standpoint of permitting rapid review of data, that summary forms, including sample results and quality control information, be in CLP format. **However, other formats are acceptable, provided that all necessary information is included.**

Validation of data requires that appropriate quality assurance and quality control (QA/QC) procedures be followed, and that adequate documentation be included for all data generated both in the laboratory and in the field. Professionals trained in data validation procedures review this information, "flag" data with qualifiers when QA/QC criteria are not met, and prepare the data validation report. The validation reports are then used as sources of data quality indicators, which are used to conduct a data quality assessment relative to the pre-established DQOs.

The QA/QC documentation provided by any laboratory, in conjunction with the sample results, allows for the evaluation of the following indicators of data quality:

- Integrity and stability of the samples;
- Instrument performance during sample analysis;

- Possibility of sample contamination;
- Identification and quantitation of analytes;
- Analytical precision; and
- Analytical accuracy.

The general laboratory documentation requirements discussed in this document are formatted into two (2) sections, pertaining to general quality assurance requirements (1.0) and specific analytical requirements (2.0).

1.0 GENERAL QUALITY ASSURANCE

- LABORATORY shall follow the quality assurance (QA) requirements described in each QA Project Plan (QAPP) and in this Requirements Document.

1.2 The project may include blind quality control samples. These may consist of blanks and/or spikes. Successful performance on the spike shall be defined as proper identification and quantitation of the target analyte(s) within the established quantitative acceptance windows. Successful performance for the blank shall be defined as no contaminants present that interfere with the analytical integrity of the target analytes.

1.3 LABORATORY shall establish and implement a comprehensive quality assurance (QA) program in order to define the reliability of the analytical results for analyses performed under this package. Such a QA program shall be documented in a written QA Plan.

1.4 LABORATORY's written QA Plan must present the policies, organization, objectives, functional guidelines , and specific QA and QC activities designed to achieve the data quality requirements in the QAPP and in this requirements document. Where applicable, SOPs pertaining to each element listed in this document shall be referenced as part of this QA Plan and provided upon request.

1.5 LABORATORY's written QA Plan shall be approved by SESD Office of Quality Assurance (OQA) prior to the initiation of work. A copy of updates to the laboratory's QA Plan and SOPs must be provided to OQA as soon as revision are made.

1.6 The QA Plan shall include provisions for corrective action when QC exceedances occur. All corrective actions with respect to analytical operations must be documented. Any corrections to instrument raw data or reduced data must be initialed and dated by the laboratory staff making the correction.

1.7 LABORATORY's QA Plan must describe the procedures which **have been** implemented to achieve the following:

- Maintain data integrity, validity and usability.
- Ensure that analytical measurement systems are maintained in an acceptable state of accuracy, stability and reproducibility.
- Detect problems through quality control indicators and establish corrective action procedures which keep all analytical processes reliable.

- Document all aspect of the measurement process in order to provide data which are technically sound and legally defensible.

1.8 LABORATORY's QA Plan must address the following elements:

A. Organization and Personnel

1. QA Policy and Objectives

2. QA Management

   a. Organizational chart
   b. Assignment of QA and QC Responsibilities
   c. Reporting Relationship Between QA and Management
   d. QA Document Control Procedures
   e. QA Program Assessment Procedures

3. Personnel

   a. Resumes
   b. Education and Experience
   c. Training Goals

B. Facilities and Equipment

1. Instrumentation and Backup Alternatives
2. Maintenance Activities and Schedules

C. Document Control

1. Laboratory Notebook Policy
2. Sample Tracking/Custody Procedures
3. Logbook Maintenance and Archiving Procedures
4. Project File Organization, Preparation and Review Process
5. Procedures for Preparation, Review, Revision and Distribution of SOPs
6. Process for Revision of Technical or Documentation Procedures

D. Analytical Methodology

1. Receipt and Review of Analysis Request
2. Calibration Procedure and Frequency
3. Sample Preparation/Extraction Procedures
4. Sample Analysis Procedures
5. Standards Preparation Procedures
6. Decision Processes, Procedures, and Responsibility for Initiation of Corrective Action

E. Data Generation

1. Data Collection Procedures
2. Data Reduction Procedures
3. Data Validation Procedures
4. Data Reporting and Authorization Procedures

F. Quality Control

1. Solvent, Reagent, and Adsorbent Check Analysis

2. Reference Material Analysis
3. Internal Quality Control Checks
4. Determination of QC Acceptance Limit Procedures
5. Determination of Corrective Action Procedures
6. Responsibility Designation

G. Quality Assurance

1. Data Quality Assurance
2. Systems/Internal Audits
3. Performance/External Audits
4. Corrective Action Procedures
5. Quality Assurance Reporting Procedures
6. Responsibility Designation

1.9 LABORATORY shall provide reports and other deliverables as specified in this document. In addition, the laboratory shall follow document control procedures. The goal of the laboratory document control program is to assure that all documents for a specified project will be accounted for when the project is complete. Accountable documents used by LABORATORY shall include, but are not limited to, logbooks, chain-of-custody records, sample work sheets, sample run logs, instrument raw data, bench sheets, sample preparation records and other documents relating to the sample analysis.

1.10 All <u>original</u> documentation not provided to EPA with the data package related to the preparation and analysis of the samples shall be kept on file for a minimum of five years. If at the end of the five year period, the LABORATORY desires to dispose of the original documents, the LABORATORY should first contact the EPA quality assurance officer for permission to dispose of the documents. If directed by the EPA contract officer, the laboratory shall ship all project documents to EPA rather than disposing of the documents.

2.0 INORGANIC ANALYSES

2.1 Documentation

The data package submitted for EPA data validation will consist of five (5) sections:

A. Narrative;

B. Chain-of-Custody documentation;

C. Summary of results for environmental samples (including quantitation limits);

D. Summary of QA/QC results; and

E. Raw data.

2.2 Narrative (Section A)

The narrative will be written on laboratory letterhead and the release of data will be authorized by the laboratory manager or his/her designee. The Narrative will consist of the following information:

- EPA's sample identification and the corresponding laboratory identification;
- Parameters analyzed for each sample and the methodology used; when applicable, cite EPA method numbers;
- Whether the holding times were met or exceeded;
- Detailed description of all problems encountered;
- Discussion of possible reasons for any QA/QC sample results outside acceptance limits; and
- Observations regarding any occurrences which may affect sample integrity or data quality.

2.3 Chain-of-Custody Documentation (Section B)

Legible copies of Chain-of-Custody forms for each sample shall be submitted in the data package. The date of receipt and the observed sample condition at the time of receipt must be described on the Chain-of-Custody form.

2.5 Summary of Environmental Results (Section C)

The following information is to be included in the summary of results for each environmental sample. The summary should follow the CLP format if possible, but other formats are acceptable provided that all necessary information is included.

- Form name;
- Client's sample identification and the corresponding laboratory identification;
- Sample collection date;

- Sample matrix;
- Date of sample digestion and quantity of sample subjected to digestion, as applicable;
- Date and time of analysis;
- Identification of the instrument used for analysis;
- Instrument specifications;
- Weight or volume of sample used for analysis/digestion;
- Dilution or concentration factor for the samples;
- Percentage of moisture in the soil samples;
- Instrument detection limits (IDL) or method detection limits (MDL);
- Analytical results; and
- Definitions for any data qualifiers used.

2.6 Summary of QA/QC Requirements and Results (Section D)

The following QA/QC sample results must be presented on summary forms to facilitate data validation and data quality assessment activities. These summaries should follow the CLP format, if possible. Other formats are acceptable provided that all necessary information is included and the summary is easy to follow.

2.6.1 **Instrument Calibration** (CLP Form II equivalent)

- For instruments using external calibration standards, the calibration curves must consist of a least three standard, in addition to a zero standard, and have a linear correlation coefficient greater than 0.995. Instruments must be fully calibrated each day of use, unless the analytical method expressly permits verification of the initial calibration with fewer standards. The order for reporting of calibrations for each analyte must follow the chronological order in which the standards were analyzed.

  Initial Calibration Verification

  The initial calibration must be verified each time EPA samples are analyzed. The initial calibration verification standard should be a standard reference material from the National Institute of Standards and Technology (or secondary standards traceable thereto), or from sources which attest to the authenticity and concentration of the standard solutions. Report the concentration for the true value, the concentration found, the percent recovery, and the control limits for each parameter analyzed. The date and time of analysis must also be reported.

  Continuing Calibration Verification

Analyze a continuing calibration verification (CCV) standard after a maximum of 20 EPA sample to demonstrate that the system is maintaining calibration. Report the source for the continuing calibration standards which may be the same as the initial calibration standards. Report the concentration for the true value, the concentration found, the percent recovery, and the control limits for each element analyzed. The date and time analysis must also be reported.

Sensitivity Verification Standard

Analyze and report results for a low-level standard which is 3-5 times the laboratory's method detection limit to verify instrument sensitivity (that the reported detection limits can be achieved) in the manner described for continuing calibration verification. This standard may be analyzed immediately after calibration verification if desired.

2.6.2 Method Blank Analysis (CLP Form III equivalent)

Prepare and analyze a method blank with each batch of samples which is prepared ans analyzed. Report analyte concentrations found in the method blank. The method blank must be prepared and analyzed in the exact same manner as samples and include all reagents used in sample preparation and analysis. The date and time of analysis must also be reported.

2.6.3 Method Detection Limit

Analyze and report the method detection limit according to the procedure found in 40CFR Part 136, Appendix B. This should be done at least yearly or whenever instrument operating conditions or instruments are changed. Supporting data for the MDL is required to be reported only each time the MDL is determined, not with each data package.

2.6.4 Precision and Accuracy

- Matrix spike (MS) analysis (CLP Form V equivalent)

Analyze an MS at a frequency of 1 per 20 EPA samples. Report the concentration of the spiked sample result, the sample result and the quantity of spiking solution added to the spike for each analyte. Calculate and report the percent recovery and list the control limits.

- Matrix Duplicate Analysis

Analyze a Matrix Duplicate at a frequency of 1 per 20 analytical EPA samples. Report the original concentration, duplicate concentration and relative percent difference (RPD). List the control limits.

2.6.5 Other QC Criteria

- All QC samples, including the method blank, spikes and duplicates, and standards should be prepared and digested in the same manner as the samples.

- DORM/Oyster SRM should be analyzed with the fish samples and a soil SRM must be analyzed with the soil and periphyton samples to demonstrate the accuracy of the method.

- Accuracy and precision of the QC data must meet the control limits specified in the laboratory’s EPA approved QA Plan.

- An example calculation showing how the final result was obtained for each analyte must be included in each data package. The calculations performed by the laboratory must include all information needed by a third party data reviewer to reconstruct the final reported result.

2.7 Raw data (Section E)

This section shall include **legible copies** of the raw data for the following:

- Environmental sample results (arranged in increasing client's sample number order);

- Instrument calibrations; and

- QC sample analysis data.

The raw data for each analysis shall include the following:

- Measurement print-outs and quantitation reports for each instrument used;

- Absorbance, titrimetric, or other measurements for wet chemical analysis;

- Sample preparation and digestion logs;

- Instrument analysis logs for each instrument used; and

- Percent moisture in the soil samples (when applicable).

Legible copies of the raw data shall be organized systematically, and each page shall be numbered, and a table of contents must be included in each package. All data should include the analyst name or initials and date of sample preparation and/or analysis.

3.0 REFERENCES

EPA, 1996. Guidance for the Data Quality Assessment Process. EPA QA/G-9. Pre-Publication Copy. February, 1996.

EPA, 1994. Guidance for the Data Quality Objectives Process. EPA QA/G-4. September, 1994.

EPA, 1994a. *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*. EPA-540/R-94-013. PB94-963502. Publication 9240.1-05-01. (February 1994).

EPA, 1994b. *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review*. EPA-540/R-94-012. PB94-963502. Publication 9240.1-05. (February 1994).

EPA, 1994c. *USEPA Contract Laboratory Program Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration.* OLM03.1. EPA-540/R-94-073. PB95-963503. Publication 9240.1-06. (August 1994).

EPA, 1993. Data Quality Objectives Process for Superfund, Interim Final Guidance. EPA/540/G-93/071, Publication 9355.9-01, September, 1993.

EPA, 1992. *USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis, Multi-Media Multi-Concentration.* Document Number ILM03.0 EPA-540/R-94-073. PB95-963503. Publication 9240.1-06. (November 1992).

Attachment 2

Project Data Quality Objectives (DQOs)

South Florida Ecosystem Assessment Project
Decision-Based Data Quality Objectives

Data Quality Objectives

The Data Quality Objectives (DQOs) were prepared generally following the Guidance for the Data Quality Objectives Process EPA QA/G-4 (EPA 1994). This US Environmental Protection Agency (EPA) Guidance document, however, is not entirely appropriate for research projects. The EPA Quality Assurance Management Staff are in the process of preparing DQO guidance for research projects, but this guidance is not currently available. The South Florida Ecosystem Assessment Project is a research project that, in part, is developing risk-based criteria for decisions because the existing criteria are not appropriate or no criteria exist. Therefore, two separate, but complementary, approaches were used to develop DQOs. The first approach was to use the EPA QA/G-4 documentation as guidance in developing decision-based DQOs, which are discussed in this document. This document uses the EPA QA/6-4 report format. The second approach revised the DQOs originally proposed in the REMAP Research Plan (Stober et al. 1993). These revised DQOs are listed in Appendix A.

Background

In 1989, a Florida panther, an endangered species, died because of mercury toxicosis. Since then, over 2 million acres in South Florida have been placed under fish consumption advisories because of mercury contamination. The EPA Region 4 Science and Ecosystem Support Division (SESD), therefore, was charged by the EPA Regional Administrator to develop an action plan to evaluate the mercury issue and provide a scientific basis for evaluating options and strategies to eliminate mercury contamination in the South Florida Everglades Ecosystem. Subsequently, the Region 4 SESD prepared a research plan, had this plan peer-reviewed, and initiated the study as a Regional Environmental Monitoring and Assessment Program (REMAP) Project. As the Project planning and pilot Project proceeded, it became obvious that the environmental issues in South Florida (eutrophication, mercury contamination habitat alteration, hydroperiod modification) are highly interactive and need to be addressed through an integrated monitoring and assessment program. Therefore, the REMAP Project was expanded to become the South Florida Ecosystem Assessment Project addressing these multiple environmental issues. The variables being measured in this Project will permit answers to questions on these multiple environmental issues. A central goal of the Project, however, remains to answer assessment questions related to the magnitude, extent, trends, and transformation processes in mercury contamination of the South Florida Everglades Ecosystem.

State the Problem - a description of the problem(s) and specification of available resources and relevant deadlines for the study.

(1) *Identify the members of the team* - The team consists of the Region 4 Project Manager,

SESD; Assistant Project Manager, Water Division; Quality Assurance Officer; Southeast Environmental Research Program manager, Florida International University; and systems ecologist and QA support, FTN Associates, Ltd.

(2) *Identify the primary decision maker(s)* - The primary decision maker is the South Florida Ecosystem Assessment Project Manager. Other decision makers include the Assistant Project Manager, Division Directors for the Water Division and Science and Ecosystem Support Division.

(3) *Develop a concise description of the problem* - Mercury contamination, nutrient loading, hydropattern modification, and habitat alteration are impacting fish and wildlife in the South Florida Everglades Ecosystem. The sources, causes, and interactions among many of these environmental stressors are unknown. Environmentally-sound, cost-effective restoration of the South Florida Everglades Ecosystem, however, depends on identifying these sources, causes and interactions. Almost one billion dollars are estimated to be spent on this restoration effort.

(4) *Specify the available resources and relevant deadlines for the study* - Approximately $1 million dollars/year are needed to determine the magnitude, extent, trends and possible causes of the mercury contamination, eutrophication, hydropattern modification and habitat alteration problems. This represents less than 0.1% of the proposed restoration expenditures. The relevant regulatory deadlines are listed in Table 1. These regulatory deadlines extend through 2004, with a major milestone in 1999 when the EPA mercury report is due to the South Florida Ecosystem Restoration Task Force.

Identify the Decision - a statement of the decision that will use environmental data and the actions that could result from this decision.

(1) *Identify the principal study questions* - The principal study questions were identified as part of the original proposal and specification of the DQOs. These seven policy-relevant questions are listed in Table 2.

(2) *Identify alternative actions that could result from resolution of the principal study questions* - The logical alternative actions and pathways that could result in answering these seven questions were identified during the initial phases of the Project. These pathways were incorporated into a Visual Basic computer program to show the logical development of these alternative actions. The expanded logic pathways from this computer program are shown in Figure 1. These logic pathways and alternative action formulations are a major part of the Problem Formulation phase of the Ecological Risk

Table 1. Mercury Related Legislative and Regulatory Deadlines.

Date	Federal	Florida
1995	NPDES Permit for the ENR project (CWA)	
1996	EIS for the Everglades Construction Project (NEPA)	
1996	404 Permit for the Everglades Construction Project (CWA)	
Oct 1997	404 Permit for STA-6 (CWA)	STA-6 NPDES Permit and 402 Certification
Sep 1998	USACOE Central & Southern Florida Project Restudy Plan Draft Report & Draft EIS (WRDA, NEPA)	
Dec 1998		Evaluation of water quality standards for the Everglades Protection Area & EAA canals (EFA)
Jan 1999	STA-1W, 2, & 5 404 Permits (CWA)	STA-1W, 2, & 5 NPDES Permits, 402 Certification (CWA)
Jul 1999	Final Restudy Report and EIS due to Congress (WRDA, NEPA)	
Dec 1999		Report to Governor and Legislature on status of EPA mercury study (EFA)
Dec 2001		Phosphorus criterion rulemaking for Everglades Protection Area and EAA canals (EFA)
Oct 2003	STA-3 & 4 404 Permits (CWA)	STA-3 & 4 NPDES Permits and 404 Certification (CWA)
Dec 2003		Revised water quality standards for the Everglades Protection Area & EAA canals (EFA)
2004	Approval of water quality standards for the Everglades Protection Area & EAA canals (CWA)	

WRDA: Federal Water Resources Development Act
EFA: Florida Everglades Forever Act
CWA: Federal Clean Water Act
STA: Stormwater Treatment Area
EAA: Everglades Agricultural Area
NEPA: Federal National Environmental Policy Act

Table 2. Policy-Relevant Questions Guiding the Project.

Status and Trends
1) What is the magnitude of the mercury problem? What are the current levels of mercury contamination in various species? What ecological resources of interest are being adversely impacted by mercury? 2) What is the extent of the mercury problem? (i.e., what is the geographic distribution of the problem? Is it habitat specific?) 3) Is the problem getting worse, better, or staying the same over time?
Diagnosis and Management
4) What factors are associated with, or contributing to, methylmercury accumulation in sensitive resources? 5) What are the relative contributions and importance of mercury from different sources (e.g., fossil fuel plants, waste incinerators, agricultural management practices, geologic pools, natural peat deposits, global atmospheric background, etc.)? 6) What are the relative risks to different ecological systems and species from mercury contamination? 7) What management alternatives are available to ameliorate or eliminate the mercury contamination problem?

Assessment Framework that forms the foundation of this study. Dichotomous trees were formulated for each of the logic pathways developed during the initial Project phases. These trees were developed prior to the initiation of the field sampling and were used to assist in the formulation of the preliminary project DQOs.

(3) *Combine the principal study questions and the alternative actions into a decision statement* - "Decide how the relative ecological risk from mercury contamination compares with the risks from nutrient additions, hydropattern modification, habitat alteration. Determine if controlling these other stressors will eliminate mercury contamination; if not, determine procedures that can be used to eliminate mercury contamination."

(4) *Organize multiple decisions* - Multi-decision pathways will be based on the outcomes from the logic pathway analyses shown in Figure 1. These logic and decision pathways will be refined as the Project proceeds and new information is collected and analyzed.

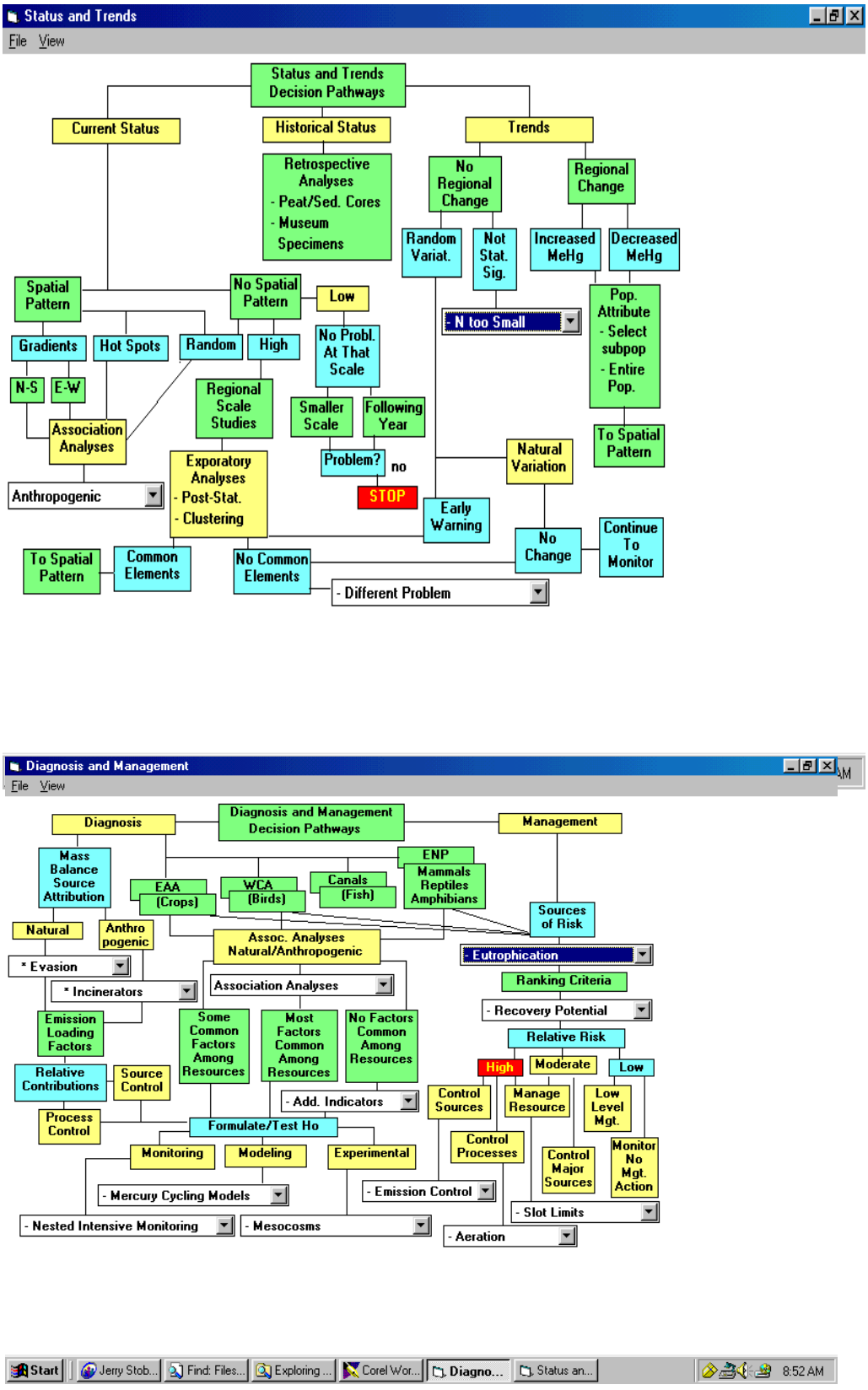


Figure 1. Logic pathways for decisions on Status and Trends and Diagnosis and Management Questions. Pathways diagram information and analyses needed to answer the seven policy-relevant questions.

Identify the Inputs to the Decision - a list of the environmental variables or characteristics that will be measured and other information needed to resolve the decision statement.

(1) *Identify the information that will be required to resolve the decision statement* - The information needed to resolve the decision statements is listed in Table 3.

(2) *Determine the sources for each item of information identified* - The South Florida Ecosystem Assessment Project (SFEA) is the primary source of the information needed to address the decision statements. The decision statements can not be resolved without this Project. Additional sources of information also are identified in Table 3.

(3) *Identify the information that is needed to establish the action level* - The criteria that will be used to establish the action level will be:

(a) Variability - ecological effects significantly different from natural variability
(b) Endpoints - reproduction, feeding efficiency, behavioral changes, and other ecologically relevant processes, in addition to toxicity
(c) Temporal scale - chronic versus acute effects
(d) Spatial scale - small versus large scale effects

For most constituents, regulatory criteria or standards do not exist. The decision will be made using risk-based action levels.

(4) *Confirm that appropriate measurement methods exist to provide the necessary data* - For conventional pollutants, EPA approved methods are being used to measure environmental variables with an approved QAPP. For some constituents, such as total phosphorus, existing EPA methods do not have the resolution needed to detect low-level background concentrations. For other constituents, such as methylmercury in water, soil, and sediment, there are no approved measurement methods. Therefore, experimental measurement methods are being developed for these constituents, with confirmatory analyses being conducted by independent laboratories.

Define the Boundaries of the Study - a detailed description of the spatial and temporal boundaries of the problem, characteristics that define the population of interest, and any practical considerations for the study.

(1) *Specify the characteristics that define the population of interest* - The target population or population of interest are all ecological resources in the South Florida study area. This includes the freshwater wetlands, open water and canals found in the Everglades National Park (ENP), Water Conservation Areas (WCAs), Big Cypress National Preserve (BiCY), and Everglades Agricultural Areas (EAA). The media to be sampled include, sediment, water, and biota. The emphasis is on mercury concentrations in biota, especially fish tissue. However, one of the desired outcomes of the Project is better estimates of

Table 3A. Information Needs, Source and Method.

Measurement Variable	Source	Method
Physical Measurements		
Site location	SFEA	Global Positioning System
Weather	SFEA, NOAA	Visual observation, meteorological stations
Discharge, structure	SFWMD	Gage readings, pump capacity
Water depth	SFEA, SFWMD	Calibrated line, depth recorders
Temperature	SFEA	Thermistor
Peat depth	SFEA	Calibrated probe
Turbidity	SFEA	Turbidimeter
Bulk density	SFEA	Balance, weighing
% Mineral content	SFEA	Combustion furnace
Ash free dry weight	SFEA	Combustion Furnace
Chemical Measurements		
Dissolved oxygen	SFEA	DO probe
Specific conductance	SFEA	Conductivity meter
pH	SFEA	pH meter
Total organic carbon	SFEA	Total carbon analyzer
Total phosphorus	SFEA	Laboratory Analysis
Sulfate	SFEA	Laboratory Analysis
Total mercury	SFEA	New method development
Methymercury	SFEA	New method development
Alkaline phosphatase	SFEA	New method development
Redox potential	SFEA	Volt meter
Total phosphorus	SFEA	Laboratory Analysis
Total Nitrogen	SFEA	New method development
Ammonium-N	SFEA	Laboratory Analysis
Nitrite-N	SFEA	Laboratory Analysis
Nitrate-N	SFEA	Laboratory Analysis
SRP	SFEA	Laboratory Analysis
Total Organic Carbon	SFEA	Laboratory Analysis
Sulfide	SFEA	Laboratory Analysis
Chlorophyll a	SFEA	Laboratory Analysis
Biological Measurements		
Resource class	SFEA	Visual inspection
Periphyton presence/absence	SFEA	Visual observation
Chlorophyll a	SFEA	Laboratory Analysis
Soil/Sediment total mercury	SFEA	New method development
Soil/Sediment methylmercury	SFEA	New method development
Fish total mercury	SFEA	New method development

Table 3B. Other Information Needs and Sources.

Information Needs	Sources
Water management operation records	SFWMD, COE
Atmospheric mercury deposition/evasion	FL DEP, EPA, FAMS, SFWMD, UFL, FSU
Nutrient loading estimates	SFWMD
Habitat changes	FWS NWI, NPS
Simulated natural hydropatterns	SFWMD
Vegetation patterns and production	NPS, FWS, SFWMD
ENR Project results	SFWMD, FL DEP
Periphyton production - nutrient relationships	SFWMD, FL DEP, FIU, UWI
Organic carbon speciation	USGS
Sulfate reduction/loading	SFWMD, USGS, FIU, UWI
Mercury methylation/demethylation	USGS, SFWMD, UMD, FIU, UFL, UWI
Fish and invertebrate impacts	FWS, NPS, FIU, UFL
Wading bird impact	FWS, NPS, UFL
Large mammal and reptile impacts	FWS, NPS, FIU, UFL, FSU, UGA

the type and proportion of ecological resources and the impacts of other stressors on these resources in South Florida.

(2) *Define the spatial boundary of the decision statement*

(a) *Define the geographic area to which the decision statement applies.* The geographic area being studied, and for which decisions apply, is approximately 160 km long and 60 km wide, resulting in an area of about 9600 km^2. The exact boundaries are listed in Table 4 below and shown in Figure 2.

Table 4. Geographic Area Boundaries.

Boundary	Description
Northern	West from Canal L8 to its junction with Lake Okeechobee and across to the Caloosachatchee River.
Western	Vertical line from the intersection of the Caloosahatchee River and Highway 833 south to the coast (the mangrove region is excluded from the target population).
Southern	Edge of the western mangrove east to the intersection with Highway US 1.
Eastern	Highway US 1 north to its intersection with Highway 27, then along the eastern boundaries of Water Conservation Areas to the Intersection with Canal L8.

(b) *When appropriate, divide the population into strata that have relatively homogeneous characteristics.* Strata of interest were based on the decision statement, rather than on homogeneity of variance. For example, there was less interest in defining the characteristics of the Big Cypress National Preserve (BiCY) than in other designated geographic areas. Therefore, BiCY was sampled with a lower inclusion probability (approximately 1/3 the density of other areas within the study boundaries). In addition, subsequent analyses have indicated the areas north of Alligator Alley, between Alligator Alley and Tamiami Trail, and south of Tamiami Trail have attributes that can influence management and policy decisions.

(3) *Define the temporal boundary*

(a) *Determine the timeframe to which the decision statement applies.* The decision statement applies from the time of the first data collection in April 1994 until at least 2004. The mercury-related legislative and regulatory deadlines are defined in Table 1. However, Project results are applicable to a longer timeframe because the South Florida Ecosystem Restoration Task Force has legislative mandates for hydropattern modification, habitat alteration and eutrophication deadlines beyond 2004 that can be addressed with results from this Project.

(b) *Determine when to collect the data*. Because time and space scales are inexorably coupled, the synoptic sampling approach spatially dictates that the temporal sampling frequency be seasonal. There are two distinct hydrologic seasons in Florida. The dry season extends from November to April and the wet season extends from June until September. May and October are transitional months. Sampling during only one season could result in biased and flawed decisions on

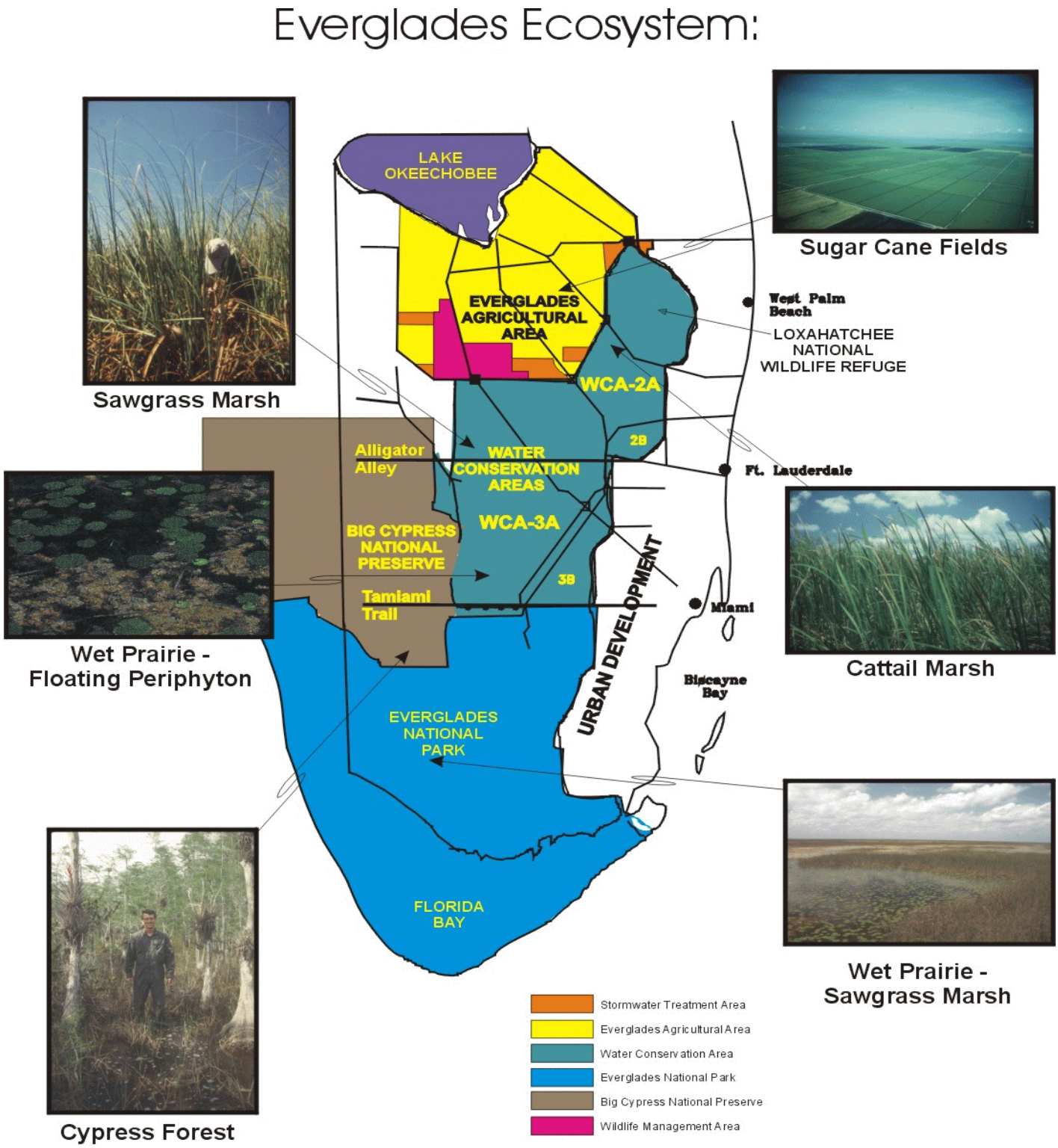


Figure 4. Everglades ecosystem communities.

management or regulatory issues, because of seasonal variability. Sampling, therefore, needs to be done during both the dry and wet season. Decisions will be made over the next decade, based, in part, on spatial and temporal trends in information. These trends can not be defensibly determined with only one set (wet and dry season) of data at the beginning and end of the decision time frame. Two reference periods define change, not trends. Power analyses will be conducted to determine the number of sampling intervals needed to detect statistically defensible trends and contribute to the decision process.

(4) *Define the scale of decision making* - Decisions on mercury management and restoration issues must be made for the entire South Florida ecosystem. The environmental issues arose because of small-scale, piecemeal approaches to managing the system.

(5) *Identify practical constraints on data collection* - The large geographic area for sampling, and the need to collect synoptic samples requires that sampling be conducted by multiple teams using helicopters and airboats. The sampling period should be no longer than 10 days to minimize large scale changes in meteorology affecting water depth and quality measurements. The number of samples and sample volume need to be minimized to reduce weight and time for collection, but with sufficient volume to permit precision and accuracy requirements to be achieved. Clean sampling procedures are required for the mercury analyses, both in the field and in the laboratory. Low concentration nutrient analyses also are required because of the ultraoligotrophic condition of the Everglades wetlands.

Develop a Decision Rule - to define the parameter of interest, specify the action level and integrate previous DQO outputs into a single statement that describes a logical basis for choosing among alternative actions.

[NOTE: This DQO guidance statement is not compatible with the South Florida Ecosystem Restoration goals and objectives. The issues in South Florida are not independent; they are highly interactive. Multi-media decisions are required for multiple issues. There is no single statement can be formulated that will permit decisions among alternative actions. The greatest threat to the Everglades ecosystem is to assume these issues are independent and derive one single statement to address all issues. The Project, in part, will determine what the criteria should be for multiple issues such as phosphorus loading, water depth, distribution and timing, methylmercury concentrations in multi-media, and habitat types.**]**

(1) *Specify the statistical parameter that characterizes the population of interest* - REMAP is an exploratory research program so no single statistical parameter has been selected to characterize the population of interest. In addition, the emphasis is not on one single constituent, such as a hazardous material that might exceed a regulatory standard. Rather,

several statistical parameters are needed to characterize different population attributes, including:

(a) mean concentrations of selected constituents (see Table 3 for constituents)
(b) cumulative distributions of constituents, by season, by area
(c) distributional differences among constituents
(d) spatial patterns of constituents, and
(e) spatial/temporal associations among constituents.

(2) *Specify the action level(s) for the study* - Three action levels currently exist:

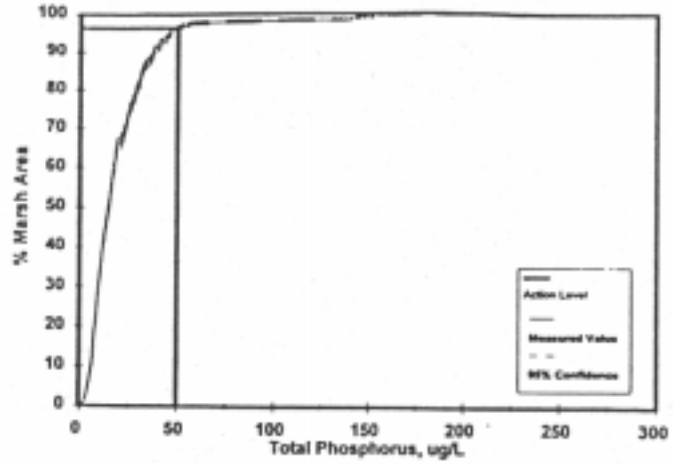
(a) Phase I control target for total phosphorus of 50 μg/L (ppb);
(b) Water total mercury criterion for protection of aquatic life of 12 ng/L (ppt); and
(c) Proposed predator protection level for mercury of 100 μg/kg (ppb) for prey species.

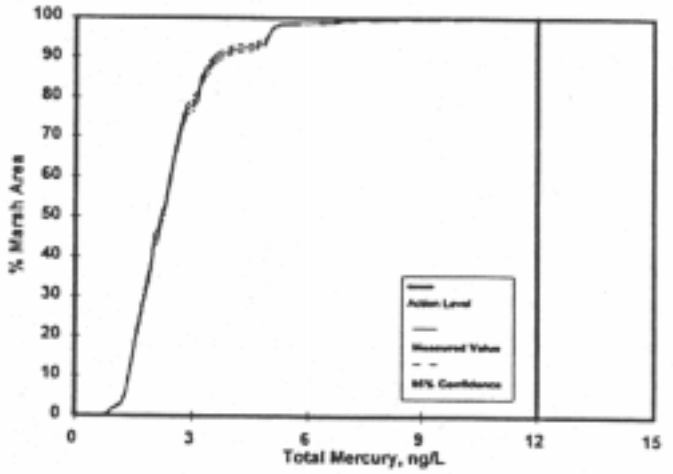
All three of these levels are underprotective. New risk-based action levels need to be determined. Currently, 95% of the marsh has total phosphorus concentrations less than 50 ppb; 100% of the marsh has total mercury concentrations less than 12 ppt, and 68% of the marsh has prey fish species with mercury concentrations greater than 100 ppb (Figure 3). Developing appropriate risk-based action levels for total phosphorus and mercury is one of the objectives of this Project. The detection and minimum quantitation limits for all three of these constituents are less than the respective criterion. Because risk-based action levels are needed, methods with increased sensitivity have been developed and are being tested.

(3) *Develop a decision rule (an "if...then" statement)* - Decision rules express what the decision maker ideally would like to resolve. The decision has been made that revised criterion are needed, based on the information developed to date from the Project. Preliminary decision rules, given this need, are listed in Table 5. Subsequent revisions of the DQO document will expand and refine these decision rules as additional information becomes available. Logic flow paths have been formulated (Figure 1) to increase the probability future information will improve the efficacy of the decision rules.

Specify Tolerable Limits on Decision Errors - the decision maker's tolerable decision error rates based on a consideration of the consequences of making a decision error.

(1) *Determine the possible range of the parameter(s) of interest* - The possible range of the parameters of interest are listed in Table 6. These ranges are based on this Project and other studies conducted in the South Florida Everglades ecosystem.





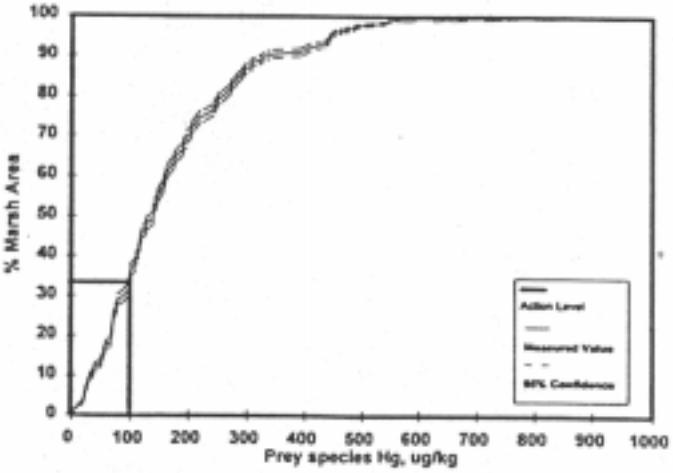


Figure 3. Distribution of total phosphors and total mercury in water and total mercury in a fish prey species, with 95% confidence intervals. Note: The existing action levels are under protective because ecological effects are observed from eutrophication and mercury contamination.

Table 5. Preliminary Decision Rules for South Florida.

	Decision Rules
1a	If the South Florida Everglades Ecosystem can not be managed to achieve all desired ecological uses, then a comparative ecological risk assessment shall be conducted to determine which stressors, and their interactions, are placing the system at greatest risk.
1b	If the South Florida Everglades Ecosystem can be managed to achieve all desired ecological uses, then the management, regulatory and control practices shall be maintained.
	Based on the results of this comparative risk assessment, the following decision rules might be used:
2a	If phosphorus concentrations exceed a risk-based action level, then nutrient loads will be reduced until phosphorus concentrations are less than the action level.
2b	If phosphorus concentrations are less than a risk-based action level, then BMPs and other nutrient control programs will be maintained.
3a	If hydropattern modification varies by more than 10% from the desired natural hydropattern rule curve, then the hydropattern will be modified to match the desired natural hydropattern rule curve while maintaining flood control and water supply.
3b	If the hydropattern modification is within 10% of the desired natural hydropattern rule curve, and flood control and water supply purposes are satisfied, then the hydropattern management and operational programs will be maintained.
4a	If hydropattern modification varies by more than 10% from the desired natural rule curve and either flood control and/or water supply requirements can not be satisfied, then alternative flood control and water supply options will be investigated to return the hydropattern to within 10% of the desired natural rule curve.
4b	If hydropattern modification can not be returned to within 10% desired natural rule curve and achieve water supply and/or flood control demands, then a risk-based benefit/cost analysis will be performed to determine which alternatives have the lowest benefit/cost ratio and that use eliminated.
5a	If habitat alteration exceeds risk-based landscape action level metrics, then habitat alternation, a benefit/cost analysis will be done to determine if this habitat alteration including urban development or agricultural production, should be banned and habitat restoration under taken.
5b	If habitat alteration is less than risk-based landscape action level metrics, then habitat alteration will be permitted until these values are within 5% of the lower limit of the action level.

Table 5. (Continued).

Decision Rules	
6a	If mercury concentrations exceed a risk-based action level, then mercury sources will be controlled until mercury concentrations are less than this risk-based level.
6b	If mercury concentrations are less than the risk-based action level, mercury sources will be controlled to ensure the action level is not exceeded.
7a	If hydropattern modification greater than 10% from the risk-based desired natural rule curve aggravates mercury contamination of fish and wildlife, then the hydroperiod shall be modified to achieve the risk-based action level.
7b	If the hydropattern modification less than 10% of the risk-based desired natural rule curve aggravates mercury contamination of fish and wildlife, then a comparative risk assessment and risk-based benefit/cost analysis shall be conducted to determine which stressor places that system at greatest risk and has the lowest benefit/cost ratio; that stressor then will be reduced.
8a	If nutrient loading exceeds the nutrient risk-based action level and aggravates mercury contamination of fish and wildlife, then nutrient loading shall be reduced to achieve the risk-based action level.
8b	If nutrient loading is less than the nutrient risk-based action level and aggravates mercury contamination of fish and wildlife, then a comparative risk assessment and risk-based benefit/cost analysis shall be conducted to determine which stressor places that system at greatest risk and has the lowest benefit/cost ratio; that stressor then will be reduced.
9a	If habitat alteration exceeds risk-based landscape action level metrics and aggravates mercury contamination of fish and wildlife, then additional habitat alteration shall be banned and habitat restoration under taken.
9b	If habitat alteration is within the risk-based landscape action level metrics and aggravates mercury contamination of fish and wildlife, then a comparative risk assessment and risk-based benefit/cost analysis shall be conducted to determine which stressor places that system at greatest risk and has the lowest benefit/cost ratio; that stressor then will be reduced.

Table 6A. Water Constituents Ranges in South Florida.

Measurement Variable	Range			
	Minimum		Maximum	
Physical Measurements				
Site location (deg.)	Latitude 25.30	Longitude 80.22	Latitude 26.93	Longitude 81.13
Water depth (m)	0.5		6	
Temperature (°C)	18		36	
Turbidity (NTU)	0.1	80	180	61
Chemical Measurements				
Dissolved oxygen (mg/L)	0		15	
Specific conductance (μS)	10		2150	
pH (s.u.)	5.5		8.8	
Total organic carbon (mg/L)	5		80	
Total phosphorus (mg/L)	0.001		0.500	
Sulfate (mg/L)	1.0		850	
Total mercury (ng/L)	0.02		12	
Methymercury (ng/L)	0.03		1.5	
Alkaline phosphatase	0.01		8.0	
Biological Measurements				
Resource class *(canal, sawgrass marsh, cattails, etc.)* (Numeric rank)	1		7	
Periphyton presence/absence (1,0)	0		1	
Chlorophyll a (μg/L)	0		100	
Periphyton total mercury (μg/kg)	4		600	
Periphyton methylmercury (μg/kg)	0.08		25	
Fish total mercury (μg/kg)	5.0		1000	

Table 6B. Soil/Sediment Constituents Ranges in South Florida.

Measurement Variable	Range	
	Minimum	Maximum
Physical Measurements		
Peat depth (m)	0	>4.25
Bulk density (g/cc)	0.05	1.4
% Mineral content (%)	3%	99%
Ash free dry weight (%)	1.0	96.0
Redox potential (mV)	-250	+600
Chemical Measurements		
Soil/Sediment total mercury (μg/kg)	3.0	500
Soil/Sediment methylmercury (μg/kg)	0.01	50
Soil/Sediment total phosphorous (μg/kg)	10	9000
Soil/Sediment sulfate (μg/kg)	20	850

(2) *Identify the decision errors and choose the null hypotheses*

(a) *Define both types of decision errors and establish the true state of nature for each decision error.* By convention, a Type I (false positive) error is rejecting the null hypothesis when it is true. A Type II (false negative) error is not rejecting the null hypothesis when it is false. The two types of decision errors for the Project are (I) deciding the risk-based action level is exceeded when it truly is not, and (II) deciding the risk-based action level is not exceeded when it truly is.

The true state of nature for decision error (I) is that the null hypothesis is true.

The true state of nature for decision error (II) is that the null hypothesis is false.

(b) *Specify and evaluate the potential consequences of each decision error.* The consequences of deciding the risk-based action levels are exceeded when they truly are not (decision error I) means there will be increased control costs associated with nutrient and mercury source reduction, restricted urban and agricultural development, habitat restoration, and restricted hydropattern modification around the natural hydropattern rule curve, which could result in flood damage or water supply shortages.

The consequences of deciding the risk-based action levels are not exceeded when they truly are (decision error II) means that ecological restoration of the South Florida Everglades ecosystem will not be successful.

(c) *Establish which decision error has more severe consequences near the action level.* Based on current laws and regulations related to the South Florida Everglades ecosystem (e.g., Everglades Forever Act), the decision II error has the more severe consequences near the action level because of the risk to both ecological and human health and ecological restoration. However, this consequence must be based on a comparative risk assessment and a risk-based benefit/cost analysis of the risks and impacts. The economic consequences are in the billion dollar category for both types of decision errors.

(d) *Define the null hypothesis (baseline condition) and the alternative hypothesis and assign the terms "false positive" and "false negative" to the appropriate decision error*. Null hypotheses for DOQs are not equivalent to experimental null hypotheses for statistical testing. Null hypotheses for DQOs reflect the decision error that has the most adverse potential consequences. The DQO null hypothesis is equal to the true state of nature that exists when the more severe decision error occurs. The null hypotheses for this Project, therefore, would be:

H_o = The comparative ecological risk assessment indicates the interactions among stressors puts the South Florida Everglades ecosystem at risk.
H_o = The risk-based action levels for nutrient concentrations are exceeded.
H_o = The risk-based action levels for mercury concentrations are exceeded.
H_o = The risk-based landscape action level metrics are exceeded.
H_o = The risk-based action levels for hydropattern modification exceed by X% the natural hydropattern rule curve.

A "false positive" has the greatest consequences for each of these hypotheses.

(3) *Specify a range of possible values of the parameter of interest where the consequences of decision errors are relatively minor (gray region)* - The purpose of this research project is to determine the action level values. Until these action levels are defined, it is not possible to specify actual numeric values to an area of minor importance. It is, however, possible to indicate these areas of minor importance will be at the extremes of the distribution. In this portion of the action level curve, there will be a low probability of making either type of decision error.

(4) *Assign probability values to points above and below the action level that reflect the tolerable probability for the occurrence of decision errors.* - The QA G-4 Guidance manual indicates the gray region where greater tolerable errors are permitted are around the action level, with lower tolerable errors around the extreme values. The planning team disagrees with this concept. The greater tolerable errors are permitted at the extemes of the distribution because it is unlikely that large errors in the metric would alter the conclusion that the action level was either exceeded or not exceeded. However, near the action level, particularly as values approach the lower limit of the action level, decision errors can have significant consequences on subsequent actions (Figure 4). Tolerable error around the action level in this region should be no more than 10%.

Optimize the Design - The REMAP monitoring design for South Florida was revised to provide more resource-effective information at reduced cost without compromising the DQOs for the marsh samples.

Appendix A contains statements for data representativeness, completeness, comparability, precision and accuracy for each of the constituents measured in the EPA Region 4 South Florida Ecosystem Assessment Program. These quantitative DQO criteria will be revised as additional data become available to the program.

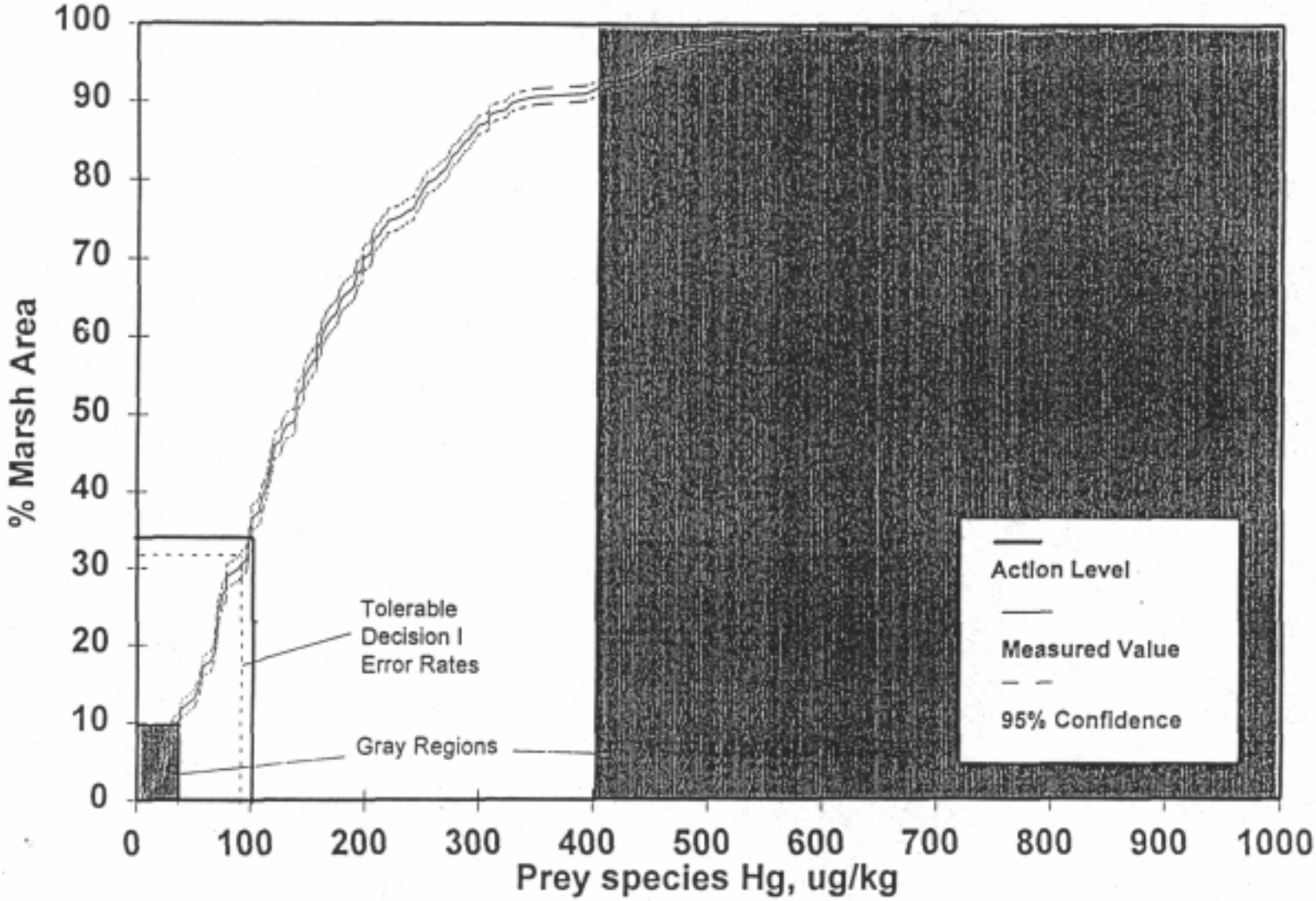


Figure 4. Proposed mercury Action Level for predator prey species identifying The Gray Regions and area for 10% Tolerable Decision 1 Error Rates.

Attachment A

Data Quality Objective Criteria

Table A1A. Data Quality Objective Criteria.

Measurement Variable	Representativeness	Completeness	Comparability (Split Samples SOPs, Std. Units)	Precision RPD [1] (Colocated Samples)	Precision RPD [1] (Lab Duplicates "split" samples)	Accuracy (% Spike Recovery in SRM, blank spikes, PE)
SURFACE WATER						
Dissolved Oxygen	Design-based statistically representative	90%	SOPs	NA	NA	±0.2*
pH	"	"	SOPs	NA	NA	±0.2*
Temperature	"	"	SOPs	NA	NA	±0.15*
Conductance	"	"	SOPs	NA	NA	±1*
Redox	"	"	SOPs	NA	NA	SRC
Depth	"	"	SOPs	NA	NA	SRC
Turbidity	"	"	SOPs	136	<20%	SRC
Total Phosphorus	"	"	SOPs, SFWMD	64	<20%	75-125
Total Nitrogen	"	"	SOPs, SFWMD	45	<20%	75-125
Ammonium-N	"	"	SOPs, SFWMD	--	<20%	75-125
Nitrite-N	"	"	SOPs, SFWMD	--	<20%	75-125
Nitrate-N	"	"	SOPs, SFWMD	--	<20%	75-125
Soluble Reactive Phosphate	"	"	SOPs, SFWMD	--	<20%	75-125
Total Organic Carbon	"	"	SOPs	29	<20%	75-125
Sulfate	"	"	SOPs, USGS	65	<20%	75-125
Sulfide [2]	"	"	SOPs	98	<20%	75-125
Alkaline Phosphatase [2]	"	"	SOPs	63	<20%	75-125

[1] For sample result >5 times the MDL
[2] Non-critical, research parameter or media introduced in Phase II

* Actual Units
RPD: Relative Percent Difference
SOPs: Standard Operation Procedures
SRC: Suitable for Relative Comparisons

Table A1A. (Continued).

Measurement Variable	Representativeness	Completeness	Comparability (Split Samples SOPs, Std. Units)	Precision RPD [1] (Colocated Samples)	Precision RPD [1] (Lab Duplicates "split" samples)	Accuracy (% Spike Recovery in SRM, blank spikes, PE)
Chlorophyll a [2]	"	"	SOPs	--	<20%	SRC
Total Mercury	"	"	SOPs, Battelle	74	<30%	75-125
Methyl Mercury	"	"	SOPs, Battelle	71	<30%	75-125
PORE WATER						
Total Phosphorus [2]	Design-based statistically representative	90%	SOPs	--	<30	SRC
Ammonium-N [2]	"	"	SOPs	--	<30	SRC
Nitrite-N [2]	"	"	SOPs	--	<30	SRC
Nitrate-N [2]	"	"	SOPs	--	<30	SRC
Soluble Reactive Phosphate [2]	"	"	SOPs	--	<30	SRC
Bromide [2]	"	"	SOPs	--	<30	SRC
Chloride [2]	"	"	SOPs	–	<30	SRC
Fluoride [2]	"	"	SOPs	--	<30	SRC
Sulfate [2]	"	"	SOPs	--	<30	SRC
Sulfide [2]	"	"	SOPs	--	<30	SRC
SOIL/SEDIMENT						
Type	Design-based Statistically representative	90%	SOPs	NA	<30	SRC
Thickness	"	"	SOPs	NA	<30	SRC

[1] For sample result >5 times the MDL
[2] Non-critical, research parameter or media introduced in Phase II

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Table A1A. (Continued).

Measurement Variable	Representativeness	Completeness	Comparability (Split Samples SOPs, Std. Units)	Precision RPD [1] (Colocated Samples)	Precision RPD [1] (Lab Duplicates "split" samples)	Accuracy (% Spike Recovery in SRM, blank spikes, PE)
pH	"	"	SOPs	NA	<30	SRC
Redox (in situ)	"	"	SOPs	NA	NA	SRC
Redox (lab)	"	"	SOPs	NA	NA	SRC
Total Mercury	"	"	SOPs	83	<30%	75-125
Methyl Mercury	"	"	SOPs	--	<30%	75-125
Ethyl Mercury [2]	"	"	SOPs	121	<20%	75-125
Sulfate [2]	"	"	SOPs	220	<20%	75-125
Sulfide [2]	"	"	SOPs	--	<30	SRC
Total Phosphorus	"	"	SOPs	75	<20%	75-125
Ash Free Dry Weight	"	"	SOPs	--	<20%	SRC
Bulk Density	"	"	SOPs	87	<20%	SRC
Mineral Content [2]	"	"	SOPs	--	<30	SRC
CH4 [2]	"	"	SOPs	–	NA	SRC
CO2 [2]	"	"	SOPs	--	NA	SRC
Alkaline Phosphatase [2]	"	"	SOPs	--	<30	SRC
FLOC						
Total Mercury [2]	Design-based statistically representative	90%		–	<30	SRC
Methyl Mercury [2]	"	"		--	<30	SRC

[1] For sample result >5 times the MDL
[2] Non-critical, research parameter or media introduced in Phase II

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SRC: Suitable for Relative Comparisons

Table A1A. (Continued).

Measurement Variable	Representativeness	Completeness	Comparability (Split Samples SOPs, Std. Units)	Precision RPD [1] (Colocated Samples)	Precision RPD [1] (Lab Duplicates "split" samples)	Accuracy (% Spike Recovery in SRM, blank spikes, PE)
Alkaline Phosphatase [2]	"	"		--	<30	SRC
Ash Free Dry Weight [2]	"	"		--	<30	SRC
Bulk Density [2]	"	"		--	<30	SRC
Total Phosphorus [2]	"	"		--	<30	SRC
CH_4 [2]	"	"		--	NA	SRC
CO_2 [2]	"	"		--	NA	SRC
PERIPHYTON - Epiphytic						
Total Mercury [2]	Design-based statistically representative	90%	SOPs, Battelle	--	<30	SRC
Methyl Mercury [2]	"	"	SOPs, Battelle	--	<30	SRC
Ethyl Mercury [2]	"	"	SOPs	--	<30	SRC
Biomass [2]	"	"	SOPs	--	NA	SRC
Surface Area (% cover) [2]	"	"	SOPs	--	NA	SRC
Diatoms [2]	"	"	SOPs	--	NA	SRC
Pigments [2]	"	"	SOPs	--	NA	SRC
PERIPHYTON - Mat						
Total Mercury [2]	Design-based statistically representative	90%	SOPs, Battelle	--	<30	SRC
Methyl Mercury [2]	"	"	SOPs, Battelle	--	<30	SRC

[1] For sample result >5 times the MDL
[2] Non-critical, research parameter or media introduced in Phase II

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Table A1A. (Continued).

Measurement Variable	Representativeness	Completeness	Comparability (Split Samples SOPs, Std. Units)	Precision RPD [1] (Colocated Samples)	Precision RPD [1] (Lab Duplicates "split" samples)	Accuracy (% Spike Recovery in SRM, blank spikes, PE)
Ethyl Mercury [2]	"	"	SOPs	--	<30	SRC
Biomass [2]	"	"	SOPs	--	NA	SRC
Diatoms [2]	"	"	SOPs	--	NA	SRC
Pigments [2]	"	"	SOPs	--	NA	SRC
SAWGRASS						
Total Mercury [2]	Design-based statistically representative	90%	SOPs, Battelle	--	<30	SRC
Methyl Mercury [2]	"	"	SOPs, Battelle	--	<30	SRC
Ethyl Mercury [2]	"	"	SOPs	--	<30	SRC
Biomass [2]	"	"	SOPs	--	NA	SRC
Surface Area (% cover) [2]	"	"	SOPs	--	NA	SRC
CATTAILS						
Total Mercury [2]	Design-based statistically representative	90%	SOPs, Battelle	--	<30	SRC
Methyl Mercury [2]	"	"	SOPs, Battelle	--	<30	SRC
Ethyl Mercury [2]	"	"	SOPs	--	<30	SRC
Biomass [2]	"	"	SOPs	--	NA	SRC
Surface Area (% cover) [2]	"	"	SOPs	--	NA	SRC

[1] For sample result >5 times the MDL
[2] Non-critical, research parameter or media introduced in Phase II

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Table A1A. (Continued).

Measurement Variable	Representativeness	Completeness	Comparability (Split Samples SOPs, Std. Units)	Precision RPD [1] (Colocated Samples)	Precision RPD [1] (Lab Duplicates "split" samples)	Accuracy (% Spike Recovery in SRM, blank spikes, PE)
HABITAT EVALUATION						
Percent Cover (presence/absence) [2]	Design-based statistically representative	90%	SOPs	NA	NA	SRC
MOSQUITO-FISH						
Total Mercury	Design-based statistically representative	90%	SOPs, Battelle	91	<20%	70-130
Length	"	"	SOPs	–	NA	SRC
Weight	"	"	SOPs	--	NA	SRC
Sex	"	"	SOPs	--	NA	SRC

[1] For sample result >5 times the MDL
[2] Non-critical, research parameter or media introduced in Phase II

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Attachment 3

ESAT SOP XXXII Standard Operating Procedures for Sampling Water Sediment and Biota in Expansive Wetlands

SOP XXXII

STANDARD OPERATING PROCEDURES

FOR SAMPLING WATER, SEDIMENT, AND BIOTA

IN EXPANSIVE WETLANDS

Prepared by:

Biological Assessment Team
ManTech Environmental Technology Inc.
Athens, Georgia

For:

U.S. Environmental Protection Agency
Region 4
Athens, Georgia
1996
(Revised August 2000)

Contract No. 68-D6-0004
DCN: ESAT-4B-6000

This document is to serve as an "operations manual" for staging and executing sampling events in expansive wetlands containing remote sampling stations that require boats, airboats, helicopters for access. Outlined in the document are schedules of daily activities for both the field and a near-site laboratory, or base of operations, as well as lists of materials and supplies needed to carry out large-scale sampling events. A set of detailed procedures for collecting and processing samples is also included. Emphasis has been placed on sampling low levels of mercury. Included in the detailed procedures are guidelines for labeling, packaging, shipping, and tracking samples. QA/QC measures (when applicable) are appended to each procedure.

Table of Contents page

[1]Actual schedule and sampling routine may vary based on the specific goals of a project.

DAILY SCHEDULE (example)

0600-0630 Technical Support Personnel Arrive

Calibrate Hydrolab (s) (p. 12)
Pack sampling equipment and supplies (p. 3 or 5)
Disconnect GPS from charger and pack instruments (p. 3 or 5)

0715-0730 Field Personnel Arrive

Load equipment and supplies into van/truck for transport to helicopter/boat
Field team loads personal equipment (see p. 3 or 5)

0800 Load Equipment and Supplies into Helicopter/Boat

Field team departs for field
Support team returns to the laboratory (base).

0830-1730 Sampling and Support Activities

Field team	collects samples
Support team	Services and repairs field equipment
	Finishes bench-top analyses of water samples from previous day
	Finishes labeling/packaging samples from previous day (p. 27)
	Ships samples from previous day(s) (p. 29)
	Assembles packs of sample containers for next day

1730-1830 Post-sampling Activities

Unload helicopter/boat and transport samples/equipment/supplies back to base
Verify and then turn in Field Data Sheets (p. 10)
Add preservative to samples if necessary
Download information from GPS Unit(s) (p. 25)
End-calibrate Hydrolab(s)
Field team leaves when tasks are completed

1730-2130 Support Activities

Generate FORMS labels for newly collected samples
Start labeling samples
Process sediment samples (p. 26)
Perform bench-top analyses on water samples (pp. 30-33)
Support team leaves when tasks are completed

EQUIPMENT CHECKLIST - DEEP CHANNEL/CANAL STUDY

HELICOPTER/BOAT

LOAD OUT BY: _____ DATE:_____ CREW _____

TRIP BOX

- _____ EXTRA PUMP
- _____ LARGE DARK GARBAGE BAG
- _____ PAPER TOWELS
- _____ EXTRA LATEX GLOVES
- _____ EXTRA SHOULDER LENGTH GLOVES
- _____ PENCIL BOX
- _____ BAG OF SEDIMENT CUPS
- _____ SPOON (for mixing sediment)

PENCIL BOX

- _____ PENS, MARKERS, PENCILS
- _____ ELECTRICAL TAPE
- _____ WHITE LABEL TAPE
- _____ MESSENGER (for Ponar)
- _____ 2 ROLLS FILM - NUMBERED ___ ___
- _____ SMALL ZIPLOCK BAGS FOR SPIDERS
- _____ QUICK-RELEASE PLASTIC "TIES"
- _____ EXTRA SYRINGE & FILTER HEAD
- _____ CHLOROPHYLL & PARTICULATES KIT
  - _____ 2 SYRINGES
  - _____ 2 FILTER HEADS
  - _____ CUP OF FILTERS
  - _____ FORCEPS
  - _____ BAG OF MICROFUGE TUBES
  - _____ CELLOPHANE TAPE
- _____ 8 SAMPLE PACKS
  - _____ 4 SEDIMENT SPECIMEN CUPS
  - _____ 1 GLASS VIAL(for particulates)
  - _____ 1 PAIR LATEX GLOVES
  - _____ 1 PAIR LATEX GLOVES AND SHOULDER GLOVES
  - _____ 2-125 ML NALGENE BOTTLES
  - _____ 1 STORMOR
  - _____ SMALL ZIPLOCK BAG WITH 2 NYTEX SCREENS
  - _____ SMALL ZIPLOCK BAG FOR FISH

METAL CLIPBOARD/FOLDER

_____ FIELD DATA SHEETS
_____ MAPS OF STATIONS
_____ LIST OF PHONE NUMBERS
_____ COLLECTING PERMITS
_____ GPS COORDINATES LIST

TEFLON BOTTLE COOLER

_____ (8) 2 LITER TEFLON SAMPLE BOTTLES
_____ TRIP BLANK

INSTRUMENT BOX (unload contents into chopper, leave box)

_____ GPS UNIT
_____ CALIBRATED HYDROLAB W/ STIRRER
_____ HAND HELD 2-WAY RADIO
_____ CAMERA

MISC.

_____ SMALL COOLER W/ ICE (for fish) and DARK BOTTLE (for chlorophyll)
_____ VACUUM CHAMBER W/ PUMP, TUBING, AND SCREENING APPARATUS
_____ FISH NET
_____ GLASS PAN
_____ PETIT PONAR W/ ROPE

PERSONAL GEAR

_____ FLIGHT HELMET
_____ NOMEX FLIGHT SUIT
_____ NOMEX FLIGHT GLOVES
_____ CHEST WADERS
_____ SUNSCREEN
_____ HAT
_____ FOOD AND DRINK
_____ FIRST AID KIT

VAN : __TRIP __CLIP __TEFLON __INSTR __FISH __SYRINGE KIT

EQUIPMENT CHECKLIST - MARSH STUDY

HELICOPTER/BOAT _____

LOADOUT BY_____DATE _____CREW _____

TRIP BOX

- _____ EXTRA PUMP
- _____ LARGE DARK GARBAGE BAG
- _____ PAPER TOWELS
- _____ EXTRA LATEX GLOVES
- _____ EXTRA SHOULDER LENGTH GLOVES
- _____ 10 SEDIMENT BUCKETS WITH LIDS
- _____ 2 EXTRA 125ML NALGENE BOTTLES
- _____ 2 EXTRA SEDIMENT CUPS
- _____ PENCIL BOX
  - _____ PENS, MARKERS, PENCILS
  - _____ ELECTRICAL TAPE
  - _____ WHITE LABEL TAPE
  - _____ 2 ROLLS FILM - NUMBERED ___ ___
  - _____ SMALL ZIPLOCK BAGS FOR SPIDERS
  - _____ QUICK-RELEASE PLASTIC "TIES"
  - _____ 10 EXTRA NYTEX SCREENS
  - _____ MESSENGER WEIGHT (for Ponar Dredge)
  - _____ SUNGLASSES
- _____ 10 SAMPLE PACKS
  - _____ 3 PERIPHYTON SPECIMEN CUPS
  - _____ 1 PAIR LATEX GLOVES
  - _____ 1 PAIR LATEX GLOVES AND SHOULDER GLOVES
  - _____ 2-125 ML NALGENE BOTTLES
  - _____ 1 STORMOR
  - _____ SMALL ZIPLOCK BAG WITH 1 NYTEX SCREEN
  - _____ SMALL ZIPLOCK BAG FOR FISH
  - _____ 1 125 ml SULFIDE TEST BOTTLE

METAL CLIPBOARD

- _____ FIELD DATA SHEETS
- _____ MAPS OF STATIONS
- _____ LIST OF PHONE NUMBERS
- _____ COLLECTING PERMITS
- _____ GPS COORDINATES LIST

TEFLON BOTTLE COOLER

- _____ (10) 2 LITER TEFLON SAMPLE BOTTLES
- _____ TRIP BLANK

SEDIMENT COOLER

- _____ GLASS PAN
- _____ 2 SHORT CORING TUBES
- _____ 2 LONG CORING TUBES
- _____ TEFLON SPATULA
- _____ BOTTLE BRUSH
- _____ METRIC RULER
- _____ 1 SOIL PROFILE EH PROBE W/ CONTROL BOX
- _____ EXTRA CLIPS FOR EXTENSION POLES
- _____ 2 STOPPERS
- _____ 2 PLUNGERS
- _____ 1 HANDLE BAR

INSTRUMENT BOX (unload contents into chopper, leave box)

- _____ GPS UNIT
- _____ CALIBRATED HYDROLAB W/ STIRRER
- _____ HAND HELD 2-WAY RADIO
- _____ CAMERA

MISC.

- _____ SMALL COOLER W/ ICE FOR FISH
- _____ BLACK CASE W/ pH METER AND REFERENCE ELECTRODE
- _____ VACUUM CHAMBER W/ PUMP, TUBING, AND SCREENING APPARATUS
- _____ 1 LONG AND 1 SHORT FISH NETS
- _____ 2 ALUMINUM CORE CAP
- _____ 4 STAINLESS STEEL EXTENSIONS AND HANDLES
- _____ 12 FEET OF SECTIONAL MEASURING ROD
- _____ ANCHOR AND ROPE

PERSONAL GEAR

- _____ FLIGHT HELMET
- _____ NOMEX FLIGHT SUIT
- _____ NOMEX FLIGHT GLOVES
- _____ CHEST WADERS
- _____ SUNSCREEN
- _____ HAT
- _____ FOOD AND DRINK
- _____ FIRST AID KIT
- _____ MASK AND SNORKEL

VAN : __TRIP __CLIP __BLK CASE __TEFLON __SED __INSTR __FISH

FIELD SAMPLING ROUTINE

The following routine is an example of a routine designed for sampling water, sediment, and biota at remote sampling sites using a helicopter. The routine is fairly rigid, due to the priority consideration given to clean sampling protocols, although the actual order in which samples are taken is somewhat flexible[1]. All tasks are divided between a crew of two samplers. A sampler in the front seat of the helicopter usually operates the GPS equipment. The sampler in the back seat tends all sample containers (in two ice chests), operates the water sampling equipment and records data. Sediment and fish sampling equipment is stored in the rear compartment of the helicopter. The actual sampling is done from the pontoon of the helicopter.

Sequence:

1. Give helicopter pilot co-ordinates for each sampling station for the day before taking off.

2. Navigate to within 0.5 mile of sampling site using helicopter GPS. Then pinpoint the exact location of the site with the portable GPS and set down. If it is unsafe to land (pilot's decision), move to the nearest landing site with similar habitat where it is safe to land.

3. Upon landing, log GPS co-ordinates electronically and write co-ordinates on the Field Data Sheet (p. 10).

4. Fill in basic information on the Field Data Sheet (date, pilot's name, investigators' name, which investigator is crew chief, if water field blank will be taken, if duplicate samples will be taken, Eh probe #, Hydrolab #, camera model).

5. Fill in appropriate information on the marquee (station #, date, and film roll #) and photograph marquee (see Fig. 1 below). Write the frame number of the photograph on the Field Data Sheet.

Fig. 1

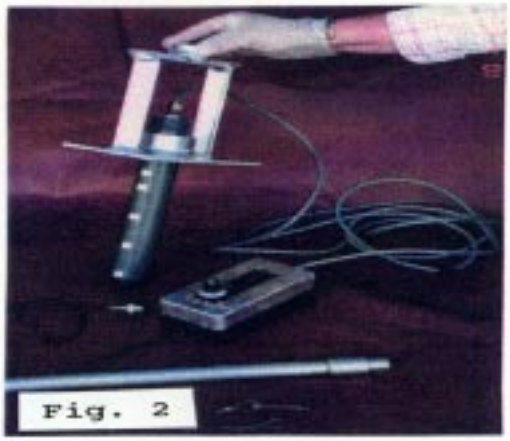
[1] The actual type and number of samples collected will be determined by the specific goals of the project.

6. Take ground-level photograph of the sampling site. Write the frame number on the Field Data Sheet.

7. Sampling: Integrate the execution of the following tasks in an order that enables the tasks to be completed by two investigators simultaneously while maintaining a clean technique.

. Setup vacuum chamber and collect water samples. Classical first followed by trace level mercury samples. (p. 15).

Insert Eh probe into sediment (**marsh study only**) and start timing. Hook up reference electrode to switch box (attached to the probe) and then connect switch box to a meter (see Fig 1. below). After 15 min. read and record (on Field Data Sheet) Eh's displayed on the meter, switching the dial on the switch box to read the Eh at five predetermined depths in the sediment.



Collect chlorophyll and particulates samples (**deep channel/canal study only**) (p. 18).

Collect surface water sulfide samples.

Insert Hydrolab probe beneath the surface of the water (**marsh study**), measure and record (on Field Data Sheet) temperature, pH, conductivity, dissolved oxygen, and redox. For **channel/canal studies**, measure and record same water parameters at 1 foot intervals to construct a profile for the water column.

Insert porewater sampler and collect nutrient and sulfide samples.

Collect soil cores and retain and collect the floc samples from the water column off the top of the core (p.20). Take a photograph (soil type) of at least one sediment core while

it is still in the coring tube (**marsh study only**). Record frame number of photograph on Field Data Sheet.

Collect periphyton and/or macrophyte samples. (**marsh study only**) (p. 22).

Collect fish (p. 23).

8. Pack samples and equipment for transport.

9. Fill in remaining blanks on Field Data Sheet (weather, number and type samples collected, vegetation type etc.) No blanks should remain. Both team members check and sign.

10. Depart. At an altitude of approximately 100', take an aerial photograph of the sampling site. Record the frame number of the photograph in the Field Data Sheet.

QA/QC

1. Review Field Data Sheet before leaving sampling station. Leave no blank spaces.

2. Before leaving sampling site, both investigators must review the Field Data Sheet(s) for completeness and accuracy and then sign them.

STEP WISE FIELD SAMPLING PROTOCOL (Everglades 1999)

ALWAYS WEAR GLOVES DURING SAMPLING

RECORD LATITUDE AND LONGITUDE
TAKE ONE FIELD BLANK PER DAY PER HELICOPTER
(Label with station, date, time, and helicopter #)
Take photos of station ID and landscape

SURFACE WATER SAMPLES:

VACUUM PUMPED SAMPLES: Use new nitex screen at each station
Pump 2 liter poly ¼ full to rinse 2 liter poly bottle
Pump 2 liter poly (½ to ¾ full, enough to rinse each bottle 3 times and fill it)
fill 1-125ml poly (TP/TOC/TN, Turbidity & AP, selected anions)(1-white & blue)
fill 1-125 ml poly (green) (SO4 and selected anions)
fill 1-125 ml poly (green) (TP, TOC)*
fill 1- 8oz glass and preserve (TKN, NO2, NO3)(green & red)*
Put on shoulder length gloves over regular vinyl gloves.
Record Teflon Bottle Number and Pump 2 liter Teflon full
Pump a second Teflon bottle*
Place full Teflon Bottle in ziploc bag and place in dark plastic bag in cooler

SULFIDE SAMPLE:
Attach syringe to the side port of the 3-way valve on the pre-preserved syringe
Remove protective cap from syringe
Place syringes under water and pull sample through side port to remove air from tip of syringe
Keeping tips of syringes under water, turn valve to off position on side port (arrow will point to side port) and pull sample into pre-preserved 60 ml syringe
Turn "off" valve back to 60 ml syringe, remove from water
Cap syringe, remove side port syringe, and place sample in sulfide box.

FILTERED GF/F NUTRIENT SAMPLES: (120 ml syringe)
Fill syringe with surface water, attach filter, rinse filter and bottle 3 times
(refill syringe as needed and add new filter and rinse 3 times as needed)
fill 1-60 ml poly (nutrients)(white)
fill 1-60 ml poly (green & red), preserved in the lab (Ammonia)(H2SO4)*
fill 1-60 ml poly (green) (NO2,NO3,PO4)*
PLACE ALL FILTERED SAMPLES ON ICE

DEPLOY HYDROLAB AND RECORD DATA
MEASURE WATER, FLOC, SOIL DEPTH AND RECORD
DEPLOY Eh PROBE AND SET TIMER (record after 15 minutes)

POREWATER:
Set out "sippers". Fill 60 ml syringe with 30 mls of surface water and flush filter & bottle 3X,s

Fill syringe with 30 mls of porewater
Flush filter and bottle with 10 mls porewater
fill 1-30 ml poly (Nutrients)(white) with remaining 20 mls porewater
Attach pre-preserved syringe to "sipper" tube, purge air with extra syringe attached to side port as with surface water, close off side port and
fill 1-60 ml pre- preserved syringe with 30 mls pore water (for H2S)(blue & red)
fill 1-30 ml poly (green) Filtered nutrients (without NH4), use 10 mls for rinse*

PLACE ON ICE

PERIPHYTON (floating mat): fill 1 (32oz) bucket in field
Note on the field sheet if it is the dominant type
If mats are present take "cookies" with cutter (enough to fill a 4 oz cup if possible) and record the actual number of cookies in the cup. Volume/wt ratio and AFDW

PERIPHYTON (epiphytic): fill 1 (32oz) bucket in field
Note on the field sheet if it is the dominant type

PERIPHYTON (soil mat): fill 1 (32oz) bucket in field
Note on the field sheet if cup is from soil mat

FLOC:
Before removing the sediment core from the tube pour the H20 and floc into the Imhoff Cone and allow the floc to settle while processing the soil cores (3 core total). Fill one 500ml storemore with concentrated floc.

SOIL:
Place 3 cores (top 10cm of soil) in a plastic bucket and seal. Collect more cores if needed.

FISH:
Collect 2 small bags of 15 fish each (Collect full compliment in order of priority) (When QA/QC sample is taken, it will take precedence over USGS sample)
1 bag for FIU (HgT)
Collect 1-2oz. pre-preserved (10% Formalin) jar of 20 fish.
1 bag for USGS (Isotope analysis)
1 bag for EPA (HgT)*

FLAG STATION
MAKE SURE ALL BLANKS ON FIELD DATA SHEET ARE FILLED IN BEFORE LEAVING THE STATION!
TAKE AERIAL PHOTO OF STATION
****BOLD, ITALICIZED LETTERING INDICATES SAMPLES TAKEN AT "DUPLICATE STATIONS".***
COLLECT DAILY CHAMBER BLANKS IN THE LAB

South Florida Mercury Study Data Field Sheet (Cycle 5) **9/07/99**

Station# _____

Date _____/_____/_____

Duplicate Station Y / N

GPS start time _____

lat _____

long_____

EPA____ Other_____

Investigators

Helicopter #

Pilot

Weather
1 clear
2 slt ovrcst
3 med ovrcst
4 vry ovrcst
5 drizzle
6 rain

Flow
1 sheetflow
2 isolated pool
3 unsure

Camera_____ Roll#_____
Frames:
station ID _____
ground _____
soil _____
air _____
other _____

Surface Water
(2 L poly)
125 ml poly (b&w) _____
125 ml poly (grn)_____

125 ml poly (grn)_____
8 oz glass (grn&rd) _____

Surface Water
2 L teflon bottle #_____
Dup teflon bottle#_____

Water field blank Y / N
Teflon bottle # _____

Sulfide
60 ml syringe
(b&rd)

_____

Filter GF/F Nutrients
(120 ml syringe)

60 ml poly (w) _____

60 ml poly(grn&rd)__ __(NH4)
60 ml poly (grn)_____(nut)

In-situ water measurements
Hydrolab unit # _____
Depth (in)_____
Temp (°C)_____
DO (mg/L)_____
Redox (+/-) _____
Cond _____
pH _____

Surface water depth (0.1ft) _____
Floc thickness (0.1ft) _____
Soil thickness (0.1ft) _____

Deploy Eh probe & set timer

Porewater
30 ml poly (w) _____
30 mls in 60 ml syringe (b&rd) _____

30 ml poly (grn) _____

Soil Eh

Probe # _____
Start time_____
Stop time_____

Depth (cm)	Eh (+/-)
2.5	_____
5.0	_____
10.0	_____
15.0	_____
20.0	_____

Periphyton

type	#buckets	*QA #cups*
pm	_____	_____
ps	_____	_____
pu	_____	_____

if mat, # cookies _____
Dominant type _____

Soil type

1 peat
2 peat layers
3 marl
4 marl layers
5 sand
6 other

	Amt.	*QA amt.*
soil cores (3)	_____	_____
floc stmr	_____	_____

Vegetation type ____ Fish habitat _____
1 wet prarie
2 sawgrass marsh
3 pond/gater hole
4 cattail
5 willow
6 cypress
7 brush
8 other

Signed_____

Signed_____

Fish

bag (15) _____
jar (20) _____
bag (15) _____

bag (15) _____

Comments:

b=blue, rd=red, grn=green, w=white ***shading/italics indicates QA/QC samples*** pm=periphyton floating mat, ps=periphyton soil mat, pu=epiphytic periphyton on*Utricularia*

MAY/SEPTEMBER 1999 STUDY CONTAINERS BY MEDIA FOR THE EVERGLADES

PARAMETER WATER (SURFACE)	LABS	FILTER	CONTAINERS	TOTAL # OF BOTTLES	PRESERVE	SUPPLIED BY	HOLDING TIMES
TP, TOC, TN, AP,Turb. (100%) SO4, Cl, Br, F, NO2, NO3, ortho-P (10%)	F	NO	125 ml poly	151 23	NO, Fill full in field, space from AP & Turb removal	FIU	48 hr/ 28 days (Refrig.)
TP, TOC (10%)	E	NO	125 ml poly	23	NO	FIU	48 hr
TKN, NO2, NO3 (10%)	E	NO	250ml glass	23	10% H2SO4	EPA	48hr/ (P)
S04, Cl, Br, F, NO2, NO3, ortho-P (100%)	E	NO	125ml poly	151	NO	EPA	28 days
HgT (100%), MeHg (10%)	F	NO	2 L teflon split	183 (18 per day)	Yes in lab	FIU	28 days
HgT (10%)	E	NO	500 ml teflon	23	Yes in lab	EPA	28 days
HgT (10%), MeHg (100%)	B	NO	500 ml teflon	183	Yes in lab	BAT	28 days
Filtered Nutrients (100%) NH4, NO2,NO3,PO4	F	YES GF/F	filter 60 ml with syringe	151	NO	FIU	48 hr/ or frozen for 28 days
Filtered Nutrients (10%) NH4,NO2,NO3,PO4,SO4	E	YES GF/F	filter 60 ml with syringe	23 23	NO NH4/H2SO4 (Proportional 2ml/L)	EPA	48hr all except NH4 = 28 days
H2S (100%)	ESAT	NO	syringe	151	zinc acetate (field) NaOH (lab) mix well	EPA	7 days

PARAMETER	LABS	FILTER	QUANTITY	CONTAINER	TOTAL # BOTTLES (minimum)	PRESERVE	SUPPLIED BY
POREWATER							
Filtered nutrients NH4,NO2,NO3, PO4 (100%)	F	YES GF/F	30ml: 10ml washing and 20 ml sample	30ml poly	151 (FIU)	NO	FIU
Filtered nutrients NO2,NO3, PO4 (10%)	E	YES GF/F	30ml: 10 ml washing and 20 ml sample	30ml poly	23 (EPA)	NO	EPA
H2S (100%)	ESAT	NO	30 ml: 10 ml washing and 20 ml sample	syringe	151 (EPA)	YES -Zinc acetate,NaOH	EPA

PARAMETER	LABS	CORES	CONTAINERS	TOTAL # OF BUCKETS/ CUPS/JARS	SUPPLIED BY	TREATMENT
SOIL						
HgT (100%) Sulfate (100%)	E	3 per bucket	4 oz. cup (in lab)	155 cups	FIU	Blend/ split/freeze
HgT (10%), MeHg, EtHg (100%)	F		4 oz. cup (in lab)	155 cups	FIU	Blend/ split/freeze
MeHg (10%)	B		4 oz. cup (in lab)	15 cups	FIU	Blend/ split/freeze
TP, Alk-Phos, CH4 & CO2, AFDW, Bulk Den., Min. content (100%)	F		4 oz. cup (in lab)	155 cups	FIU	Blend/split; Room Temp then Frozen
Stable Isotopes (100%) USGS	U		4 oz. cup (in lab)	155 cups	FIU	Blend/ split/freeze

PARAMETER	LABS	CONTAINERS	TOTAL # OF BOTTLES	SUPPLIED BY	TREATMENT:
FLOC SAMPLE					
HgT, MeHg (100%)	F	4 OZ CUP	155	FIU	Blend/split/ freeze
HgT (10%)	E	4 OZ CUP	15	FIU	Blend/split/ freeze
MeHg (10%)	B	4 OZ CUP	15	FIU	Blend/split/ freeze
Alk-phos, CH4 & CO2 TP, AFDW, Bulk Den., Min cont. (100%)	F	4 OZ CUP	155	FIU	Blend/split/ Room Temp then freeze
Stable Isotopes (100%) USGS	U	4 OZ CUP	155	FIU	Blend/split/ freeze

$V=a'(\pi r^{2)}$
Soil = 140.6 cc
Floc = 210.94 cc

PARAMETER	LABS	CONTAINERS	TOTAL # OF CUPS/BUCKETS	SUPPLIED BY	TREATMENT:
Periphyton Mat (Surface)					
HgT, MeHg (100%); Diatom Comp. (100%); Pigment (100%)	F	4 oz. cup	155	FIU	Blend/split/ freeze
HgT (10%)	E	4 oz. cup	15	FIU	Blend/split/ freeze
MeHg (10%)	B	4 oz. cup	15	FIU	Blend/split/ freeze
Biomass Cookies, V/W ratio, AFDW, Archive (100%)	F	4 oz. cup	155	FIU	Blend/split/ freeze
Stable Isotopes (100%) USGS	U	4 oz. cup	155	FIU	Blend/split/ freeze

PARAMETER	LABS	CONTAINERS	TOTAL # OF CUPS/BUCKETS	SUPPLIED BY	TREATMENT:
Periphyton Mat (Soil)					
HgT, MeHg (100%); Diatom Comp. (100%); Pigment (100%)	F	4 oz. cup	155	FIU	Blend/split/ freeze
HgT (10%)	E	4 oz. cup	15	FIU	Blend/split/ freeze
MeHg (10%)	B	4 oz. cup	15	FIU	Blend/split/ freeze
Biomass Cookies, V/W ratio, AFDW, Archive (100%)	F	4 oz. cup	155	FIU	Blend/split/ freeze
Stable Isotopes (100%) USGS	U	4 oz. cup	155	FIU	Blend/split/ freeze

PARAMETER	LABS	CONTAINERS	TOTAL # OF CUPS/BUCKETS	SUPPLIED BY	TREATMENT:
Periphyton Epiphytic					
HgT, MeHg (100%); Diatom Comp. (100%); Pigment (100%)	F	4 oz. cup	155	FIU	Blend/split/freeze
HgT (10%)	E	4 oz. cup	15	FIU	Blend/split/freeze
MeHg (10%)	B	4 oz. cup	15	FIU	Blend/split/freeze
Biomass (V/W ratio); AFDW, Archive (100%)	F	4 oz. cup	155	FIU	Blend/split/freeze
Stable Isotopes (100%) USGS	U	4 oz. cup	155	FIU	Blend/split/freeze

Cycle 4 Only (May 1999)

PARAMETER	LABS	VOLUME	CONTAINERS	TOTAL # OF BAGS	SUPPLIED BY
SAWGRASS (Homogenate)					
HgT (100%)	F	5 whole blades; Subsample, dry, grind homogenate	Plastic Bag, 4 oz cup	150 bags; 150 4 oz cups	Bags - EPA Cups - FIU
HgT (10%)	E	Taken from homogenate	Vial	15 Scintillation Vials	Vials - FIU
Stable Isotopes (100%) USGS	U	Taken from homogenate	Vial	150 Scintillation Vials	Vials - FIU
CATTAILS (Homogenate)					
HgT, (100%)	F	5 whole blades	Plastic Bag	150 bags; 150 4 oz cups	Bags - EPA Cups - FIU
HgT (10%)	E	Taken from homogenate	Vial	15 - Scintillation Vials	Vials - FIU
Stable Isotopes (100%) USGS	U	Taken from homogenate	Vial	150 Scintillation Vials	Vials - FIU

PARAMETER	LABS	CONTAINERS	TOTAL # OF CONTAINERS	SUPPLIED BY	Treatment
FISH					
HgT (Individual) (100%)	F	Small Zip Lock Bag	150 bags (15 fish per bag)	FIU	Freeze
HgT (Individual) (10%)	E	Small Zip Lock Bag	15 (15 fish per bag)	FIU	Freeze
Gut Contents	F	2 oz. jar (10% Formalin)	150 (20 fish per jar)	EPA	Freeze
Stable Isotopes	USGS	Small Zip Lock Bag	150 bags (15 fish per bag)	FIU	Freeze

FORMS SETUP

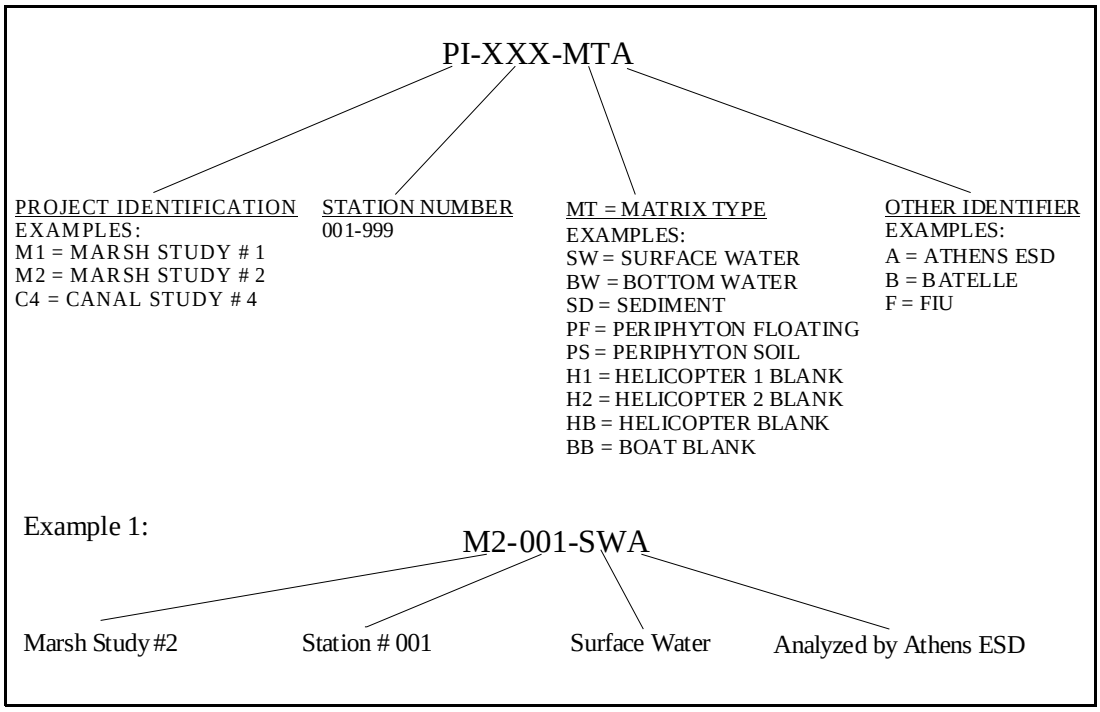
FORMS (Field Operations Record Management System) software is used to generate labels and chains-of-custody for all samples. Familiarity with the FORMS program is required.

Equipment Required:

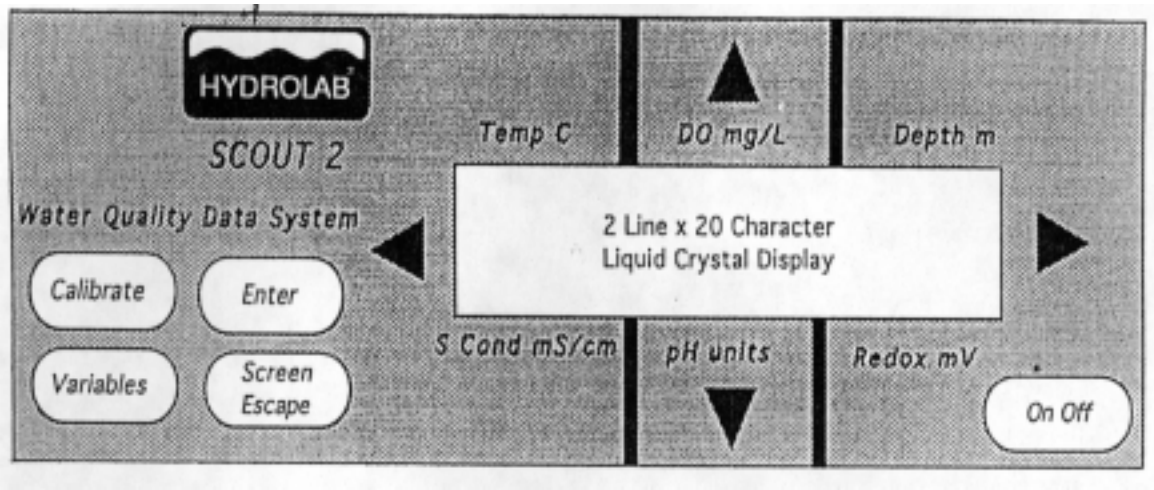
Computer with FORMS software installed.
Labels for Lazer printer (3½"x $^{15}/_{16}$" computer labels).
Sample Tags numbered in sequential order.
Log books.

Information Required to Setup FORMS:

Project number (if samples will be sent to the Region IV ESD laboratory).
A list of sampling stations complete with station identification #'s (see below).
Type of samples to be collected (water, sediment, periphyton ...etc).
List analyses to be performed on each sample type (THG, TOC, MeHg etc).
Name of each laboratory analyzing samples.
Which stations will require duplicate samples (eg. any station # ending in "0").
Which analyses and stations will require an additional QA sample (eg. stations ending in "3").
List of laboratories that will be performing QA analyses.
rmat for sample identification #'s:



BASIC CALIBRATION – HYDROLAB SCOUT 2
Refer to diagram of Scout 2 display unit panel keys (below)



1. Turn logger on by pressing ON/OFF pad on logger.

2. Press **Screen Escape** pad and wait for the readout of the number of volts (v) of battery power to appear in the upper right corner of the LCD. If the value reads less than 12v., replace the AA batteries in the logger unit with fresh batteries. If the value is 12v or greater, proceed.

3. Unscrew cup of water covering probes on end of sonde and set aside.

4. Attach (screw) a cup of standardizing solution to end of the sonde (start with pH 7.00), give solution a swirl, and then follows these steps:

   A. Press **Calibrate** key pad, then scroll through the displays by pressing the arrow key » on either side of the LCD until the appropriate parameter appears in the bottom, right corner of the LCD. For pH, the screen will display:

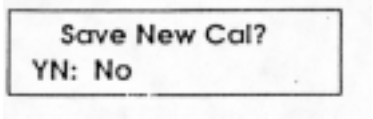
      Calibrate PCS%OARDT: pH

   B. Press the **Enter** pad. The screen should now display: Calibrate pH: 7.01

   C. Wait for the reading on the LCD to stabilize.

   D. A cursor will be flashing under the first digit. If all digits are correct, advance to the next step. If any digit is incorrect, use the arrow pads to the **right** and **left** of the LCD to place the cursor under each incorrect digit, and then use arrow keys **above** and **below** the LCD to scroll up or down until the correct digit appears.

   E. When all digits are correct, press the **ENTER** pad. The screen will display:



F. Press either the **right** or **left** arrow to change **No** to **Yes**.

G. Press **Enter**. The unit is now calibrated to the standardizing solution.

H. Remove cup from sonde, rinse probes with deionized water, attach next standardizing solution and repeat steps 4 A thru H.

5. After calibrating to pH 7.00, calibrate to pH 10.00, then simply read and record the value for pH 4.00(or *vice versa*) (see attached HYDROLAB CALIBRATION FORM p.14). (Note: if necessary, standardizing solutions can be reused for 4-5 days)

6. Next calibrate to a conductivity of 718 via steps 4 A thru H, then read and record the value for the solution with a conductivity of 1413. (Note: Do not reuse solutions)

7. Calibrate to Redox solution A via steps 4 A thru H, then read and record value for Redox solution B. (Note: the values for Redox solutions A & B will vary each time the standards are made, but the exact value for each standard is always marked on the bottle; the solution can be reused for 4- 5 days).

8. Draw chlorine-free water from a 5 gal. bucket, perform two Winkler titrations, and record the mg/l DO for each titration (see HYDROLAB CALIBRATION FORM).

9. Attach the stirrer to the end of the sonde, immerse the probes in the bucket of chlorine-free water, wait (several minutes) for the DO reading to stabilize, and then follow steps 4 A thru H as before.

10. Remove the sonde from the bucket of water. While exposed to the air, follow steps 4 A thru H to calibrate Depth to 000.0.

11. When all calibrations are completed, disconnect stirrer, reattach cup of tap water, and turn **OFF** the unit.

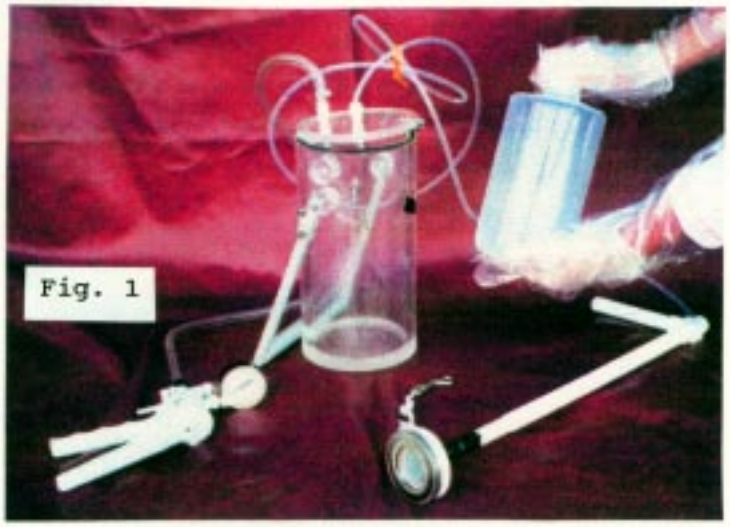
*For more detail and for servicing refer to instrument manual.

SAMPLING WATER

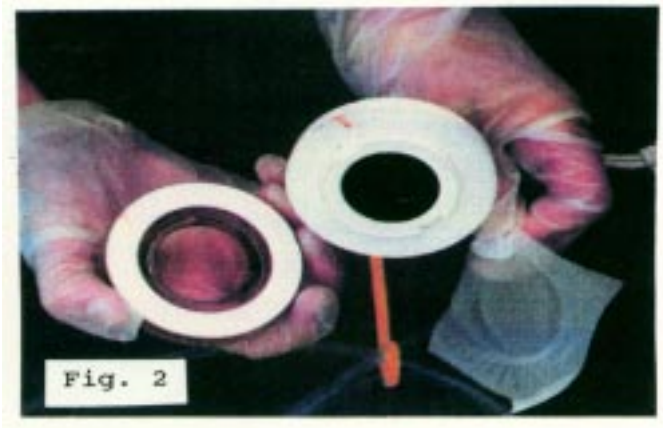
Water samples are usually collected by submerging an open sample container beneath the surface of the water. However, when collecting water for low-level mercury analysis, samples are best collected with the aid of a vacuum apparatus (see Fig. 1 below) consisting of a vacuum chamber, teflon sampling wand, and a hand pump.

Procedure:

1. Wearing latex gloves, drop an uncapped 2L polypropylene bottle into the vacuum chamber and secure the chamber lid by attaching the spring-loaded clamps.



2. Place a fresh square of Nitex® screening over end of sampling wand and secure with the magnetic ring (Fig. 2). Insert head of wand beneath surface of water and tie shaft of wand to pontoon of helicopter or to gunwale of boat (strap is provided).



3. Squeeze the hand pump until liquid starts to fill the bottle. When water level in bottle is about an inch deep, release the vacuum, remove the bottle, discard the water (swirling the water to rinse the bottle as you dump), and replace the bottle in the vacuum chamber.

4. Begin pumping again. When bottle is 3/4 full, release the vacuum, remove from chamber, and cap.

5. From the 2L polypropylene bottle, fill labeled sample containers with classical nutrient samples, making sure to rinse each bottle three times with water from the 2L bottle before filling.

    500 ml Storemore® bottle
    125 ml polypropylene bottles with screw caps

6. Place filled nutrient sample containers in a clean cooler for transport.

7. When sampling for trace level mercury, wear shoulder-length polypropylene gloves (over latex gloves), remove 2L Teflon® bottle from its protective Ziploc® bag, record on a Field Data Sheet (see p. 10) the bottle number etched near the top of the bottle, and then carefully mark the station number on the colored label attached to the bottle using waterproof marker.

8. Uncap the Teflon bottle and place bottle and cap into vacuum chamber. Secure the chamber lid and begin pumping.

9. Fill this bottle to overflowing, then release the vacuum, cap tightly, and return bottle to its protective bag.

10. Place bottle and bag in a clean cooler for transport.

QA/QC

1. Wear latex gloves when handling polypropylene sample containers.

2. Wear polypropylene gloves over latex gloves when handling Teflon sample containers.

3. Store Teflon bottles in a Ziploc® bag before and after collecting the sample, which in turn is stored in a clean cooler lined with a clean plastic trash bag.

4. Use only Teflon bottles that have been specially cleaned (FIU procedure).

5. During transport from the field, store all samples in a ice chest (lined with a plastic garbage bag) for protection.

PREPARATION AND ANALYSIS OF SULFIDE SYRINGE SAMPLES

Materials/supplies

60 ml plastic syringes with leur-loc tip
ZnAcetate solution
6N NaOH solution
3-way valves with leur-loc
Rack to hold syringes upright.

Procedure:

1. Remove cap from tip of syringe and then plunger and set parts aside on a "clean surface."

2. Attach 3-way valve to tip of syringe and turn stopcock on valve to block opening to the syringe. Place the syringe open end up in a rack.

3. Prepare a fresh batch of preservative by mixing 30 ml of ZnAcetate and 20 ml of 6N NaOH in a 60 ml polyethylene bottle. A precipitate (ZnOH) will soon form. Just before use (step 4 below) shake the bottle vigorously for a few seconds to evenly suspend the precipitate.

4. Transfer 0.5 ml of preservative to syringe.

5. Reinsert plunger, invert syringe (tip up), rotate stopcock on 3-way valve to open passage from the syringe, and carefully expell air, leaving only preservative in the syringe.

6. Close stopcock opening to the syringe, replace cap on the tip of the 3-way valve. The syringe is now ready for the field where the sample is taken.

7. The syringe is delivered to the laboratory with the preserved sample and the analyst pairs the surface and porewater samples and checks the pH.

8. If the pH is <10, drops of 6N Sodium Hydroxide are placed into the valve side port and the valve set to allow the analyst to draw the NaOH into the sample. Once the samples have a pH >=10, the syringes are stood upright for at least 30 minutes, but typically allowed to stand overnight.

9. After settling, the volume of supernatant is expelled from the syringe, and the same volume of DI water is drawn into the syringe. This step removes interferences from the samples, but retains the original concentration. The sample is now ready for analysis.

10. Each sample is matched with a pair of cuvettes. One cuvette will contain 25 ml of DI water as a blank, while the other cuvette will contain 25 ml of sample. The cuvette pairs are placed on sheets of paper identifying the run order.

11. The Hach DR/2010 Spectrometer is turned on, and analysis program 690 is recalled. If necessary, the wavelength dial is set to 665 nm. The analysis begins by adding 1 ml of Reagent 1 to each cuvette.

12. The reagent and the water in each cuvette are mixed using transfer pipets. Then 1 ml of Reagent 2 is added to each cuvette and the analyst then starts a 5 minute countdown timer and mixes using the transfer pipets.

13. The blank cuvette is placed in the meter reading chamber and after 5 minutes the analyst presses the zero button on the meter. The meter display will blink and indicate the meter is zeroing.

14. When the blinking stops, the analyst removes the blank cuvette and replaces it with the sample cuvette. The meter will then display the measured result of the sample.

15. The analyst will store the result by pressing the store button and confirming the operation by pressing the enter button. The sample may now be disposed.

16. Following analysis of all samples the stored values are downloaded from the analyzer.

SAMPLING
CHLOROPHYLL and PARTICULATES

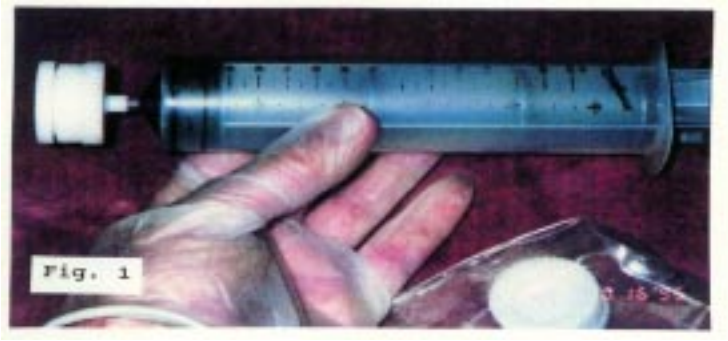
Required supplies

140 ml plastic syringe
47mm 0.45 μ membrane filters
filter holder for 47 mm filters

Procedure

1. Draw site water into syringe and expel to rinse syringe.

2. Draw water into syringe again and attach filter holder containing a fresh filter (see Fig. 1 below).



3. Apply steady pressure to the plunger to force the entire volume of water in the syringe through the filter.

4. Disconnect filter holder and repeat steps 2 and 3. Ideally, a total of three volumes of water should be filtered. This is not always physically possible. If filter becomes totally clogged before filtering three volumes, record the actual volume filtered.

5. After filtering three volumes of water (or less if the filter becomes totally clogged), remover the filter from the holder with clean forceps and stuff the filter into a microfuge tube (or small glass vial) and cap. This is the chlorophyll sample. Store on ice in the dark for transport.

6. Repeat steps 2 - 4, remove the filter from the holder with forceps, place in a glass vial, cap, and store for transport. This is the particulates sample.

7. In the laboratory, fill each microfuge tube with acetone and store at 4°C to await chlorophyll analysis.

QA/QC

1. Wear latex gloves while collecting samples.
2. Do not apply excessive force to the plunger of the syringe. Excessive force can rupture the membrane filter.

DEEP CHANNELS AND CANALS

1. Collect and lift sediment to the surface with a Petit Ponar dredge.

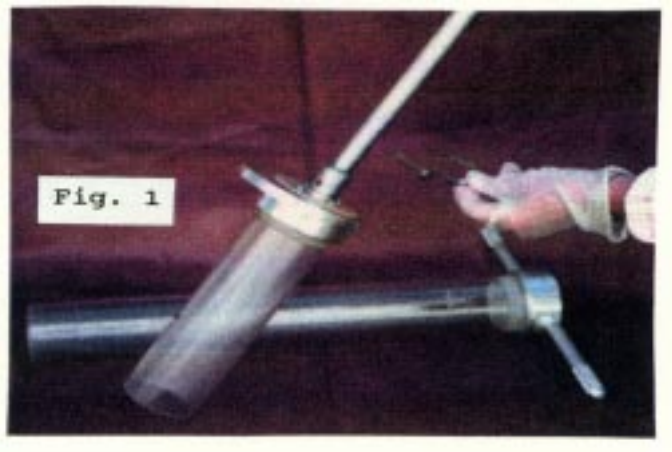
2. Homogenize sediment in a "clean" glass pan with a "clean" stainless steel or teflon-coated spoon.

3. Spoon sediment into labeled containers.

4. Store sediment samples in a cooler for transport.

MARSHES

1. Collect soil/sediment samples using the coring device pictured below (Fig. 1).



2. Collect three cores at each sampling station. Pore the floc trapped in the core top into a separate container. Place the soil core samples in a 1-gallon plastic bucket with a tight-fitting lid. Keep only the top 10 cm of sediment from each core. A plunger (provided) is used to extrude longer cores from the coring tube until only the top 10 cm of core remain.

3. Do not homogenize sediment cores in the field. Store gallon containers for transport and then process sediment in the laboratory at the end of the day (see SEDIMENT PROCESSING p. 26).

QA/QC

1. Wear latex gloves while collecting and handling sediments.
2. Keep clean glass pans and stainless steel spoons wrapped in foil until needed.
3. Rinse coring device with site water before collecting cores.
4. Take a photograph of one of the three cores while still in the coring tube.

PERIPHYTON COLLECTION

Mat Samples

1. Soil periphyton mat is collected off the top of all soil core samples taken at a site and placed in a 500 ml plastic container.

2. Floating periphyton mat samples are collected with a stainless steel “cookie cutter” with a plexiglas sheet placed under the mat. The cutter and soil core sampler both have the same 3-inch diameter. The periphyton from atleast 3 samples is placed in a 500 ml plastic container.

3. Cap and store at ambient temperature for transport.

4. Upon returning from the field, process and freeze samples.

Epiphytic Samples

1. Periphyton samples were collected from concentrations which were epiphytic on Utricularia where both occurred together. The sample was placed in a separate 500 ml plastic container.

2. Cap and store at ambient temperature for transport.

3. Upon returning from the field, process and freeze sample.

QA/QC

1. Wear latex gloves while collecting samples.
2. Keep frozen until analyzed.

FISH COLLECTION

Freshwater Marshes/Canals

The target species for freshwater marshes is the mosquito fish *Gambusia sp*. These small ubiquitous fish are found throughout weedy areas of a marsh and along the edges of canals and deep channels. They congregate around the pontoons of a helicopter or the hull of a boat and are easily collected with a "clean" dip net. The fish are handled with latex gloves., placed in a Ziploc® bag, and packed in ice for transport back the laboratory. In the laboratory the fish are frozen until they are processed for analysis. For low-level mercury analysis a minimum of 20 fish is required and a an additional minimum of 20 fish were collected for stomach analyses.

Saltwater Marshes/Tidal Creeks

The target species for saltwater marshes is the mummichog *Fundulus heteroclitus*. The distribution of these small, ubiquitous fish in the salt marsh is influenced by the daily cycle of the tides. The following procedure has proven successful in locating and collecting this species:

1. Starting about 2 hours after high tide, locate small, V-notched channels ($\leq$ 10 feet across) that drain the marsh grass as the tide recedes.

2. Plant a minnow trap in the bottom of the V-notch in about 2 feet of water. If the bottom of the notch is wider than the width of the trap, plant more than one trap so that by low tide the last of the water draining the marsh grass must pass through the trap(s) (see Fig.1 below). Note in Fig.1 that the traps are secured in place by tying them (before planting) to a length of steel conduit stuck into the mud. Set out traps at 2 to 4 small channels in the vicinity of each sampling station.

3. As the tide recedes, return to the traps every 20 to 30 minutes to check for fish. If a trap is exposed and does not contain fish, replant.

4. Wearing latex gloves, transfer fish from the traps to a "clean" glass jar fitted with a Teflon lid and then store on ice for transport back to the laboratory. In the laboratory, fish are frozen until they are processed for analysis. For organic analyses a minimum of 30 gm of fish is required (about 20 fish).

(Note: Fish can also be collected on the rising tide by planting traps in exposed V-notch channels ahead of the rising water.)

QA/QC

1. Wear latex gloves when handling fish.
2. Freeze fish within 48 hours of collection and keep frozen until processed for chemical analysis.

DOWNLOADING GPS UNIT (Trimble® Pathfinder Pro)

1. Disconnect data logger and antenna from the external battery pack and then connect external battery to charger.

2. Connect logger to computer using appropriate pigtail connector.

3. Turn "on" computer. Exit to DOS (F8). When C:\> appears, type "pfinder" and hit ENTER. The PATHFINDER program will appear on screen. Hit Okay to accept.

   NOTE: Selections are made from a menu by highlighting and pressing ENTER or by hitting ALT followed by the underlined letter in the desired heading. (e.g. to accept Okay, hit ALT then O).

4. Select Project and then Current files. The filename (e.g. MARSH1) should be displayed.

5. Now select Comm , highlight Data Files to PC, and then hit ENTER. Computer will tell you that it is **Looking for data logger on COM1**

6. Turn "on" data logger. When main menu appears scroll down to selection #9 - DATA FILE MGMT (or simply hit 9 then ENTER). Hit ENTER.

7. Now under DATA FILE MGMT select "0" TRANS SERVER and hit ENTER. (If the computer does not indicate that it is accepting data from the logger, hit ENTER on the logger again). A list of files will appear on the computer screen.

   If you wish to download all files, hit Okay.

   If you wish to download only a select number of files, first tag the files. Tagging is accomplished by highlighting a file and then hitting either ENTER or the space bar. To accept the tagged files, hit Okay.

   The computer screen will now indicate that each file in succession is being transferred, converted, and finally used in a calculation.

8. After all files have been downloaded onto the computer, check the file (MARSH1) to see if they are there. This is accomplished by returning to DOS and after C:\> appears, type pfinder\data\pfinder\ marsh1 and hitting ENTER.

9. If all downloaded files are present, copy files to a disc by inserting a disc into A drive and typing copy *.SSF A: and hitting ENTER. Computer will diplay name of each file copied.

10. After files have been copied, ERASE files from data logger (select #9 then the #2 or #3).

Equipment/Supplies

Osterizer 10-speed blender motor
High Density Polyethylene (HDPE) blender jars - 500 ml (48)
Polyethylene spoons (15)
"Blade assembly" (knurled base, stainless steel blades, gasket)
Deionized water supply
Graduated cylinder or cup - 100 mls
paper towels

Procedures:

Soil

1. With a PE spoon chop sample cores in the sample container once or twice and then sniff the sample for traces of H_2S. Record finding (yes/no, slight, strong).

2. After sniffing, continue to chip and mix sample core while removing large sticks, rocks, and roots. If necessary add DI water to the sample until the mixture becomes as slurry. Record the volume of water added.

3. Spoon mixed sample into a 500 ml HDPE jar until the jar is 3/4 full.

4. Attach blade assembly to the jar and blend for 30-60 seconds on "BLEND" setting until the sample is thoroughly homogenized.

5. Pour or spoon the homogenized sample into labeled, 120 ml specimen cups. Normally, there is enough sample to fill up to 5 cups ½ to 3/4 full. (Do not fill cups more than 3/4 full because upon freezing they may burst the container).

6. Store specimen cups for FIU-AFDW (ash-free dry wt.) **at room temperature**. Store all other cups in a freezer.

Periphyton

1. Briefly chop and stir the sample with a PE spoon. If the sample is dry add DI water until the mixture is thoroughly wet and glistening. Record the volume of water added.

2. Spoon mixture in a 500 ml HDPE blender jar until the jar is 3/4 full.

3. Attach blade assembly to the jar and blend for 30-60 seconds on "BLEND" setting until the sample is thoroughly homogenized.
4. Pour homogenized sample into labeled, 120 specimen cups as before.

5. Store all cups in freezer.

Floc (collected in 500 ml polyethylene bottle)

1. Shake the collection bottle several times and then pour contents directly into a 500 ml HDPE bender jar.

2. Attach blade assembly to the blender jar and blend for 30 to 60 seconds on "LIQUIFY" setting.

3. Pour liquified sample into labeled, 120 ml specimen cups as before filling the cups no more than 3/4 full.

4. Store all cups in freezer.

Note: To have enough floc to fill 3 to 5 specimen cups, the floc in the bottom of the collection should be at a depth of 1 to 1 ½ inches.

QA/QC

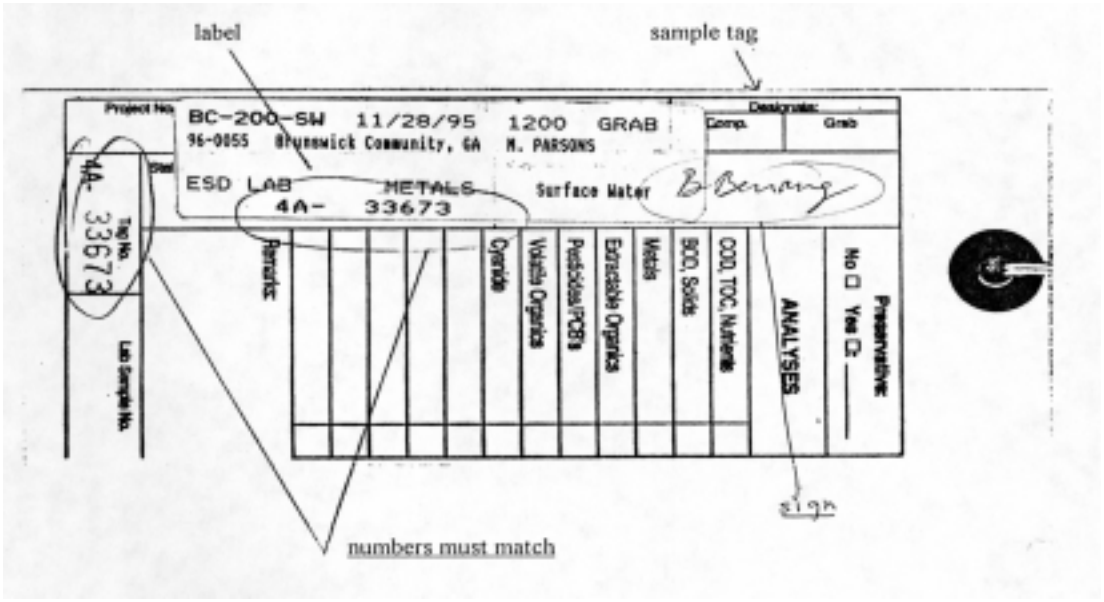
Wear latex gloves while processing sediment.

Required Supplies:

Sample tags
Custody seals (signed and dated)
Appropriate size clear plastic bags for sample containers
Electrical tape

Procedure:

1. After each label is generated using FORMS, place each label on a sample tag, taking special care to match the number on the FORMS label with the number on the sample tag (see below).



2. Have the on-site project officer or the person who collected the sample sign the sample tag in the appropriate box (see above).

3. Seal samples if necessary. Example: water samples 500 ml Storemore® bottles should be sealed with electrical tape stretched around the lid.

4. Tie the sample tag onto the appropriate sample container. Make sure that the suffix (matrix type) on the sample identification # matches the sample type. Example: M2-001-SD tag is tied to a sediment sample.

5. After the sample tag has been attached, fix a signed and dated custody seal over the lid and onto the jar or bottle.

6. Place the sample container with tag in a clear plastic bag and seal with electrical tape. Then place the sample in a holding container (eg. refrigerator at 4°C) or ship.

SHIPPING SAMPLES

Required supplies:

Large clear plastic bags for bagging ice
Large trash bags to line shipping containers packing
Adequate number of shipping containers (e.g. ice chests) for shipping samples
Strapping tape (or Duct tape)

Procedure:

1. All shipping containers (ice chests) should be clean and dry.

2. Place samples in a plastic trash bag inside the shipping container. Add packing material such as Vermiculite® to the bag if necessary, especially when shipping glass containers. Seal the bag with electrical tape. Allow enough room around the bagged samples for plenty of ice, if required, or additional packing material.

3. If ice is required, double bag all ice to insure no leakage. Take special care to insure that there is no leaking water or moisture coming from the ice chest when shipping. Federal Express is very particular about leaky ice chests and can and will stop shipment if a leak is detected. Place double-bagged ice in a separate trash bag, seal with electrical tape, and place on top of samples.

4. Put chain of custody forms in a clean dry bag and tape to the inside lid of ice chest.

5. Seal the ice chest with strapping tape. Wrap tape around each end of ice chest.

6. Place a custody seal on two opposite corners of ice chest.

7. Attach shipping weigh bill to top of ice chest. If shipping more than one ice chest to the same location label ice chest 1 of 3, 2 of 3...etc.

TURBIDITY TESTS

Turbidity tests are performed using an HF Scientific Inc. DRT-15C nephelometer. Operating instructions are posted inside the lid of the instrument.

Procedure:

1. Turn machine ON.

2. Insert vial of standard (provided) into reading chamber. Slowly rotate vial until the NTV digital readout displays a minimum value.

3. Adjust NTV readout to 0.02 by turning the appropriate knob on the machine. Remove standard. Machine is ready to read samples.

4. Mix sample by inverting 125 ml sample bottle 6-8 times. Immediately pour sample into clean vial (provided) and insert into reading chamber. Read NTV display and record.

Notes:

Wipe vials clean of fingerprints and dirt before inserting into reading chamber.

Read two subsamples (replicates) of each sample.

Keep sample well mixed between replicate readings.

Between samples, rinse vial with dH_2O and then with an aliquot of the next sample.

ALKALINE PHOSPHATASE ASSAY

1. Switch fluorometer (Guilford Fluoro IV model 1452 $_{X}$ 11) and printer "on" to warm up.

2. Remove reagents (MF and MFP) from freezer and hold in hand or put in pocket to warm to room temperature. PROTECT FROM LIGHT!

   MF (1mM MF reagent in methanol) is used to generate standard curve (step 6). Stored in glass scintillation vial.

   MFP (1mM MFP in 100 ml Tris buffer ph = 8.7) is added to samples (step 7). Stored in plastic microfuge tube.

3. Pipette 3 ml Trizma buffer into each of 4 clean styrene cuvettes.

4. Set machine to read APtase. Start by placing one of the above cuvettes (from step 3) in position #2 in the machine (slide positioning lever to the 2 position).

   a. Set wavelenth
      Press "2" ENTER
      excitation 430 ENTER
      emmision 507 RETURN

   b. Set response time
      Press "3.5" ENTER
      4 RETURN

   c. Calibrate machine (w/ cuvette still in position #2)
      Press "Calibrate" and wait for number to appear.
      Press "3.2" ENTER
      425 RETURN (sets high voltage)

5. Move cuvette (and positioning lever) to position #1

   Press "Read Print" and see if readout (on printer) is zero.
   If not, press "Autoblank" until it reads zero.

6. Prepare standard curve.

   To the 4 cuvettes containing 3 mls Trizma buffer (from step 3) add:

   add 3.0 μl MF to 1st tube, mix w/ transfer pipette, "Read Print"
   add 7.5 μl MF to 2nd tube, " "
   add 15 μl MF to 3rd tube, " " (continued on next page)
   add 30 μl MF to last tube " "

   (30 μl MF standard should read between 121.8 and 133.8)

   Fill-out log book

7. Run samples

Add 3 mls sample to a cuvette
Add 30 μl MFP to cuvette, mix w/ transfer pipette, "Read Print"

Place tube in incubator for 2 hours (minimum)
Read again.

(Note: for more than one sample, cuvettes are not labelled individually but are arranged in cuvette box sequentially)

INTERSTITIAL SOIL WATER SAMPLING PROTOCOL: SOUTH FLORIDA ECOSYSTEM ASSESSMENT PROJECT (PHASE II) REMAP

by

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and
Leonard Scinto and Ron Jones
Southeast Environmental Research Center, Florida International University, Miami, Fl

Introduction

A component of the Everglades Ecosystem Assessment (Phase II) will include experimental sampling of an array of interstitial soil (porewater) water samples at each spatially distributed randomized site. Approximately 125 sites will be sampled during the May (dry season) and another 125 sites will be sampled during the September (wet season) survey. This protocol has been modified from that developed by L. Scinto and R. Jones, FIU/SERC for the SERC Flume Project.

Objectives

1. To modify and implement (in research mode) an interstitial soil water sampling protocol which is compatable with the ecosystem scale REMAP probability sampling design.

2. To determine the extent and magnitude of an array of interstitial soil water nutrients (NH4, NO2, NO3, PO4), selected anions (Cl, Br, etc.), and sulfide.

3. To establish a baseline porewater condition against which future monitoring and assessment can be compared.

4. To explore the existence and significance of porewater spatial gradients.

5. To determine associations among porewater gradients and surface water gradients taken at the same time.

6. And, to determine, associations among constituents in porewater, surface water, soil and plant indicator species responses occurring in the ecosystem.

Protocol

Water Sample Containers

Soil interstitial water (SI) is collected into clean 30 ml high-density polyethylene (HDPE) bottles (Nalgene #2089-0001-Fisher Scientific, FS# 03-313-2A).

Soil Interstitial Water Collection

The soil interstitial water is collected via a soil interstitial water sampler (Sipper). Sippers consist of a filter (nominal porosity = 60 um) (Porex 6810, Interstate Specialty Products) held onto a male slip connector (Cole-Parmer #E-06359-05) with telfon tape. The slip connector

is attached to a hollow tube (1/16" ID x 1/8" OD, Tygon, FS# 14-169-1B) approximately 65 cm in length, the distal end of which is connected to a capped female luer fitting (CP#E-06359-25). An array of five stainless steel sippers will be installed to a maximum depth of 10 cm with individual insertion tools for each. The array will be positioned so that the distance between sippers is not less than 30cm. The insertion tool is the primary modification on the original method to provide a fixed depth insertion with an associated soil surface sealing flange. The sipper and insertion tool will remain in place until each sample has been collected. The insertion tool is also designed with water tight extensions to 120cm to prevent surface water from running down the inside of the insertion tube during periods of high water.

Steps

1. Load each insertion tool with a sipper with attached tubing.
2. Locate five sites at least 30 cm apart which are relatively free of standing vegetation.
3. Press the insertion tool into the soil firmly assuring the flange is tight against the soil surface with the flange ring imbedded into the soil. Note: A 9 cm diameter by 2.5 cm deep sharpened ring was added to the bottom side of the flange to increase contact with the soil surface. In addition a one way flapper valve was installed in the flange to allow surface water and gases to exit the sampler during installation.
4. Use the insertion rod to push the sipper into the undisturbed soil 4 cm to a depth of about 10 cm.
5. Connect a syringe (60 ml) to the female luer fitting on each sipper.
6. Apply suction and pull ten ml into the syringe. The void volume of an empty sipper, with 65 cm tubing, is approximately 1.5 ml. Pulling slightly greater volumes than this assures flushing. Disconnect syringe from luer fitting and attache a filter (Whatman GF/F, FS# 09-874-64 in syringe filter-holder Gelman FS#09-730-2250). Filter this water into the sample bottle in three 3 ml increments as rinse water discarding each in succession. Reapply suction to collect approximately 30 ml of pore water. If collection is difficult place binder clips on syringe such that suction is held and allow time for sample to be obtained.
7. Extract only one water sample from each of the five sippers for NO2, NO3, PO4; NH4; selected anions; and sulfide. Four samples will be collected leaving the fifth sipper in reserve in case a problem develops during sampling.
8. The syringe used for the sulfide sample will be pre-loaded with a zinc acetate/sodium hydroxide solution into which the sample will be drawn. Once 30 ml has been drawn into the syringe it will be disconnected, capped, labeled and placed in a cooler for transport.
9. All samples will be stored on ice until returned to the laboratory the same day.
10. Soil interstitial water samples (nutrients and anions) will be stored either in a refrigerator and analyzed immediately (<24 h) or stored frozen until analyzed (<30 d). Preserved sulfide samples will be analyzed immediately upon return from the field.
11. If surface water depth exceeds the height of the insertion tube add extension at step 1 and proceed as directed. The amount of water for flushing will be increased to 15 ml when the extension is used.
12. This procedure will be repeated consistently across all randomly selected REMAP sample sites in the ecosystem

Figure 1. Porewater sampler.

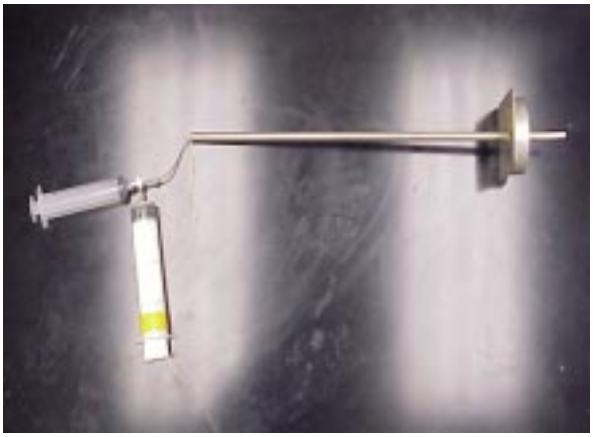

Figure 2. Sampler prior to insertion in soil.

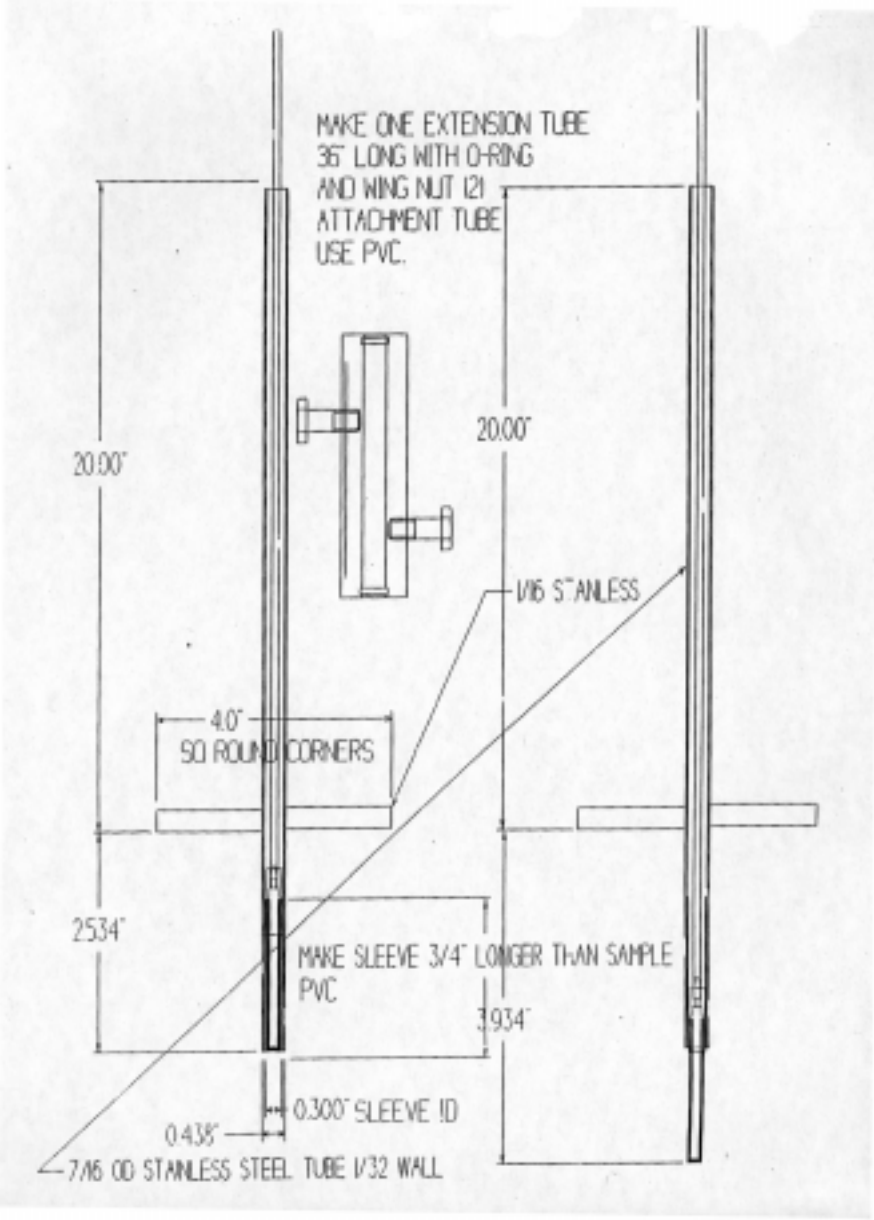

Figure 3. Design drawing of sampler.

Attachment 4

Analytical Support Branch Operations and Quality Control Manual - SESD, Region IV

ANALYTICAL SUPPORT BRANCH

OPERATIONS AND QUALITY CONTROL MANUAL

ENVIRONMENTAL PROTECTION AGENCY
SCIENCE AND ECOSYSTEM SUPPORT DIVISION
REGION 4
980 COLLEGE STATION ROAD
ATHENS, GA 30605

DISCLAIMER

The mention of trade names or commercial products in this manual is for illustration purposes, and does not constitute endorsement or recommendation for use by the Environmental Protection Agency.

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1. INTRODUCTION

1.1 This manual is designed to delineate the routine operation of the USEPA Region 4 Analytical Support Branch. The primary purpose of this document is to establish and maintain uniform operational and quality control guidance for regional analytical chemistry activities, contractor laboratory monitoring/performance, and quality assurance/quality control activities. The establishment of, and adherence to, uniform elements of an intralaboratory quality control program are essential to the production of reliable analytical data.

1.2. Coordination of Region 4 quality assurance activities and likewise, the Analytical Support Branch (ASB), rests primarily with the Region 4 Quality Assurance Officer (QAO). The functions and responsibilities of the QAO are identified in the Region 4 Quality Assurance Program Plan. The QAO functions as a focal point for the dissemination of information and provides program managers with technical advice pertaining to the development, implementation, and operation of quality assurance activities. Implementation of agency quality assurance policies applicable to the Analytical Support Branch laboratory is the responsibility of the Chief, Analytical Support Branch.

1.3 While the implementation of quality assurance policy is a management function, each individual staff person has a responsibility for the operational aspects of the quality assurance. It is the individual responsibility of each analyst and his/her supervisor to monitor quality control indicators and to provide for corrective actions when necessary.

1.4 This manual and the quality control protocols described herein are not to be viewed as all inclusive. Rather, they serve as a basic foundation on which to build a stronger quality assurance program within the Branch. Methodologies and some quality assurance documents are included by reference.

2. BRANCH ORGANIZATION AND OPERATION

2.1. The Analytical Support Branch organizational structure is shown in Figure 2-1.

2.2. The Branch is a technical support activity with the following functions:

2.2.1. Provides chemical laboratory services in support of all regional program needs.

2.2.2. Provides consultation and assistance to local, State, and other agencies in matters of analytical methodology and laboratory quality assurance.

2.2.3. Provides personnel as regional representatives to national programs relating to selection, validation, and promotion of the use of official EPA analytical methods.

2.2.4. Participates in national and regional interlaboratory method evaluation studies.

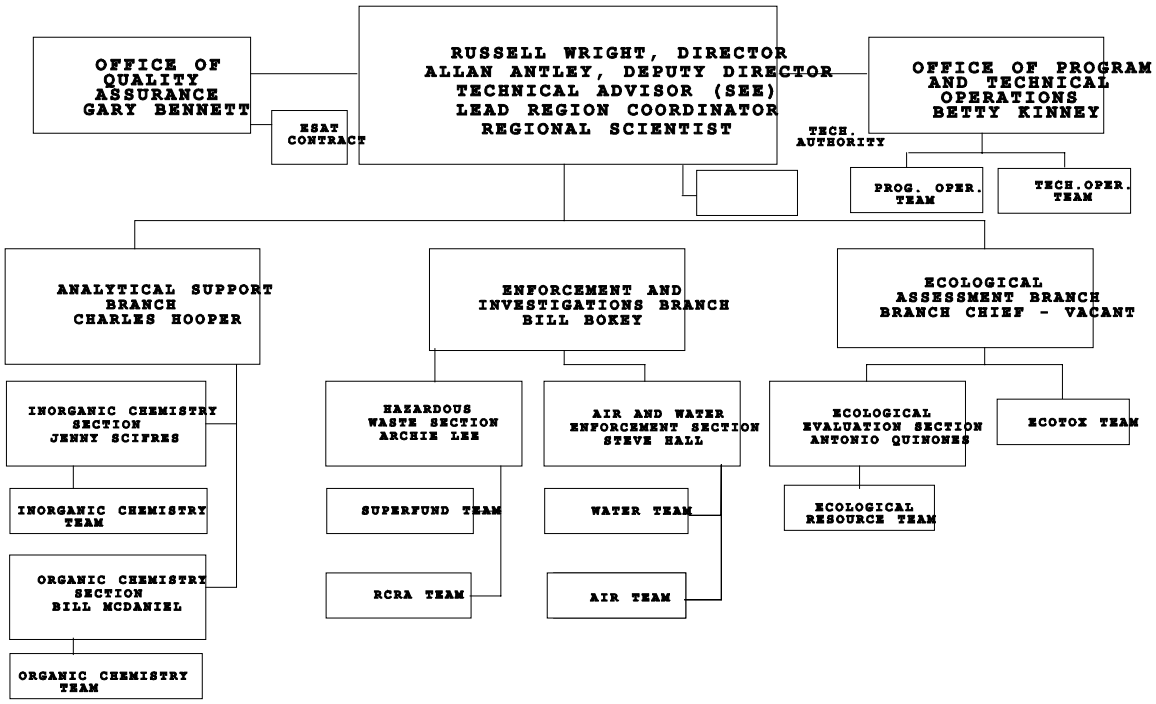

SCIENCE AND ECOSYSTEM
SUPPORT DIVISION
OFFICE OF QUALITY ASSURANCE GARY BENNETT
ESAT CONTRACT
RUSSELL WRIGHT, DIRECTOR
ALLAN ANTLEY, DEPUTY DIRECTOR
TECHNICAL ADVISOR (SEE)
LEAD REGION COORDINATOR
REGIONAL SCIENTIST
TECH. AUTHORITY
OFFICE OF PROGRAM AND TECHNICAL OPERATIONS BETTY KINNEY
PROG. OPER. TEAM
TECH.OPER. TEAM
ANALYTICAL SUPPORT BRANCH CHARLES HOOPER
ENFORCEMENT AND INVESTIGATIONS BRANCH BILL BOKEY
ECOLOGICAL ASSESSMENT BRANCH BRANCH CHIEF - VACANT
INORGANIC CHEMISTRY SECTION JENNY SCIFRES
INORGANIC CHEMISTRY TEAM
ORGANIC CHEMISTRY SECTION BILL MCDANIEL
ORGANIC CHEMISTRY TEAM
HAZARDOUS WASTE SECTION ARCHIE LEE
SUPERFUND TEAM
RCRA TEAM
AIR AND WATER ENFORCEMENT SECTION STEVE HALL
WATER TEAM
AIR TEAM
ECOLOGICAL EVALUATION SECTION ANTONIO QUINONES
ECOLOGICAL RESOURCE TEAM
ECOTOX TEAM

Figure 2-1

3. LABORATORY CHAIN-OF-CUSTODY

3.1. Introduction

3.1.1. Complete documentation of the sample collection and handling process is an extremely important aspect of a regulatory monitoring effort. Formal chain-of-custody procedures provide for a written record of sample traceability, accountability, and serve to validate sample integrity. All samples received by ASB for chemical analysis are controlled by these procedures. Field sample custody procedures are detailed in the Enforcement Investigations Branch, Standard Operating Procedures and Quality Assurance Manual.

3.1.2. All custodial documentation on samples for ecological/ biological analyses will be maintained within the records of the project biologist. The Analytical Support Branch sample custodian will not maintain these records.

3.2. Sample Custody Forms

3.2.1. The following sample custody and disposal forms are shown in Form 3-1 through 3-5:

3.2.1.1. Chain-of-Custody Seal (Form 3-1).

3.2.1.2. Chain-of-Custody Record (Form 3-2).

3.2.1.3. Custody Room Sample Log (Form 3-3).

3.2.1.4. Diagram of Custody Room (Form 3-4).

3.2.1.5. Disposal Memo Form (Form 3-5).

3.2.2. In addition to these forms, custody information is maintained in the master logbooks, Data Management System, computer sample log, the chemistry field logbooks, and in the individual analytical data books.

3.3. Standard Operating Procedure - Sample Receipt/Custody

3.3.1. Samples are received by the sample custodian or a designated alternate. The alternate must be an EPA employee on the staff of the Analytical Support Branch. Samples that arrive after hours will be secured in the custody room and the sample custodian will receive them the next business day. At the time of receipt, the custodian or designee will perform the following actions:

3.3.1.1. Sign the chain-of-custody form and record the date and time of sample receipt.

3.3.1.2. Document whether the individual samples, boxes, or ice chests were sealed upon receipt; also document unusual conditions of sample container in remarks section of the custody form.

3.3.1.3. Log all samples into the Data Management System.

3.3.1.4. Place sample numbers on all sample tags or containers and secure samples in the area designated for new samples. The designated area will be the middle shelf in the front entrance of each walk-in cooler. Walk-in coolers will be labeled as follows:

3.3.1.4.1. Metals/Organics: Storage for metals samples, extractable organics and pesticide samples.

3.3.1.4.2. Ultra Low Level: Storage for Metals, extractable organics, pesticide and classical ultra low level samples such as drinking water. Note: In special cases some low level samples will be stored in refrigerators in the laboratories such as the low level mercury samples.

3.3.1.4.3. Classical Inorganic/EAB: Storage for samples for classical analyses and Ecological Assessment Branch samples.

3.3.1.4.4. The walk-in freezer will be divided equally between the ASB and EAB for frozen samples.

3.3.1.4.5. Volatile organic samples will be stored in a secured refrigerator in the GC/MS laboratory.

3.3.1.5. After samples are placed in the area designated for new samples the following will be performed:

3.3.1.5.1. As soon as possible, the analyst(s) will move the samples onto their allotted shelves and place them in a manner that is functional for their team. See Form 3-4. Each team is responsible for keeping their area of the custody room secure, orderly, neat and maintaining space for incoming sample placement.

3.3.1.5.2. It is the responsibility of the sample custodian to insure that all areas of the custody room are maintained in a clean, orderly and secure manner.

3.3.1.6. After sample logging is completed, computer data reporting information will be available in the Database Management System for reference.

3.3.1.7. The original field custody form, along with a computer printout of the requested analytical tests, will be maintained in the ASB files. A copy of the field custody form and a copy of the computer print out will be sent to the project leader responsible for sample collection.

3.3.2. Access to the main custody room area will be by computer card as authorized by the Chief of the Analytical Support Branch or Chief of the Ecological Assessment Branch.

3.3.3. For an analyst to receive samples for analysis, he/she must assume legal custody of the samples and the following actions are required:

3.3.3.1. The analyst must complete the appropriate Custody Room Sample Log including their initials, listing the sample numbers, date and time. Samples may be removed from the custody area only after performing the appropriate documentation transferring custody to the analyst. There will be 4 separate log books: 1) metals, 2)extractable and pesticide, 3) volatile and 4) classical and inorganics. Records of samples to be stored by the Ecological Assessment Branch will be maintained in logbook #4 (for classical and inorganic chemistry analyses.)

3.3.3.2. The analyst will return the samples to the custody room when he/she is finished with the analysis. In no case will the original samples (less aliquot required for analysis) remain outside the custody room during non-duty hours. When the samples are returned, the analyst will note the date and time returned in

the appropriate Custody Room Sample Log, returning custody back to the sample custody room.

3.3.3.3. The Custody Room Sample Logs will be maintained as a permanent file.

3.4. Audit of Custody Records

3.4.1. Audits of custody information will be performed by the Branch Chief or designee. These audits will include an examination of custody documentation of randomly selected samples for traceability, completeness, and accuracy. The results of these audits will reflect the general effectiveness of the custody procedures.

3.5. Policy for Disposal of Laboratory Samples

3.5.1. No criminal investigation samples, extracts or sample containers will be disposed of until authorized by the appropriate officer of the court. Due to the timing on litigation, criminal samples usually require long term storage. Space limitations within the custody room make it necessary to store criminal samples within the HAZMAT facility using the following procedure: 1) At the completion of all required analyses the “characterization report” will be generated, denoting the sample as to its hazardous or non-harzadous status. 2) A copy of the characterization report and custody of the samples will be transfered to the Divisional SHEM Officer. 3) The SHEM Officer will maintain custody of the criminal samples while in storage and will coordinate disposal with all appropriate parties.

3.5.2. Samples and their extracts that are not part of a criminal investigation will normally be disposed of within 90 days from the completion of the final laboratory data report. The exception to this will be when a sample hold request is implemented. The "Intent to Dispose of Samples Memo" (Table 3-5) will be prepared and sent out by the Sample Custodian or designee as desingated below.

3.5.2.1. Intent To Dispose Memo and Sample Hold Request

30 Days after all laboratory analyses have been completed and the data reported, the sample custodian will submit the disposal memo (form 3-5) to the appropriate project manager. The memo will be submitted via cc mail with receipt requested. The sample custodian will maintain a file of receipts to document the notice received by the project manager. To place samples on hold (non-disposal) the project manager must so note on the form memo in the appropriate place, list why the hold is necessary, and return the memo to the sample custodian.

3.5.2.2. The sample custodian or designee will monitor samples requested for hold and the samples ready for disposal. Samples ready for disposal will be entered into the Database Management System program which then generates a list of compounds found in each sample. This “compound list” is used to identify those samples that may be disposed of as ordinary environmental samples and those that are defined as “hazardous” by regulation.

3.5.2.3. Samples that qualify as hazardous are documented within the Data Management System Program and a list is generated. Those samples characterized as hazardous will be coordinated with the Science and Ecosystem Support Division (SESD), Safety Health and Environmental Management (SHEM) Officer for disposal. Refer to Chapter 4 for more details on hazardous waste disposal.

3.5.3. For those samples characterized as non-hazardous (routine environmental), a disposal report will be generated and provided to designated staff as appropriate. Sample disposal of the routine environmental samples should be completed by the appropriate analyst within 2 weeks from disposal report distribution. The routine environmental samples will be disposed of in the following manner:

3.5.3.0.1. The tags are removed, sorted and sent to the sample collectors.

3.5.3.0.2. Water samples are disposed of by pouring the water down the sink drain and rinsing the containers out with water. These containers will be recycled. **Preserved samples must be neutralized.** Each person disposing of samples must maintain an awareness of the status of the laboratory centralized neutralization system. If the neutralization system is under maintenanace and/or not functional, the preserved waters must be neutrailzed before flushing down the sink.

3.5.3.0.3. Non-hazardous soil/sediment samples are disposed of in the dumpster.

3.6. Special Sample Handling Instructions

3.6.1. Soils from Foreign Countries

On occasion the Analytical Support Branch may receive requests for analyses of foreign soil samples. Such samples require special handling for labeling and disposal. The following procedure should be followed:

3.6.1.1. When booked into the data management system there must be a special notice of the fact that the samples are of "FOREIGN SOIL".

3.6.1.2. When the samples are received at the laboratory the sample custodian or designee is responsible for labeling the tags with the notation "FOREIGN SOIL". The tag must remain with the sample container until project completion and sample disposal.

3.6.1.3. After the samples are tagged and logged they should be stored in the custody area per standard procedures.

3.6.1.4. Unused original sample must be autoclaved prior to disposal for at least 30 minutes at 121 C and 15 psi.

3.6.1.5. After autoclaving the samples may be disposed of using standard procedures for environmental samples. **NOTE: ASB will not routinely accept foreign soil/sediment samples suspected to be hazardous as defined by statute. However, in the unlikely event that the test results indicate that they are "hazardous", disposal should be coordinated with the SESD SHEM Officer. Refer also to Chapter 4 for additional details of disposal of samples characterized as "hazardous".**

3.6.2. Handling Procedures for Potentially Hazardous Waste Samples in the Laboratory

A small percentage of samples received by ASB are characterized as concentrated waste. In these instances field personnel are required to screen the waste materials to ensure safe transportation and handling of the samples. Concentrated waste

samples are not preserved and are not required to be cooled to 4 degrees C.

The waste samples should be in a primary container that has been cleaned by field personnel to insure no contamination to the exterior of the container. The samples should then be tagged, sealed and placed within a plastic bag secured with electrical tape. Each sample will then be placed within a 6-quart plastic pail with a spill proof, tight fitting lid and packed with vermiculite as a secondary containment. There should be special notations to the sample custodian as to the hazardous nature of the samples. It is the Sample Custodian responsibility to insure that the hazardous nature of the samples is communicated to all ASB staff. Concentrated waste samples will be stored within the hood of the custody room.

3.6.2.1. When concentrated waste samples are received at the Laboratory the following procedure should be followed for the storage and handling:

3.6.2.1.1. Samples will be signed out of the designated storage area and chain of custody maintained as with routine environmental samples.

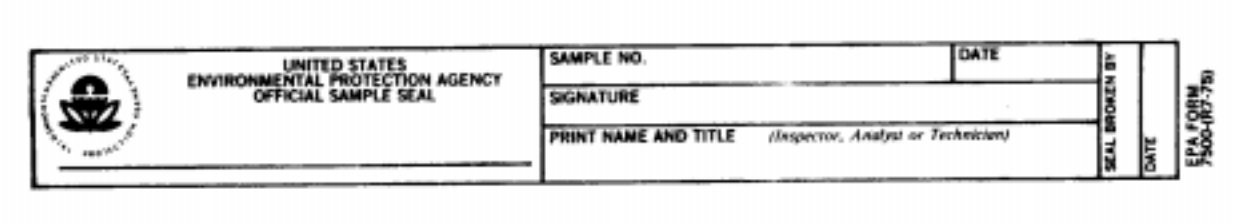
3.6.2.1.2. The samples will be transported unopened within the laboratory and placed in the appropriate preparation area **within a fume hood.** Samples should never be transported outside a fume hood unless they are sealed within the secondary containment vessel.

3.6.2.1.3. Once inside the hood, the secondary containment may be opened and the individual sample processing may begin. Care should be taken to keep the secondary containment vessel so that the original sample may be repacked after completion of the preparation for analysis. **All sample processing and manipulation should be accomplished within the hood. At no time should the raw sample be removed from the hood without being properly repacked within the primary and secondary containment vessels.**

3.6.2.1.4. Personal Protective Equipment (PPE): All initial preparation/aliquoting of the samples must be performed using the following personal protective equipment at a minimum: 1) Latex or other type of appropriate gloves; 2) Lab Coat; 3) Safety glasses or safety face shield. Higher levels of PPE may be required as determined by information received from field personnel, knowledge/experience of the analyst, or lab supervisor. These determinations will be made by results of field screening and any additional knowledge of the sample matrix. **It is the responsibility of each analyst to insure that appropriate methods and safe laboratory practices are followed at all times. If at any time an analyst has a concern about the preparation process, or if unsure about their ability to safely handle the samples, they should immediately contact their supervisor.**

3.6.2.1.5. Any glassware or equipment such as spatulas, pipets, droppers, etc. used in contact with the concentrated waste must remain within the hood until cleaned or disposed of in an appropriate fashion. These may be placed in the secondary containment container to be disposed of with the samples. Any solvents, solutions, or materials (kimwipes,etc) used to clean waste from glassware or other equipment must be collected and treated the same as the waste material. Where practical and prudent for the analytical method, choose items that are disposable. **In all cases consult with the Divisional SHEM Officer before removing and discarding any of the contaminated materials.**

3.6.2.1.6. At the completion of the sample processing the original samples should be repacked into the primary and secondary containment with care taken to insure that there is no waste contamination on the exterior of the containment vessels. When properly repacked, the samples should be returned to the custody room hood and custody is returned by signing the appropriate logbook. **Refer to Chapter 4 for details of the disposal of hazardous waste samples.**



Chain-of-Custody Seal
Form 3-1

CHAIN OF CUSTODY RECORD

EPA REGION 4
U.S. ENVIRONMENTAL PROTECTION AGENCY

ENVIRONMENTAL SERVICES DIVISION
COLLEGE STATION ROAD
ATHENS, GEORGIA 30613-7799

PROJECT NO. | PROJECT LEADER | REMARKS

PROJECT NAME/LOCATION

ESD SAMPLE TYPES

1. SURFACE WATER
2. GROUND WATER
3. POTABLE WATER
4. WASTEWATER
5. LEACHATE
6. SOIL/SEDIMENT
7. SLUDGE
8. WASTE
9. AIR
10. FISH
11. OTHER _____

SAMPLERS (SIGN)

TOTAL CONTAINERS

CIRCLE/ADD parameters desired. List no. of containers submitted

ANALYSES: COD, TOC, NUTRIENTS | BOD, SOLIDS | METALS | EXT ORG/PEST/PCBs | VOA | CYANIDE

STATION NO.	SAMPLE TYPE	19__ DATE	TIME	COMP	GRAB	STATION LOCATION/DESCRIPTION	TAG NO./REMARKS	LAB USE ONLY

RELINQUISHED BY: (PRINT) / (SIGN)	DATE/TIME	RECEIVED BY: (PRINT) / (SIGN)	RELINQUISHED BY: (PRINT) / (SIGN)	DATE/TIME	RECEIVED BY: (PRINT) / (SIGN)
RELINQUISHED BY: (PRINT) / (SIGN)	DATE/TIME	RECEIVED BY: (PRINT) / (SIGN)	RELINQUISHED BY: (PRINT) / (SIGN)	DATE/TIME	RECEIVED BY: (PRINT) / (SIGN)

DISTRIBUTION: White and Pink copies accompany sample shipment to laboratory; Pink copy retained by laboratory; White copy is returned to samplers; Yellow copy retained by samplers.

*U.S. GPO: 1989-732-186

4- 23754 (10/89)

Chain-of-Custody Form
Form 3-2

Book 15

CUSTODY ROOM
SAMPLE LOG

SAMPLE #	PARAMETER	OUT		IN		DATE	NAME
		DATE	TIME	DATE	TIME	DISPOSED	

Chain-of-Custody Form
Form 3-2

Custody Room Sample Log
Form 3-3

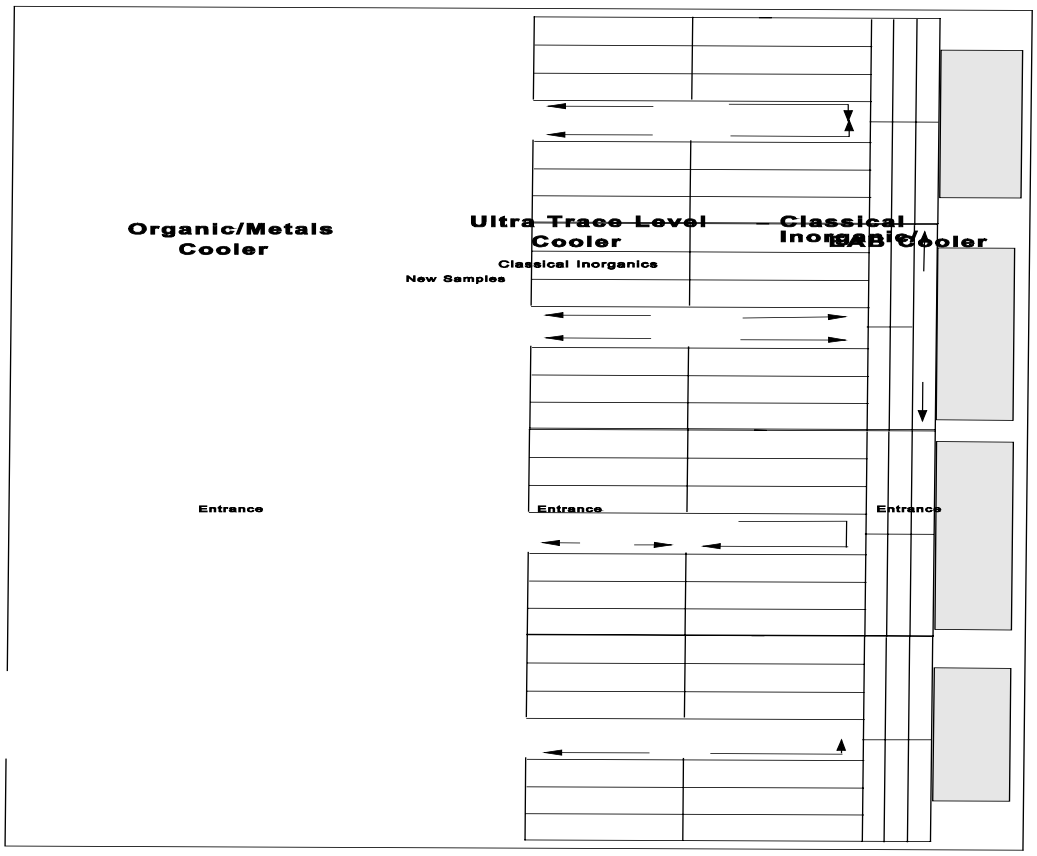


Diagram of Custody Room
Form 3-4

M E M O F O R M

SUBJECT: Notice of Intent to Dispose of Samples; (Sample Project Name; SESD project no., city, state)

FROM: Person sending memo (i.e. sample custodian/coordinator);.

TO: Project manager

This memorandum is being sent as a reminder that the Analytical Suppport Branch has completed all analyses on the subject samples. Due to our limited space for long term sample storage, we must proceed with sample disposal. Please take note that within sixty (60) days of the date of this memo the original samples will be disposed of following all applicable and appropriate statutes.

If there is any reason to hold these samples in custody for longer than 60 days, you may activate a "hold" by so indicating below and returning this memorandum via cc mail to Debbie Colquitt within the next 30 days. Also, please state briefly the reasons for retaining these samples in custody.

Thank you for your cooperation in this request.

Date:

Project Manager Name:

Reason for Hold:

4. GENERAL LABORATORY PRACTICES

4.1. Intrinsic to the production of quality analytical data is the quality of laboratory services available to the analyst. Without adequate quality control being exercised with regard to facilities, services, laboratory environment, instrumentation, and laboratory supplies, an analyst cannot be expected to produce reliable analytical data.

4.2. Recognizing the necessity of maintaining control over general laboratory operation, the subsequent sections outline provisions for maintaining the quality laboratory support services.

4.3. All quality control checks listed in this section should be recorded in the appropriate logbook or file (printed or electronic).

4.4. Laboratory Apparatus and Instruments

4.4.1. Incubators and Waterbaths

4.4.1.1. If an automatic temperature recorder is not used, place calibrated thermometer on a central shelf and record temperature at least once daily (more frequently if required) when the incubator is in use.

4.4.1.2. Periodically check temperature variations when incubator or waterbath is loaded to capacity.

4.4.1.3. Drain and clean waterbath as required and refill with laboratory pure water.

4.4.2. Refrigerators and freezers

4.4.2.1. Check and document temperature weekly.

4.4.2.2. Clean periodically and discard outdated materials.

4.4.2.3. Do not store food in any laboratory refrigerator or freezer. There is a refrigerator in the lunchroom for storage of food.

4.4.3. Autoclave and Hot Air Oven

4.4.3.1. Record date, and sterilization time, and temperature for each cycle.

4.4.3.2. Operate hot air oven at a minimum of l70^{o}C for sterilization.

4.4.4. Balances

4.4.4.1. Check with Class-S weights at least monthly and record in the QC log.

4.4.4.2. Clean and level balances as required.

4.4.4.3. Maintain annual maintenance services contract.

4.4.5. pH Meters

4.4.5.1. Date all pH buffer solutions when opened. Buffers that have reached the manufacturer's expiration should be discarded and replaced.

4.4.5.2. Standardize meter with pH 7.0 and pH 4.0 and/or pH 10 buffer before each use, or as required by regulated methods.

4.4.5.3. Use pH buffer aliquot only once.

4.4.6. Thermometers

4.4.6.1. Unless otherwise specified by regulatory methodoloy, it is the policy of ASB to use only non-mercury containing thermometers in all laboratory operations. Check all laboratory thermometers annually with a reference NIST thermometer. Mark any necessary corrections on each thermometer and record in the QC logbook.

4.5. Laboratory Supplies

4.5.1. Glassware

4.5.1.1. Glassware used in general laboratory operations must be of a high quality borosilicate glass, e.g., "Pyrex" or "Kimax." Volumetric glassware must be of a Class "A" quality.

4.5.1.2. Clean glassware in hot water with a suitable detergent, rinse in hot water to remove detergent residue, and finally rinse in laboratory pure water. Glassware used in special analyses, e.g., metals and organics require more scrupulous cleaning, e.g., acid and/or solvent washing. Glassware must be oven-dried or drained thoroughly before use or storage. Glassware used in trace metals analysis should be air dried. In some instances it may prove to be advantagous to store labware for ultrace level metal analyses in a dilute acid solution. In operations of specific, low-level analyses glassware should be isolated and maintained only for these specific operations.

4.5.1.3. If, at any time a new washing compound or cleaning application is introduced, it is imperitive that tests be performed to assure that the glassware is free of intereferences before routine analyses are begun.

4.5.2. Chemicals, Reagents, Solvents, Standards, Gases, and Culture Media

4.5.2.1. The quality of chemicals, reagents, solvents, standard gases, used in the laboratory is determined by the sensitivity and specificity of the analytical techniques being used. Reagents of lesser purity than specified by a method will not be used.

4.5.2.2. Reagents, chemicals, solvents, and standard reference materials (excluding high-demand items) should be purchased in small quantities to minimize extended shelf storage.

4.5.2.3. Date all reagents, chemicals, solvents, and standard reference materials when received and when opened or prepared, and

discard when outdated, or when evidence of discoloration or deterioration is detected.

4.5.3. Laboratory Pure Water

4.5.3.1. The laboratory pure water system consists of a deionization supply followed in individual labs by exchange modules and other modules capable of supplying high quality (18 megaohm) water suitable for the application. The system is also equipped with a direct reading resistivity meter.

4.5.3.2. Change system modules as recommended by the manufacturer or as indicated by water quality. Date modules when changed.

4.6. Laboratory Hazardous Wastes Handling and Disposal Procedures

4.6.1. It is the policy of the Analytical Support Branch to collect, store, package, label, ship and dispose of hazardous wastes in a manner which ensures compliance with all Federal, State and local laws, regulations and ordinances. These procedures are also designed to minimize employee exposure to hazards associated with laboratory generated hazardous wastes and to afford maximum environmental protection.

4.6.2. Policies and procedures for operation of the Division's environmental compliance program are detailed in the document, Safety Health and Environmental Management Program, Procedures and Policies Manual. This manual is maintained by the Divisional Safety, Health and Environmental Managment (SHEM) Officer.

4.6.3. Regulatory Requirements

4.6.3.1. ASB is subject to the Resource Conservation and Recovery Act regulations as contained in the Georgia Rules for Hazardous Waste Management for the handling, storage and disposal of laboratory-related hazardous wastes. Generally, the laboratory is subject to the rules applicable to generators of 100-1000 kg/month.

4.6.4. Waste Handling Practices

4.6.4.1. Hazardous Waste Determination. The determination of whether or not a waste is a regulated substance is made by the Divisional SHEM Officer. Generally the following criteria apply either individually or in combination:

4.6.4.1.1. Is the waste material listed in 40 CFR 261.30 - 261.33(e)?

4.6.4.1.2. Does the material conform to any of the listing characteristics specified in 40 CFR 261.20 - 261.24?

4.6.4.1.3. Does the generator have personal knowledge of the hazardous nature of the material?

4.6.4.1.4. Would disposal of the material as non-regulated waste pose an environmental threat and/or leave the Agency open to criticism?

4.6.4.2. Wastes which meet any of the above criteria must be handled and disposed of as a regulated waste.

4.6.5. Waste Minimization

4.6.5.1. The Branch Chief is responsible for ensuring that staff adhere to all Region 4 waste handling and disposal requirements for all laboratory operations. This includes the implementation of procedures (i.e., technical and/or management) designed to minimize the generation of hazardous wastes.

4.6.5.2. Waste minimization should be a prime consideration of initial experimental design and investigation planning. The degree to which waste minimization is achieved ultimately impacts the operational and cost effectiveness of our overall hazardous waste management program.

4.6.6. Tracking

4.6.6.1. A tracking system is maintained to account for monthly and annual hazardous waste generation. This system is maintained by the Divisional SHEM Officer.

4.6.6.2. Jim Gray, SHEM Officer, phone 355-8613, is responsible for waste logging, acceptance for storage, and periodic shipments as required by policies and procedures.

4.6.7. Waste Accumulation Limits

4.6.7.1. As a small quantity generator, the laboratory is subject to the following waste accumulation limits:

4.6.7.1.1. Hazardous waste

4.6.7.1.1.1. Generate no more than 1000 kg/mo and accumulate no greater than 6000 kg of wastes. Wastes must be disposed of within 180 days of the start of accumulation, or within 270 days if waste is transported more than 200 miles for disposal.

4.6.7.1.1.2. Wastes generated in excess of these limits subjects the laboratory to the full generator rules (40 CFR 262.34 (a)).

4.6.7.1.2. Acutely Hazardous Waste

4.6.7.1.2.1. Those wastes specifically listed in 40 CFR 261.31 and 261.33 (e)(f) are considered acute hazardous waste. The laboratory cannot generate more than 1 kg/mo of acute hazardous waste and retain its' small quantity generator status. The 180/270-day storage limit also applies to acutely hazardous wastes if less than 1 kg/mo is generated.

4.6.7.1.3. Waste accumulation will be monitored to ensure that the applicable generation and accumulation (i.e., quantity/time) limits are not exceeded. Waste will be disposed of as required to ensure conformance with the regulatory limits (i.e., 180 days) and at a minimum of twice per year.

4.6.8. Waste Packaging and Labeling

4.6.8.1. All hazardous wastes designated for temporary storage must be packaged in an appropriate container designed to avoid loss or spillage of the materials. The determination of the hazardous nature of a waste is the responsibility of the SHEM Officer.

4.6.8.2. Before transporting or offering a hazardous waste for storage the SHEM Officer must be consulted. The SHEM Officer will ensure that all containers shipped off-site are properly packaged and labeled and that the transport vehicle is appropriately placarded and manifest documentation is complete.

4.6.9. Waste Storage

4.6.9.1. Except for in-laboratory accumulation (i.e., satellite storage (40 CFR 262.34 (c)(1)), all hazardous wastes generated at the Region 4, College Station Road facility and accumulated for disposal will be stored in the Hazardous Materials (HAZMAT) Storage Facility . The HAZMAT facility is located adjacent to and detached from the main SESD building. The building is specifically designed for the storage of hazardous materials.

4.6.9.2. Materials stored in the HAZMAT are segregated according to compatibility groups.

4.6.9.3. The HAZMAT storage facility will be inspected on a weekly basis as required 40 CFR 265.15. These inspections and the required documentation thereof are the responsibility of the Divisional SHEM Officer.

4.6.9.4. Inspection of emergency equipment and spill control equipment will be conducted at appropriate intervals by the SHEM Officer.

4.6.9.5. Additional housekeeping and security inspections of the HAZMAT facility will be performed on a regular basis in conjunction with safety inspections conducted by the SESD Health and Safety Committee. An inspection report will be provided to the SHEMP Officer and to Divisional Senior Management.

4.6.9.6. Hazardous waste generated at EPA’s leased space at the US Department of Agriculture, Russell Reasearch Laboratory will be stored in the SESD HAZMAT Facility. This is appropriate due to the close proximity of the locations of the two laboratories.

4.6.10. Waste Disposal

4.6.10.1. Disposal of regulated laboratory wastes is the culmination of the waste management process. As such, selection of a responsible waste transporter and disposal facility is vitally important. The selection of a waste transporter must be predicated on their being permitted to transport hazardous wastes coupled with an absence of prior RCRA/DOT violations and a proven record of successful performance.

4.6.10.2. The method of waste disposal will, in part, dictate the selection of a waste disposal facility. To the extent possible, it will be the policy of Region 4 to dispose of all hazardous wastes by incineration, and/or chemical treatment/fixation. Landfilling of hazardous wastes will be avoided if at all

possible. Factors considered in the selection of a waste disposal facility include: current permit status, compliance with the EPA Off-Site Policy, (SARA Sec. 121), past performance, effectiveness of treatment processes and ability to provide a certificate of disposal. To the extent possible, all hazardous waste will be disposed of at facilities which comply with the EPA off-site policy.

4.6.10.3. Non-regulated solid wastes will be disposed of in the building dumpster. Non-regulated aqueous wastes will be flushed to the sewer system. See Section 3.5.42.4.2 for proper disposal. Spent sample containers disposed of in the dumpster should have their labels removed or obliterated.

4.6.11. Recordkeeping

4.6.11.1. All records related to the generation and disposal of hazardous wastes will be retained as permanent facility records.

4.6.11.2. These records will be maintained in the files of the Divisional SHEM Officer.

4.6.12. Contingency Measures

4.6.12.1. As required by 40 CFR 265.50 - 265.56, a Hazardous Waste Contingency Plan has been developed which outlines facility emergency response procedures.

4.7. Procedures for Satellite Hazardous Waste Accumulation

4.7.1. Many laboratory operations necessitate the generation of hazardous wastes (e.g., solvents, acids, etc.) which are routinely accumulated near the point of generation. The in-laboratory "satellite" accumulation of such waste should be carefully controlled by the laboratory manager working with the SHEM Officer so as to avoid creating an unsafe situation and also comply with RCRA temporary storage requirements.

4.7.2. The RCRA regulations (40 CFR 262.34(c)(1)) permit the temporary accumulation of hazardous waste or acutely hazardous wastes at or near the point of generation. Waste accumulated in this manner are considered to be in "satellite accumulation."

4.7.3. Hazardous Wastes. The following procedures apply to satellite accumulation of hazardous waste in ASB facilities:

4.7.3.1. All waste containers must be clearly marked with a red "Hazardous Waste" label. These labels are available from the SHEM Officer, Jim Gray, at 355-8613.

4.7.3.1.1. The contents of the container must be marked on the label. Be specific in the identification of the contents.

4.7.3.1.2. All satellite storage containers must be closed except during periods of waste transfer. Some operations (e.g., AA, LC, ICP, etc.) may require using a container lid with a hole for introducing the waste via a tube. Waste collection vessels requiring zero back pressure can be fitted with an open-to-the-air absorbent trap (e.g., carbon filled).

4.7.3.1.3. The volume of waste accumulated in the laboratory should not exceed 8 gallons. Exceptions would be instrument (i.e., AA, ICP) waste acid reservoirs and TCLP process waste.

4.7.3.1.4. Volatile and/or flammable wastes should be temporarily stored in laboratory fume hoods nearest the point of generation.

4.7.3.1.5. Caution must be exercised by the analysts to avoid creating incompatible and/or reactive waste mixtures.

4.7.3.1.6. Waste removed from "satellite" storage for disposal will be handled according to the procedures contained in the Safety, Health and Environmental Management Program, Procedures and Policies Manual.

4.7.3.2. All satellite accumalation containers must be placed in secondary containment.

4.7.4. Acutely Hazardous Wastes

4.7.4.1. Acutely hazardous wastes are those listed in 40 CFR 261.31-261.33 and must be accounted for separately from non-acute wastes. The following procedures apply to the satellite storage of acutely hazardous wastes:

4.7.4.1.1. The acute waste must be collected in separate containers from the non-acute hazardous waste and be labeled as containing acute waste.

4.7.4.1.2. Accumulation of acute waste cannot exceed one (1) quart and remain in the laboratory. Once the volume reaches one quart, the waste container must be dated and removed to the permanent hazardous waste storage area within three (3) days.

4.7.4.1.3. Except for the labeling and accumulation limits, acute wastes will be handled in the same manner as hazardous wastes.

4.7.4.2. Laboratory managers and supervisors should conduct periodic walk-through inspections to ensure the proper application of temporary waste accumulation procedures.

4.8. Guidelines for Disposal of Environmental Samples

4.8.1. Samples submitted to the laboratory for analysis are excluded from regulation as hazardous waste under 40 CFR 261.4(d) provided the samples are being transported to or from the laboratory, or are being analyzed, are being held for analysis, are being maintained in custody for legal reasons. However, once a decision is made to dispose of laboratory samples, the exclusion provisions of 40 CFR 261.4(d) no longer apply. Depending upon the characteristics and/or contents of such samples, they may be subject to regulation as a hazardous waste under RCRA or as a PCB-containing material under TSCA and must be handled accordingly.

4.8.2. Not all samples are routinely subjected to characteristic testing and are not readily classified as a hazardous waste. To address the problem of proper sample handling and disposal in the laboratory, guidelines have been developed to aid laboratory and environmental compliance personnel in making a decision whether or not

to handle a particular spent laboratory sample as either a RCRA regulated or non-regulated waste containing potentially hazardous/ toxic substances or simply a solid waste. Application of these guidelines provides an environmentally conservative approach to the disposal of spent laboratory samples and minimizes the potential for non-compliance with RCRA regulations.

4.8.3. At the completion of each project, the laboratory generates a report from its laboratory information management system which describes each analysis performed on the individual samples together with a parameter by parameter listing of positive results. The computer program has been designed to deliver a list of samples that are potentially hazardous as defined by statute. Samples that are indicated by the computer program as potentially hazardous are referred to the Divisional SHEM Officer who then makes the decision as to the proper disposition of the samples.

4.8.4. Acidified water samples are subject to elementary neutralization and would be classified as "hazardous" solely upon the basis of pH. See Section 3.5 for proper disposal. This method of disposal is applicable to all water samples which are classified as a hazardous waste exclusively on the basis of corrosivity.

4.8.5. All non-acidified water samples are disposed of via the laboratory sinks.

4.8.6. All samples containing total PCB's greater than 50 mg/kg are subject to TSCA provisions contained in 40 CFR 761 and are disposed of as PCB containing materials.

4.8.6.0.1. Disposal of Foreign Soil- See Section 3.7

4.9. Handling, Storage, Disposal and Reporting Procedures for PCB Containing Materials

4.9.1. The handling, storage, disposal and reporting of PCB items, containers, and articles containing PCB's in concentrations greater than 50 ppm are regulated under the Toxic Substances Control Act (TSCA). Applicable regulations are contained in 40 CFR Part 76l.1.

4.9.2. REGULATORY REQUIREMENTS

4.9.2.1. Marking Requirements: Any PCB article or container (40 CFR 76l.3) of PCB materials in a concentration greater than 50 ppm must be properly marked according to 40 CFR 76l.40.

4.9.2.2. Storage Requirements: Any PCB containing material (i.e., item, article, etc.) designated for disposal shall be disposed of within l year from the date it was first placed in storage (40 CFR 76l.65 (a).

4.9.3. The storage facility shall comply with the requirements specified in 40 CFR 76l.65 (b) (l) (e.g., roof, walls, floor, curbing, location, marking, inspection, etc.).

4.9.3.1. Temporary Storage: PCB wastes stored in laboratories are considered to be in temporary storage as described in 40 CFR 761.65 (c) without having to comply with the storage requirements provided that: (1) the wastes container displays a proper PCB label, (2) contains the date accumulation started, (3) are stored in a DOT specification container as described in 40 CFR 761.65 (c)(6), and (4) are not stored in the laboratory for more than 30 days.

4.9.3.2. Reporting and Records: If at any time the facility stores 45 kg (99.4 pounds) of PCB material with a concentration greater than 50 ppm, the following information will be compiled in an annual report: volume of PCB's stored, storage dates, disposal dates, and PCB source (40 CFR 76l.l80(a)). An Annual Report will prepared by the Divisional SHEM Officer.

4.9.3.3. Disposal Requirements: Destruction of PCB containing materials must be done in an incinerator which complies with the requirements contained in 40 CFR 761.70.

5. LABORATORY EQUIPMENT MAINTENANCE AND SERVICE

5.1. Proper maintenance of laboratory instrumentation is a key ingredient to both the longevity of the instrumentation, as well as, providing the analyst with equipment capable of producing reliable analyses. Proper equipment maintenance requires an alert analytical staff which recognizes the need for equipment maintenance coupled with available support services provided either by in-house personnel or vendor specialists.

5.2. Responsibility for maintenance and repair of all Branch laboratory equipment is shared by the analysts and on occasion, vendor specialists.

5.3. The primary elements of the equipment maintenance program include:

5.3.1. All major equipment receives a daily check for such things as: cooling fan operation, pump operation, indicator readings, mechanical checks, clean air filters, etc.

5.3.2. Service schedules are established for performing routine preventative maintenance on all major equipment items.

5.3.3. Records are maintained for all equipment repairs.

5.3.4. Instrument utilization records; including operating, and downtime, are maintained for all GC, AA, GC/MS and ICAP instruments.

5.3.5. A conservative inventory of critical spare parts is maintained for high-use instrumentation.

5.3.6. Vendor operation and maintenance manuals are maintained for all laboratory instrumentation.

6. LABORATORY SAFETY

6.1. INTRODUCTION

6.1.1. All Branch employees must accept the responsibility for acting in accordance with safety rules and practices and for reporting any observed safety hazard. This section highlights some general guidelines and rules that specifically apply to the Analytical Support Branch. Obviously no set of rules will cover all possible situations. Therefore, in addition to adhering to these rules, each person is expected to exercise good judgement in all situations and to maintain a high level of safety consciousness.

6.1.2. The rules and guidelines listed in this section only supplement or highlight the following official publications:

6.1.2.1. Safety and Health Manual, draft (proposed effective Feb 1998).

6.1.2.2. EPA Occupational Health and Safety Manual, October l984.

6.1.2.3. Laboratory Health Monitoring Requirements, March 6, 1987.

6.2. General

6.2.1. Lab coats and safety glasses should be worn at all times in laboratories. The only exception to this is when personnel are working at computer terminals or microscopes. When working with corrosives and/or toxic substances, lab coats should be left in the laboratory.

6.2.2. Open sandals and shorts will not be worn in laboratories.

6.2.3. When working in any of the laboratories, it is recommended that all jewelry be removed and that personnel wash their hands frequently. Always wash hands thoroughly when leaving the laboratory.

6.2.4. When working with flammable materials, nylon or other totally synthetic clothing should be avoided to minimize the possibility of static sparks.

6.2.5. All containers should be labeled as to contents, with particular care to note corrosive or hazardous materials.

6.2.6. There will be no eating, drinking, or smoking in any laboratory.

6.2.7. Glassware that is chipped but still usable, must be fire polished before use; otherwise it must be discarded.

6.2.8. Never use any lab glassware as a container for food or drink.

6.2.9. An inventory of all chemicals maintained in the laboratory will be prepared and updated on an annual basis.

6.2.10. Return all chemicals to their proper storage areas after use.

6.2.11. Never pipet by mouth.

6.2.12. Designated personnel are to conduct a safety inspection of their laboratory at least quarterly.

6.2.13. No perchloric acid or perchlorate salts will be stored in the Analytical Support Branch. If at any time these chemicals are required in a method, special precautions will be necessary and should be coordinated with the Chief of the Inorganic Chemistry or Organic Chemistry Section.

6.2.14. All work areas should be cleaned at the end of each work day. Spills should be cleaned up immediately.

6.2.15. Samples should be in laboratories only during preparation and analysis; otherwise, keep them in the custody room, or proper volatile organic storage area.

6.2.16. All stock standards of a toxic nature should be prepared in a hood and stored in designated areas. Only experienced personnel should handle these standards.

6.2.17. Work of a hazardous nature will not be performed in a laboratory after normal business hours when only one person is present.

6.2.18. New personnel must be familiarized with safety practices, location of safety equipment, and made aware of possible hazards in the areas in which they will be working.

6.2.19. When conducting routine maintenance of electrical equipment, observe all shock hazard warnings displayed on instrumentation.

6.2.20. Use safety guards where appropriate when using electrical equipment or ventilation/fumehood systems.

6.2.21. Observe all cryoprotective warnings regarding cylinders and sample storage areas.

6.2.22. When using pressurized systems, take care to tighten restraints before pressurizing system and depressurize system before loosening restraints.

6.3. Sample Receiving and Logging

6.3.1. When possible, determine the source of the samples and any special hazards that might be associated with them. **(Refer also to Section 3, Laboratory Chain-of-Custody and Sample Handling.)**

6.3.2. Some samples, especially domestic waste when sealed in containers will build up pressure. Care should be taken in handling these type samples. Also, gloves should be used to handle these samples during analysis, due to the possibility of the transmission of a variety of human enteric pathogens that cause diseases.

6.3.3. Broken samples should be handled with protective gloves and disposed of immediately according to the waste disposal procedures.

6.3.4. A small percentage of samples received by ASB would be characterized as concentrated waste. These samples will require special handling. **(Refer to Section 3.5.6.2 for Handling Procedures.)**

6.4. Compressed Gases

6.4.1. Compressed gases should be handled in accordance with Chapter 3 of the Safety and Health Manual, Science and Ecosystems Support Division.

6.4.2. It is the responsibility of each Team to maintain current inventory and status of compressed gases used within their respective areas. Each Section Chief and/or Team must designate individuals to perform these inventories.

6.5. Radioactivity

6.5.1. Electron Capture Detectors require wipe tests for radioactivity every six months.

6.5.2. The Divisional SHEM Officer will be the person responsible for the wipe tests and to maintain documentation of the tests.

6.6. Laboratory Waste Disposal Practices

6.6.1. ASB is subject to the Resource Conservation and Recovery Act (RCRA) regulations as contained in the Georgia Rules for Hazardous Waste Managment for handling, storage, and disposal of laboratory related wastes. While knowledge of the hazardous waste handling and disposal regulations is the primary responsiblity of the Divisional SHEM Officer, each ASB staff member should become familiar with the basic policies and procedures for waste disposal as it pertains to his/her area. **ALL WASTE DISPOSAL MUST BE COORDINATED WITH THE SHEM OFFICER. (Refer also to Section 4.6)**

7. METHODOLOGY

7.1. A detailed listing and discussion of specific chemical methods are not included in this manual. Instead, lists containing the methods (and analytical technique) used in this laboratory for organic analysis of all sample types are listed in Table 7-1. Table 7-1 contains the method tracking number and method summary. The method tracking number is listed in extraction logbooks to identify the organic methods of extraction and analysis. (See Table 7-2.) See Section 11 for references to inorganic methods.

7.2. Details on the applications, limitations, precision, and accuracy are found within the listed methods.

7.3. Reporting Units

7.3.1. Table 7-3 lists the reporting concentration units for all parameters in waters, soil/sediments (solids), fish (tissue), air, and waste. These units are always to be used unless sample matrix or methodology criteria require a change. Changes in units must be coordinated between the Organic and Inorganic Chemistry Sections.

TABLE 7-1
LIST OF TEST PROCEDURES

Parameter	Method	Reference	Tracker #
Surface Water, Monitoring Wells, Wastewater			
1. Extractable Organics	Capillary GC/MS	8270/625/CLP	47A
2. Volatile Organics	Capillary GC/MS	8260/624/CLP	46A
3. Organochlorine Pesticides/PCBs	Capillary GC/ECD	8081/608/CLP	55A,55
4. Acid Herbicides	Capillary GC/ECD	8151/515.1	38A
5. Organophosphorus Pesticides	Capillary GC/NPD	8141	57
6. Formaldehyde	Capillary GC/MS	ASB Method	48A,48
	HPLC	FORM-10/83/8315	
Drinking Water			
1. Extractable Organics	Capillary GC/MS	525/8270	47A
2. Volatile Organics	Capillary GC/MS	524.2/8260	46A
3. Organochlorine Pesticides/PCBs	Capillary GC/ECD	508/8081	55A,55
4. Acid Herbicides	Capillary GC/ECD	515.1/8151	38A
5. EDB and DBCP	Capillary GC/ECD	504	52A
6. Screening for PCBs	Capillary GC/ECD	508A	62

TABLE 7-1 (cont.)

LIST OF TEST PROCEDURES

Parameter	Method	Reference	METHOD TRACKER #
SEDIMENT/SOIL			
1. Extractable Organics	Capillary GC/MS	3550/8270/CLP	43,43A,54
2. Volatile Organics	Capillary GC/MS	8260/CLP	43D,54B
3. Organochlorine Pesticides/PCBs	Capillary GC/ECD	3550/8080/CLP	43,43A,43B, 43C,54
4. Acid Herbicides	Capillary GC/ECD	8151	51A
5. Organophosphorus Pesticides	Capillary GC/NPD	3550/8141	57
6. Formaldehyde	Capillary GC/MS HPLC	ASB Method FORM-10/83/8315	48A,48
7. PCBS for TSCA	Capillary GC/ECD	3540/8080	31A
WASTE			
1. Extractable Organics	Capillary GC/MS	3580/8270 3550/8270	54A 54C
2. Volatile Organics	Capillary GC/MS	8260	54B
3. Organochlorine Pesticides/PCBs	Capillary GC/ECD	3580/8080 3550/8080	54A 54C
4. Acid Herbicides	Capillary GC/ECD	8151	51A
5. PCBs in Waste Oil	Capillary GC/ECD	600/4-81-045 8080	35
TISSUE			
1. Extractable Organics	Capillary GC/MS 8270	ASB Sonicator	44
2. Volatile Organics	Capillary GC/MS	ASB Method/8260	44
3. Organochlorine Pesticides/PCBs	Capillary GC/ECD 8080	ASB Sonicator	44

TABLE 7-1 (cont.)

LIST OF TEST PROCEDURES

Parameter	Method	Reference	METHOD TRACKER #
AIR			
1. Extractable Organics	Capillary GC/MS	PUF by TO13 8270	50C
2. Volatile Organics	Capillary GC/MS	Canister by TO14/8260	56
3. Organochlorine Pesticides/PCBs	Capillary GC/ECD	PUF by TO4 8080	50
4. Formaldehyde	HPLC	Trap by TO11	59

Method#	Descriptor	Sample Type
46A	VLW	Volatile low water
43D	VLS	Volatile low soil/sed
54B	VMS	Volatile medium soil/sed
54B	VMW	Volatile medium waste
56	VAC	Volatile air canister
60	VTC	VOLATILE BY TCLP EXTRACTION
47	SLW	Semivolatile low water - separatory funnel
47A	SLW	Semivolatile low water - continuous liquid ext.
43	SLG	Semivolatile low soil/sed w/GPC
43A	SLS	Semivolatile low soil/sed wo/GPC
54	SMS	Semivolatile medium soil/sed
54A	SHW	Semivolatile high waste wo/son.
54C	SMW	Semivolatile medium waste w/son.
44	SLT	Semivolatile low tissue
50B	SAP	Semivolatile air PUF
58	SCW	Semivolatile Cartridge ext. water
60	STC	Semivolatiles by TCLP extraction
31C	SSS	SEMIVOLATILE LOW SOIL WITH SOXHLET
55	PLW	Pesticide low water - separatory funnel
55A	PLW	Pesticide low water - continuous liquid ext.
43	PLG	Pesticide low soil/sed w/GPC
43A	PLS	Pesticide low soil/sed wo/GPC
43B	PSA	Pesticide low soil/sed w/acid cleanup
43C	PSH	Pesticide low soil/sed w/hex/acetone
44	PLT	Pesticide low tissue
44A	PTH	Pesticide low tissue w/hexane
44B	PTA	Pesticide low tissue w/acid cleanup
50B	PAP	Pesticide air PUF
54	PMS	Pesticide medium soil/sed
54A	PHW	Pesticide high waste wo/son.
54C	PMW	Pesticide medium waste w/son.
52B	PCW	Pesticide Cartridge ext. water
57	PNP	Pesticide low water nitrogen/phosphorous
60	PTC	Pesticides by TCLP extraction
35	PWO	PCBs waste oil
31A	PCS	PCB LOW SOIL WITH SOXHLET
31C	PSS	PEST/PCB LOW SOIL WITH SOXHLET
38	HLW	Herbicides low water
51	HLS	Herbicides low soil/sed
60	HTC	Herbicides by TCLP extraction
48	FLW	Formaldehyde low water
48	FLS	Formaldehyde low soil/sed
59	FAC	Formaldehyde air cartridge
61	CLW	Carbamates low water w/HPLC
61	CLS	Carbamates low soil/sed w/HPLC

Table 7-2

METHOD SOURCES FOR TABLES 7-1 & 7-2

1. 1000-8000 Methods: USEPA, Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, 1986 plus the 1st and 2nd Updates.

2. 500 Methods: USEPA, Methods for the Determination of Organic Compounds in Drinking Water, EPA/600/4-88/039, Dec., 1988.

3. 600 Methods: USEPA, Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act-40CFR Part 136, Federal Register of Oct. 26, 1984.

4. TO Methods: USEPA, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA-600/4-84-041, Apr. 1984 plus the Supplements of 1986 and 1988.

5. CLP Methods: USEPA Contract Laboratory Program Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration, 1990.

6. USFDA Methods: Pesticide Analytical Manual, Volumes I and II.

7. Region 4 Methods: Adaptations of Published Methods when Official Methods are not Available.

TEST #	TEST DESCRIPTION	UNITS WATER	UNITS SOLIDS	UNITS TISSUE	UNITS WASTE	UNITS AIR
7736	% ALCOHOL	%	%	%	%	UG/M3
4045	% LIPIDS					
9999	% MOISTURE		%	%	%	
1046	% SOLIDS	%	%			
1028	% WATER	%	%	%	%	
5215	2,4,5-T	UG/L	UG/KG	MG/KG	MG/KG	
5205	2,4-D	UG/L	UG/KG	MG/KG	MG/KG	
5206	2,4-DB	UG/L	UG/KG	MG/KG	MG/KG	
6105	ACENAPHTHENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6100	ACENAPHTHYLENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6716	ACETALDEHYDE	UG/L	UG/KG	MG/KG	MG/KG	
4055	ACETATE	MG/L	MG/KG	MG/KG	MG/KG	
7051	ACETONE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
3005	ACIDITY	MG/L	MG/KG	MG/KG	MG/KG	
7015	ACROLEIN	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7020	ACRYLONITRILE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
5867	ALACHLOR (LASSO)	UG/L	UG/KG	MG/KG	MG/KG	
5793	ALDICARB	UG/L	UG/KG	MG/KG	MG/KG	
5794	ALDICARB SULFONE	UG/L	UG/KG	MG/KG	MG/KG	
5795	ALDICARB SULFOXIDE	UG/L	UG/KG	MG/KG	MG/KG	
5005	ALDRIN	UG/L	UG/KG	MG/KG	MG/KG	
3008	ALKALINITY, BICARBONATE (AS CACO3)	MG/L				
3009	ALKALINITY, CARBONATE (AS CACO3)	MG/L				
3010	ALKALINITY, TOTAL (AS CACO3)	MG/L	MG/KG	MG/KG	MG/KG	
2125	ALUMINUM	UG/L	MG/KG	MG/KG	MG/KG	
5884	AMBUSH (PERMETHRIN)	UG/L	UG/KG	MG/KG	MG/KG	
1008	AMES TEST					
5830	AMETRYN	UG/L	UG/KG	MG/KG	MG/KG	
5791	AMINOBENZIMIDAZOLE, 2- (2-AB)	UG/L	UG/KG	MG/KG	MG/KG	
3015	AMMONIA	MG/L	MG/KG	MG/KG	MG/KG	
3016	AMMONIA, DISSOLVED	MG/L				
3018	AMMONIA, UNIONIZED (AS NH3)	MG/L				
6160	ANTHRACENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
2065	ANTIMONY	UG/L	MG/KG	MG/KG	MG/KG	
5878	ANTOR	UG/L	UG/KG	MG/KG	MG/KG	
5115	AROCLOR 1016 (PCB-1016)	UG/L	UG/KG	MG/KG	MG/KG	
5095	AROCLOR 1221 (PCB-1221)	UG/L	UG/KG	MG/KG	MG/KG	
5100	AROCLOR 1232 (PCB-1232)	UG/L	UG/KG	MG/KG	MG/KG	
5085	AROCLOR 1242 (PCB-1242)	UG/L	UG/KG	MG/KG	MG/KG	
5105	AROCLOR 1248 (PCB-1248)	UG/L	UG/KG	MG/KG	MG/KG	

Table 7-3

TEST #	TEST DESCRIPTION	UNITS WATER	UNITS SOLIDS	UNITS TISSUE	UNITS WASTE	UNITS AIR
5090	AROCLOR 1254 (PCB-1254)	UG/L	UG/KG	MG/KG	MG/KG	
5110	AROCLOR 1260 (PCB-1260)	UG/L	UG/KG	MG/KG	MG/KG	
5111	AROCLOR 1262 (PCB-1262)	UG/L	UG/KG	MG/KG	MG/KG	
5112	AROCLOR 1268 (PCB-1268)	UG/L	UG/KG	MG/KG	MG/KG	
5895	AROCLORS, TOTAL (PCBS)	UG/L	UG/KG	MG/KG	MG/KG	
2010	ARSENIC	UG/L	MG/KG	MG/KG	MG/KG	
1005	ASBESTOS (FIBROUS)	F/L	UG/KG	MG/KG	MG/KG	
1007	ASBESTOS, BULKED				%	
1006	ASH		%		%	
5861	ATRAZINE	UG/L	UG/KG	MG/KG	MG/KG	
5781	AZODRIN (MONOCROTOPHOS)	UG/L	UG/KG	MG/KG	MG/KG	
1010	BAC-T					
5894	BALAN (BENEFIN)	UG/L	UG/KG	MG/KG	MG/KG	
2020	BARIUM	UG/L	MG/KG	MG/KG	MG/KG	
5789	BENOMYL	UG/L	UG/KG	MG/KG	MG/KG	
5868	BENOMYL (BENLATE)	UG/L	UG/KG	MG/KG	MG/KG	
7105	BENZENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6020	BENZIDINE	UG/L	UG/KG	MG/KG	MG/KG	
6190	BENZO(A)ANTHRACENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6215	BENZO(B AND/OR K)FLUORANTHENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6210	BENZO(B AND/OR K)FLUORANTHENE *	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6211	BENZO(B)FLUORANTHENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6235	BENZO(GHI)PERYLENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6216	BENZO(K)FLUORANTHENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6220	BENZO-A-PYRENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6256	BENZOIC ACID	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6795	BENZONITRILE	UG/L	UG/KG	MG/KG	MG/KG	
6770	BENZOPHENONE	UG/L	UG/KG	MG/KG	MG/KG	
6241	BENZYL ALCOHOL	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6180	BENZYL BUTYL PHTHALATE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6765	BENZYLIC ACID	UG/L	UG/KG	MG/KG	MG/KG	
2025	BERYLLIUM	UG/L	MG/KG	MG/KG	MG/KG	
5020	BHC, ALPHA-	UG/L	UG/KG	MG/KG	MG/KG	
5025	BHC, BETA-	UG/L	UG/KG	MG/KG	MG/KG	
5035	BHC, DELTA-	UG/L	UG/KG	MG/KG	MG/KG	
5030	BHC, GAMMA- (LINDANE)	UG/L	UG/KG	MG/KG	MG/KG	
3012	BICARBONATE (AS HCO3 ION)	MG/L	MG/KG			
6080	BIS(2-CHLOROETHOXY)METHANE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6040	BIS(2-CHLOROETHYL) ETHER	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6050	BIS(2-CHLOROISOPROPYL) ETHER	UG/L	UG/KG	MG/KG	MG/KG	UG/M3

TEST #	TEST DESCRIPTION	UNITS WATER	UNITS SOLIDS	UNITS TISSUE	UNITS WASTE	UNITS AIR
6805	BIS(2-ETHYLHEXYL) ADIPATE	UG/L	UG/KG	MG/KG	MG/KG	
6185	BIS(2-ETHYLHEXYL) PHTHALATE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
2705	BISMUTH	UG/L	MG/KG	MG/KG	MG/KG	
4004	BOD (LONG TERM)	MG/L				
4007	BOD, 20 DAY	MG/L				
4005	BOD, 5 DAY	MG/L				
4009	BOD, 5 DAY (CARBONACEOUS)	MG/L				
4006	BOD, 5 DAY (DISSOLVED)	MG/L				
4008	BOD, 60 DAY	MG/L				
5885	BOLSTAR (SULPROFOS)	UG/L	UG/KG	MG/KG	MG/KG	
2015	BORON	UG/L	MG/KG	MG/KG	MG/KG	
5862	BROMACIL	UG/L	UG/KG	MG/KG	MG/KG	
2727	BROMATE	UG/L	UG/KG	MG/KG	MG/KG	
3017	BROMIDE	MG/L				
7131	BROMOBENZENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6810	BROMOCHLOROACETONITRILE	UG/L	UG/KG	MG/KG	MG/KG	
7059	BROMOCHLOROMETHANE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7085	BROMODICHLOROMETHANE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7130	BROMOFORM	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7030	BROMOMETHANE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6150	BROMOPHENYL PHENYL ETHER, 4-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7737	BTEX	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
5902	BUTACHLOR	UG/L	UG/KG	MG/KG	MG/KG	
5801	BUTYLATE	UG/L	UG/KG	MG/KG	MG/KG	
7907	BUTYLBENZENE, N-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7905	BUTYLBENZENE, SEC-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7903	BUTYLBENZENE, TERT-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6173	BUTYLISOCYANATE, N- (BIC)	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6713	BUTYLISOCYANATE, N- (BIC)	UG/L	UG/KG	MG/KG	MG/KG	
2030	CADMIUM	UG/L	MG/KG	MG/KG	MG/KG	
2135	CALCIUM*	MG/L	MG/KG	MG/KG	MG/KG	
2136	CALCIUM (LOW LEVEL)	UG/L	MG/KG	MG/KG	MG/KG	
5782	CAPTAN	UG/L	UG/KG	MG/KG	MG/KG	
6295	CARBAZOLE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
5790	CARBENDAZIM (MBC)	UG/L	UG/KG	MG/KG	MG/KG	
5904	CARBOFURAN	UG/L	UG/KG	MG/KG	MG/KG	
7052	CARBON DISULFIDE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7080	CARBON TETRACHLORIDE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
4038	CARBON, PARTICULATE ORGANIC	MG/L	MG/L			
4037	CARBON, PURGEABLE ORGANIC	MG/L				

Table 7-3

TEST #	TEST DESCRIPTION	UNITS WATER	UNITS SOLIDS	UNITS TISSUE	UNITS WASTE	UNITS AIR	
3022	CARBON, TOTAL		MG/KG		%		
4035	CARBON, TOTAL ORGANIC	MG/L	MG/KG	MG/KG	MG/KG		
4036	CARBON, TOTAL ORGANIC (DISSOLVED)	MG/L					
3013	CARBONATE (AS CO3 ION)	MG/L	MG/KG				
5871	CARBOPHENOTHION (TRITHION)	UG/L	UG/KG	MG/KG	MG/KG		
1076	CATION EXCHANGE CAPACITY (CEC)	UG/L	MQ/KG	MG/KG	MG/KG		
2715	CERIUM	UG/L	MG/KG	MG/KG	MG/KG		
4010	CHEMICAL OXYGEN DEMAND	MG/L	MG/KG		MG/KG		
4011	CHEMICAL OXYGEN DEMAND, DISSOLVED	MG/L					
5787	CHLORAMBEN (AMIBEN)	UG/L	UG/KG	MG/KG	MG/KG		
2728	CHLORATE	UG/L	UG/KG	MG/KG	MG/KG		
5080	CHLORDANE (TECH. MIXTURE)/1	UG/L	UG/KG	MG/KG	MG/KG		
5165	CHLORDANE, ALPHA-/2	UG/L	UG/KG	MG/KG	MG/KG		
5155	CHLORDANE, GAMMA-/2	UG/L	UG/KG	MG/KG	MG/KG		
5135	CHLORDENE/2	UG/L	UG/KG	MG/KG	MG/KG		
5140	CHLORDENE, ALPHA-/2	UG/L	UG/KG	MG/KG	MG/KG		
5142	CHLORDENE, BETA- /2	UG/L	UG/KG	MG/KG	MG/KG		
5145	CHLORDENE, GAMMA-/2	UG/L	UG/KG	MG/KG	MG/KG		
5824	CHLORDIMEFORM	UG/L	UG/KG	MG/KG	MG/KG		
3020	CHLORIDE	MG/L	MG/KG		MG/KG		
3021	CHLORINE				%		
8005	CHLORINE, RESIDUAL	MG/L					
2729	CHLORITE	UG/L	UG/KG	MG/KG	MG/KG		
6270	CHLORO-3-METHYLPHENOL, 4-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3	
6076	CHLOROANILINE, 4-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3	
6803	CHLOROBENZALDEHYDE, O-	UG/L	UG/KG	MG/KG	MG/KG		
7150	CHLOROBENZENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3	
5880	CHLOROBENZILATE	UG/L	UG/KG	MG/KG	MG/KG		
5874	CHLOROBENZILATE *	UG/L	UG/KG	MG/KG	MG/KG		
7040	CHLOROETHANE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3	
7125	CHLOROETHYLVINYL ETHER, 2-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3	
7065	CHLOROFORM	UG/L	UG/KG	MG/KG	MG/KG	UG/M3	
7025	CHLOROMETHANE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3	
6095	CHLORONAPHTHALENE, 2-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3	
6240	CHLOROPHENOL, 2-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3	
6760	CHLOROPHENOL, 4-	UG/L	UG/KG	MG/KG	MG/KG		
6125	CHLOROPHENYL PHENYL ETHER, 4-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3	
1082	CHLOROPHYLL A (FLUORIMETER)	UG/L	UG/KG	MG/KG	MG/KG		
1083	CHLOROPHYLL A (HPLC)	UG/L	UG/KG	MG/KG	MG/KG		
1081	CHLOROPHYLL A (UV/VIS)	UG/L	UG/KG	MG/KG	MG/KG		

TEST #	TEST DESCRIPTION	UNITS WATER	UNITS SOLIDS	UNITS TISSUE	UNITS WASTE	UNITS AIR
5869	CHLOROTHALONIL	UG/L	UG/KG	MG/KG	MG/KG	
5792	CHLOROTHALONIL *	UG/L	UG/KG	MG/KG	MG/KG	
7185	CHLOROTOLUENE, M-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7180	CHLOROTOLUENE, O-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7190	CHLOROTOLUENE, P-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
2040	CHROMIUM	UG/L	MG/KG	MG/KG	MG/KG	
2155	CHROMIUM, HEXAVALENT	UG/L	MG/KG	MG/KG	MG/KG	
6195	CHRYSENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
2035	COBALT	UG/L	MG/KG	MG/KG	MG/KG	
1090	COLIFORM, FECAL MF/100ML					
1085	COLIFORM, FECAL MPN/100ML					
1100	COLIFORM, TOTAL MF/100ML					
1095	COLIFORM, TOTAL MPN/100ML					
1018	COLOR (ADMI @ ORIG. SMPL. PH)					
1016	COLOR (ADMI @ PH 7.6)	ADMI				
1015	COLOR (APPARENT-PTCO)	PTCO				
1014	COLOR (TRUE-PTCO)	PTCO				
1020	CONDUCTIVITY	UMHOS				
2045	COPPER	UG/L	MG/KG	MG/KG	MG/KG	
1025	CORROSIVITY (PH)				PH	
1026	CORROSIVITY (STEEL)		MM/YR		MM/YR	
5863	CYANAZINE	UG/L	UG/KG	MG/KG	MG/KG	
3025	CYANIDE*	MG/L	MG/KG	MG/KG	MG/KG	
3026	CYANIDE (LOW LEVEL)	UG/L	MG/KG	MG/KG	MG/KG	
3038	CYANIDE MICRODIFFUSION/METHOD 4282	UG/L	UG/KG	MG/KG	MG/KG	
3039	CYANIDE WEAK DISSOCIABLE/METHOD 4500	UG/L	UG/KG	MG/KG	MG/KG	
3027	CYANIDE, AMENABLE TO CHLORINATION	UG/L	MG/KG	MG/KG	MG/KG	
3028	CYANIDE, FREE	UG/L	MG/KG	MG/KG	MG/KG	
3037	CYANIDE, REACTIVE(AS HCN)		MG/KG		MG/KG	
5803	CYCLOATE	UG/L	UG/KG	MG/KG	MG/KG	
5886	CYGON (DIMETHOATE)	UG/L	UG/KG	MG/KG	MG/KG	
5783	DALAPON	UG/L	UG/KG	MG/KG	MG/KG	
5785	DASANIT	UG/L	UG/KG	MG/KG	MG/KG	
5858	DDD, 2,4'- (O,P'-DDD)	UG/L	UG/KG	MG/KG	MG/KG	
5060	DDD, 4,4'- (P,P'-DDD)	UG/L	UG/KG	MG/KG	MG/KG	
5857	DDE, 2,4'- (O,P'-DDE)	UG/L	UG/KG	MG/KG	MG/KG	
5055	DDE, 4,4'- (P,P'-DDE)	UG/L	UG/KG	MG/KG	MG/KG	
5856	DDT, 2,4'- (O,P'-DDT)	UG/L	UG/KG	MG/KG	MG/KG	
5050	DDT, 4,4'- (P,P'-DDT)	UG/L	UG/KG	MG/KG	MG/KG	
5859	DDT, TOTAL RESIDUES (TDDTR)	UG/L	UG/KG	MG/KG	MG/KG	

Table 7-3

TEST #	TEST DESCRIPTION	UNITS WATER	UNITS SOLIDS	UNITS TISSUE	UNITS WASTE	UNITS AIR
5883	DDVP (2,2-DICHLOROVINYLDIETHYLPHOSPHATE)	UG/L	UG/KG	MG/KG	MG/KG	
5839	DECACHLOROBIPHENYL (DCB)	UG/L	UG/KG	MG/KG	MG/KG	
5899	DEET	UG/L	UG/KG	MG/KG	MG/KG	
5825	DEF	UG/L	UG/KG	MG/KG	MG/KG	
5822	DELNAV (DIOXATHION)	UG/L	UG/KG	MG/KG	MG/KG	
5819	DEMETON-S	UG/L	UG/KG	MG/KG	MG/KG	
1002	DENSITY (20 DEG. C)	GM/ML			GM/ML	
6165	DI-N-BUTYLPHTHALATE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6779	DI-N-BUTYLSEBACATE	UG/L	UG/KG	MG/KG	MG/KG	
6205	DI-N-OCTYLPHTHALATE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
5833	DIALLATE	UG/L	UG/KG	MG/KG	MG/KG	
5860	DIAZINON	UG/L	UG/KG	MG/KG	MG/KG	
6230	DIBENZO(A,H)ANTHRACENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6111	DIBENZOFURAN	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
5817	DIBROM (NALED)	UG/L	UG/KG	MG/KG	MG/KG	
7908	DIBROMO-3-CHLOROPROPANE, 1,2-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
5873	DIBROMO-3-CHLOROPROPANE, 1,2- (DBCP)	UG/L	UG/KG	MG/KG	MG/KG	
6811	DIBROMOACETONITRILE	UG/L	UG/KG	MG/KG	MG/KG	
7110	DIBROMOCHLOROMETHANE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7720	DIBROMOETHANE, 1,2- (EDB)	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7091	DIBROMOMETHANE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6772	DIBUTYL TIN	UG/L	UG/KG	MG/KG	MG/KG	
5836	DICAMBA	UG/L	UG/KG	MG/KG	MG/KG	
6806	DICHLOROACETIC ACID	UG/L	UG/KG	MG/KG	MG/KG	
6812	DICHLOROACETONITRILE	UG/L	UG/KG	MG/KG	MG/KG	
6035	DICHLOROBENZENE, 1,2- (EXTRACTABLE)	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7205	DICHLOROBENZENE, 1,2- (VOLATILE) *	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6025	DICHLOROBENZENE, 1,3- (EXTRACTABLE)	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7195	DICHLOROBENZENE, 1,3- (VOLATILE) *	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6030	DICHLOROBENZENE, 1,4- (EXTRACTABLE)	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7200	DICHLOROBENZENE, 1,4- (VOLATILE) *	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6200	DICHLOROBENZIDINE, 3,3'-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
5221	DICHLOROBENZILATE	UG/L	UG/KG	MG/KG	MG/KG	
6714	DICHLOROBENZOIC ACID	UG/L	UG/KG	MG/KG	MG/KG	
6816	DICHLOROBENZOPHENONE, 4,4'- (EXTRACTABLE) *	UG/L	UG/KG	MG/KG	MG/KG	
5881	DICHLOROBENZOPHENONE, 4,4'- (PESTICIDE)	UG/L	UG/KG	MG/KG	MG/KG	
7005	DICHLORODIFLUOROMETHANE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7055	DICHLOROETHANE, 1,1-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3

Table 7-3

TEST #	TEST DESCRIPTION	UNITS WATER	UNITS SOLIDS	UNITS TISSUE	UNITS WASTE	UNITS AIR
7070	DICHLOROETHANE, 1,2-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7050	DICHLOROETHENE, 1,1- (1,1-DICHLOROETHYLENE)	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7061	DICHLOROETHENE, 1,2- (TOTAL)	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7056	DICHLOROETHENE, CIS-1,2-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7060	DICHLOROETHENE, TRANS-1,2-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6260	DICHLOROPHENOL, 2,4-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6261	DICHLOROPHENOL, 2,6-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
5806	DICHLOROPROP	UG/L	UG/KG	MG/KG	MG/KG	
7090	DICHLOROPROPANE, 1,2-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7141	DICHLOROPROPANE, 1,3-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7057	DICHLOROPROPANE, 2,2-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6813	DICHLOROPROPANONE, 1,1-	UG/L	UG/KG	MG/KG	MG/KG	
7076	DICHLOROPROPENE, 1,1-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7120	DICHLOROPROPENE, CIS-1,3-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7095	DICHLOROPROPENE, TRANS-1,3-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
5876	DICOFOL (KELTHANE)	UG/L	UG/KG	MG/KG	MG/KG	
5045	DIELDRIN	UG/L	UG/KG	MG/KG	MG/KG	
6135	DIETHYL PHTHALATE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6003	DIETHYLENE GLYCOL MONOETHYL ETHER	UG/L	UG/KG	MG/KG	MG/KG	
5818	DIMETHOATE	UG/L	UG/KG	MG/KG	MG/KG	
6110	DIMETHYL PHTHALATE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7730	DIMETHYLAMINE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6255	DIMETHYLPHENOL, 2,4-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6800	DINITROBENZENE, 1,3-	UG/L	UG/KG	MG/KG	MG/KG	
6275	DINITROPHENOL, 2,4-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6115	DINITROTOLUENE, 2,4-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6120	DINITROTOLUENE, 2,6-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
5887	DINOSEB (DNBP)	UG/L	UG/KG	MG/KG	MG/KG	
6775	DIPHENYL TIN	UG/L	UG/KG	MG/KG	MG/KG	
6015	DIPHENYLHYDRAZINE, 1,2-/AZOBENZENE	UG/L	UG/KG	MG/KG	MG/KG	
5810	DIQUAT	UG/L	UG/KG	MG/KG	MG/KG	
1001	DISTRIBUTION COEFFICIENT					
5842	DISULFOTHION (DISULFTON)	UG/L	UG/KG	MG/KG	MG/KG	
5831	DISULFOTON	UG/L	UG/KG	MG/KG	MG/KG	
5896	DISYSTON	UG/L	UG/KG	MG/KG	MG/KG	
5828	DIURON	UG/L	UG/KG	MG/KG	MG/KG	
5889	DURSBAN (CHLORPYRIFOS) (LORSBAN)	UG/L	UG/KG	MG/KG	MG/KG	
5040	ENDOSULFAN I (ALPHA)	UG/L	UG/KG	MG/KG	MG/KG	
5070	ENDOSULFAN II (BETA)	UG/L	UG/KG	MG/KG	MG/KG	

TEST #	TEST DESCRIPTION	UNITS WATER	UNITS SOLIDS	UNITS TISSUE	UNITS WASTE	UNITS AIR
5075	ENDOSULFAN SULFATE	UG/L	UG/KG	MG/KG	MG/KG	
5065	ENDRIN	UG/L	UG/KG	MG/KG	MG/KG	
5125	ENDRIN ALDEHYDE	UG/L	UG/KG	MG/KG	MG/KG	
5220	ENDRIN KETONE	UG/L	UG/KG	MG/KG	MG/KG	
7725	EPICHLOROHYDRIN	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
5888	EPN	UG/L	UG/KG	MG/KG	MG/KG	
5811	EPTC (EPTAM)	UG/L	UG/KG	MG/KG	MG/KG	
5872	ETHION	UG/L	UG/KG	MG/KG	MG/KG	
5843	ETHOPROP	UG/L	UG/KG	MG/KG	MG/KG	
7155	ETHYL BENZENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6001	ETHYLENE GLYCOL	UG/L	UG/KG	MG/KG	MG/KG	
4060	FDCC BLUE DYE	MG/L	MG/KG			
5854	FENITROTHION (SUMITHION)	UG/L	UG/KG	MG/KG	MG/KG	
5841	FENSULFOTHION	UG/L	UG/KG	MG/KG	MG/KG	
5882	FENTHION	UG/L	UG/KG	MG/KG	MG/KG	
1030	FLASH POINT				DEG C	
8015	FLOW	MGD				
6170	FLUORANTHENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6130	FLUORENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
3030	FLUORIDE	MG/L	MG/KG	MG/KG	MG/KG	
3031	FLUORINE				%	
5812	FONOFOS (DYFONATE)	UG/L	UG/KG	MG/KG	MG/KG	
6715	FORMALDEHYDE	UG/L	UG/KG	MG/KG	MG/KG	
1027	FREE LIQUID		ML/KG		ML/KG	
1048	GEOTECH PARAMETERS					
1115	GROSS ALPHA, TOTAL	PC/L	PC/G			
1120	GROSS BETA, TOTAL	PC/L	PC/G			
5879	GUTHION	UG/L	UG/KG	MG/KG	MG/KG	
4051	HALOGEN, PURGEABLE ORGANIC	UG/L				
4050	HALOGEN, TOTAL ORGANIC	UG/L	UG/KG		MG/KG	
1035	HARDNESS (AS CACO3)	MG/L				
1031	HEAT CONTENT (HEAT OF COMBUSTION)		BTU/#		BTU/#	
5010	HEPTACHLOR	UG/L	UG/KG	MG/KG	MG/KG	
5015	HEPTACHLOR EPOXIDE	UG/L	UG/KG	MG/KG	MG/KG	
410	HEPTACHLORODIBENZODIOXIN (TOTAL)	NG/L	NG/KG	NG/KG	NG/KG	
6784	HEPTACHLORODIBENZODIOXIN (TOTAL)	NG/L	NG/KG	NG/KG	NG/KG	
409	HEPTACHLORODIBENZODIOXIN, 1,2,3,4,6,7,8-	NG/L	NG/KG	NG/KG	NG/KG	
424	HEPTACHLORODIBENZOFURAN (TOTAL)	NG/L	NG/KG	NG/KG	NG/KG	
6790	HEPTACHLORODIBENZOFURAN (TOTAL)	NG/L	NG/KG	NG/KG	NG/KG	

Table 7-3

TEST #	TEST DESCRIPTION	UNITS WATER	UNITS SOLIDS	UNITS TISSUE	UNITS WASTE	UNITS AIR
422	HEPTACHLORODIBENZOFURAN, 1,2,3,4,6,7,8-	NG/L	NG/KG	NG/KG	NG/KG	
423	HEPTACHLORODIBENZOFURAN, 1,2,3,4,7,8,9-	NG/L	NG/KG	NG/KG	NG/KG	
5190	HEPTACHLORONORBORNENE (HCNB)	UG/L	UG/KG	MG/KG	MG/KG	
5786	HERBAN (NOREA)	UG/L	UG/KG	MG/KG	MG/KG	
7910	HEXACHLORO-1,3-BUTADIENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6145	HEXACHLOROBENZENE (HCB) (EXTRACTABLE)*	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
5200	HEXACHLOROBENZENE (HCB) (PESTICIDE)	UG/L	UG/KG	MG/KG	MG/KG	
6065	HEXACHLOROBUTADIENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6090	HEXACHLOROCYCLOPENTADIENE (HCCP)	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
408	HEXACHLORODIBENZODIOXIN (TOTAL)	NG/L	NG/KG	NG/KG	NG/KG	
6783	HEXACHLORODIBENZODIOXIN (TOTAL)	NG/L	NG/KG	NG/KG	NG/KG	
405	HEXACHLORODIBENZODIOXIN, 1,2,3,4,7,8-	NG/L	NG/KG	NG/KG	NG/KG	
406	HEXACHLORODIBENZODIOXIN, 1,2,3,6,7,8-	NG/L	NG/KG	NG/KG	NG/KG	
407	HEXACHLORODIBENZODIOXIN, 1,2,3,7,8,9-	NG/L	NG/KG	NG/KG	NG/KG	
421	HEXACHLORODIBENZOFURAN (TOTAL)	NG/L	NG/KG	NG/KG	NG/KG	
6789	HEXACHLORODIBENZOFURAN (TOTAL)	NG/L	NG/KG	NG/KG	NG/KG	
417	HEXACHLORODIBENZOFURAN, 1,2,3,4,7,8-	NG/L	NG/KG	NG/KG	NG/KG	
418	HEXACHLORODIBENZOFURAN, 1,2,3,6,7,8-	NG/L	NG/KG	NG/KG	NG/KG	
419	HEXACHLORODIBENZOFURAN, 1,2,3,7,8,9-	NG/L	NG/KG	NG/KG	NG/KG	
420	HEXACHLORODIBENZOFURAN, 2,3,4,6,7,8-	NG/L	NG/KG	NG/KG	NG/KG	
6045	HEXACHLOROETHANE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
5185	HEXACHLORONORBORNADIENE (HCNBD)	UG/L	UG/KG	MG/KG	MG/KG	
6796	HMX (EXPLOSIVE)	UG/L	UG/KG	MG/KG	MG/KG	
6712	HYDROCARBONS, TOTAL PETROLEUM(TPHC)	MG/L	MG/KG	MG/KG	MG/KG	
6819	HYDROCARBONS, TOTAL POLYAROMATIC(PAH)	UG/L	UG/KG	MG/KG	MG/KG	
3023	HYDROGEN				%	
5150	HYDROXYCHLORDENE, 1- /2	UG/L	UG/KG	MG/KG	MG/KG	
6225	INDENO (1,2,3-CD) PYRENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
2145	IRON*	MG/L	MG/KG	MG/KG	MG/KG	
2146	IRON (LOW LEVEL)	UG/L	MG/KG	MG/KG	MG/KG	
2730	IRON, DISSOLVED	UG/L				
5840	ISODRIN	UG/L	UG/KG	MG/KG	MG/KG	
6085	ISOPHORONE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7733	ISOPROPYL ETHER	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7900	ISOPROPYLBENZENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7906	ISOPROPYLTOLUENE, P-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
2720	LANTHANUM	UG/L	MG/KG	MG/KG	MG/KG	
2060	LEAD	UG/L	MG/KG	MG/KG	MG/KG	

TEST #	TEST DESCRIPTION	UNITS WATER	UNITS SOLIDS	UNITS TISSUE	UNITS WASTE	UNITS AIR
4015	LINEAR ALKYL SULFONATE	MG/L	MG/KG	MG/KG	MG/KG	
2726	LITHIUM	UG/L	MG/KG	MG/KG	MG/KG	
2140	MAGNESIUM*	MG/L	MG/KG	MG/KG	MG/KG	
2141	MAGNESIUM (LOW LEVEL)	UG/L	MG/KG	MG/KG	MG/KG	
5875	MALATHION	UG/L	UG/KG	MG/KG	MG/KG	
5901	MALINATE	UG/L	UG/KG	MG/KG	MG/KG	
6804	MALONONITRILE	UG/L	UG/KG	MG/KG	MG/KG	
2130	MANGANESE	UG/L	MG/KG	MG/KG	MG/KG	
5807	MCPA	UG/L	UG/KG	MG/KG	MG/KG	
5808	MCPP	UG/L	UG/KG	MG/KG	MG/KG	
2122	MERCURY, DIMETHYL-(AS MERCURY)	NG/L	UG/KG	UG/KG		
6002	MERCURY, DIMETHYL- (EXTRACTABLE)	UG/L	UG/KG	MG/KG	MG/KG	
2124	MERCURY, MONOETHYL-(AS MERCURY)	NG/L	UG/KG	UG/KG		
2123	MERCURY, MONOMETHYL-(AS MERCURY)	NG/L	UG/KG	UG/KG		
2120	MERCURY, TOTAL	UG/L	MG/KG	MG/KG	MG/KG	
2121	MERCURY, TOTAL UTL	NG/L	UG/KG	UG/KG		
5788	MERPHOS (FOLEX)	UG/L	UG/KG	MG/KG	MG/KG	
7732	METHANE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
5877	METHOMYL (LANNATE)	UG/L	UG/KG	MG/KG	MG/KG	
5175	METHOXYCHLOR	UG/L	UG/KG	MG/KG	MG/KG	
7142	METHYL BUTYL KETONE (2-HEXANONE)	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7058	METHYL ETHYL KETONE (2-BUTANONE)	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7086	METHYL ISOBUTYL KETONE (4-METHYL-2-PENTANONE)	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
5865	METHYL PARATHION	UG/L	UG/KG	MG/KG	MG/KG	
6280	METHYL-4,6-DINITROPHENOL, 2-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7045	METHYLENE CHLORIDE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6066	METHYLNAPHTHALENE, 2-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6243	METHYLPHENOL, (3-AND/OR 4-)	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6242	METHYLPHENOL, 2-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6244	METHYLPHENOL, 4-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
5853	METOLACHLOR	UG/L	UG/KG	MG/KG	MG/KG	
5780	MEVINPHOS (PHOSDRIN)	UG/L	UG/KG	MG/KG	MG/KG	
5837	MIREX	UG/L	UG/KG	MG/KG	MG/KG	
6999	MISCELLANEOUS EXTRACTABLES	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
5999	MISCELLANEOUS PESTICIDES	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7999	MISCELLANEOUS VOLATILES	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
5802	MOLINATE	UG/L	UG/KG	MG/KG	MG/KG	
2050	MOLYBDENUM	UG/L	MG/KG	MG/KG	MG/KG	
6807	MONOBROMOACETIC ACID	UG/L	UG/KG	MG/KG	MG/KG	

TEST #	TEST DESCRIPTION	UNITS WATER	UNITS SOLIDS	UNITS TISSUE	UNITS WASTE	UNITS AIR
6771	MONOBUTYL TIN	UG/L	UG/KG	MG/KG	MG/KG	
6808	MONOCHLOROACETIC ACID	UG/L	UG/KG	MG/KG	MG/KG	
6774	MONOPHENYL TIN	UG/L	UG/KG	MG/KG	MG/KG	
6055	N-NITROSODI-N-PROPYLAMINE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6010	N-NITROSODIMETHYLAMINE	UG/L	UG/KG	MG/KG	MG/KG	
6140	N-NITROSODIPHENYLAMINE/DIPHENYLAMINE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6075	NAPHTHALENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
2055	NICKEL	UG/L	MG/KG	MG/KG	MG/KG	
3035	NITRATE/NITRITE NITROGEN	MG/L	MG/KG	MG/KG	MG/KG	
3036	NITRATE/NITRITE NITROGEN, DISSOLVED	MG/L				
3033	NITRATE/NITROGEN	MG/L	MG/KG	MG/KG	MG/KG	
3034	NITRITE/NITROGEN	MG/L				
6096	NITROANILINE, 2-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6121	NITROANILINE, 3-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6126	NITROANILINE, 4-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6060	NITROBENZENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6778	NITRODIPHENYLAMINE, 2-	UG/L	UG/KG	MG/KG	MG/KG	
3032	NITROGEN				%	
3065	NITROGEN, TOTAL KJELDAHL	MG/L	MG/KG	MG/KG	MG/KG	
3066	NITROGEN, TOTAL KJELDAHL (DISSOLVED)	MG/L				
6245	NITROPHENOL, 2-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6290	NITROPHENOL, 4-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
5170	NONACHLOR, CIS- /2	UG/L	UG/KG	MG/KG	MG/KG	
5160	NONACHLOR, TRANS-/2	UG/L	UG/KG	MG/KG	MG/KG	
5195	OCTACHLOROCYCLOPENTENE (OCCP)	UG/L	UG/KG	MG/KG	MG/KG	
411	OCTACHLORODIBENZODIOXIN	NG/L	NG/KG	NG/KG	NG/KG	
6785	OCTACHLORODIBENZODIOXIN	NG/L	NG/KG	NG/KG	NG/KG	
425	OCTACHLORODIBENZOFURAN	NG/L	NG/KG	NG/KG	NG/KG	
6791	OCTACHLORODIBENZOFURAN	NG/L	NG/KG	NG/KG	NG/KG	
5855	OCTACHLORONAPHTHALENE	UG/L	UG/KG	MG/KG	MG/KG	
1011	ODOR (60 DEGREE C)	TOD				
1012	ODOR (ROOM TEMP)	TOD				
4020	OIL AND GREASE	MG/L	MG/KG	MG/KG	MG/KG	
4025	OIL IDENTIFICATION					
5866	ORDRAM	UG/L	UG/KG	MG/KG	MG/KG	
5805	ORTHENE (ACEPHATE)	UG/L	UG/KG	MG/KG	MG/KG	
5796	OXAMYL	UG/L	UG/KG	MG/KG	MG/KG	
5850	OXYCHLORDANE	UG/L	UG/KG	MG/KG	MG/KG	
5172	OXYCHLORDANE (OCTACHLOREPOXIDE)/2	UG/L	UG/KG	MG/KG	MG/KG	
3019	OXYGEN				%	

Table 7-3

TEST #	TEST DESCRIPTION	UNITS WATER	UNITS SOLIDS	UNITS TISSUE	UNITS WASTE	UNITS AIR
8011	OXYGEN, DISSOLVED(ELECTRODE)	MG/L				
8010	OXYGEN, DISSOLVED(WINKLER)	MG/L				
5815	PARAQUAT	UG/L	UG/KG	MG/KG	MG/KG	
5870	PARATHION, ETHYL-	UG/L	UG/KG	MG/KG	MG/KG	
1047	PARTICULATE, TOTAL SUSPENDED					
5813	PEBULATE (TILLAM)	UG/L	UG/KG	MG/KG	MG/KG	
6286	PENTACHLOROANISOLE	UG/L	UG/KG	MG/KG	MG/KG	
6817	PENTACHLOROBENZENE	UG/L	UG/KG	MG/KG	MG/KG	
404	PENTACHLORODIBENZODIOXIN (TOTAL)	NG/L	NG/KG	NG/KG	NG/KG	
6782	PENTACHLORODIBENZODIOXIN (TOTAL)	NG/L	NG/KG	NG/KG	NG/KG	
403	PENTACHLORODIBENZODIOXIN, 1,2,3,7,8-	NG/L	NG/KG	NG/KG	NG/KG	
416	PENTACHLORODIBENZOFURAN (TOTAL)	NG/L	NG/KG	NG/KG	NG/KG	
6788	PENTACHLORODIBENZOFURAN (TOTAL)	NG/L	NG/KG	NG/KG	NG/KG	
414	PENTACHLORODIBENZOFURAN, 1,2,3,7,8-	NG/L	NG/KG	NG/KG	NG/KG	
415	PENTACHLORODIBENZOFURAN, 2,3,4,7,8-	NG/L	NG/KG	NG/KG	NG/KG	
6818	PENTACHLORONITROBENZENE	UG/L	UG/KG	MG/KG	MG/KG	
6285	PENTACHLOROPHENOL	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
8020	PH (FIELD)	SU	SU	SU	SU	
1013	PH (LABORATORY)	PHUN	PHUN		PHUN	
1021	PH (METHOD 9040)	PHUN	PHUN		PHUN	
1019	PH (METHOD 9045)	PHUN	PHUN		PHUN	
1022	PH (METHOD 9045B)	PHUN	PHUN	PHUN	PHUN	
6155	PHENANTHRENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6250	PHENOL	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
4030	PHENOLS (4AAP)	UG/L	MG/KG	MG/KG	MG/KG	
5900	PHORATE (THIMET)	UG/L	UG/KG	MG/KG	MG/KG	
5816	PHOSDRIN	UG/L	UG/KG	MG/KG	MG/KG	
3040	PHOSPHORUS, ORTHO-PHOSPHATE	MG/L	MG/KG	MG/KG	MG/KG	
3070	PHOSPHORUS, TOTAL	MG/L	MG/KG	MG/KG	MG/KG	
3072	PHOSPHORUS, TOTAL(LOW LEVEL)	UG/L	UG/KG	UG/KG	UG/KG	
3060	PHOSPHORUS, TOTAL DISSOLVED	MG/L				
5784	PICLORAM	UG/L	UG/KG	MG/KG	MG/KG	
5898	PIPERONYL BUTOXIDE	UG/L	UG/KG	MG/KG	MG/KG	
2160	POTASSIUM*	MG/L	MG/KG	MG/KG	MG/KG	
2161	POTASSIUM (LOW LEVEL)	UG/L	MG/KG	MG/KG	MG/KG	
5897	PROMETON	UG/L	UG/KG	MG/KG	MG/KG	
5829	PROMETRYNE	UG/L	UG/KG	MG/KG	MG/KG	
5903	PROPACHLOR	UG/L	UG/KG	MG/KG	MG/KG	
5864	PROPAZINE	UG/L	UG/KG	MG/KG	MG/KG	
5797	PROPICONAZOLE (TILT)	UG/L	UG/KG	MG/KG	MG/KG	

TEST #	TEST DESCRIPTION	UNITS WATER	UNITS SOLIDS	UNITS TISSUE	UNITS WASTE	UNITS AIR
7901	PROPYLBENZENE, N-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6777	PROPYLENE GLYCOL DINITRATE	UG/L	UG/KG	MG/KG	MG/KG	
5890	PYDRIN (FENVALERATE)	UG/L	UG/KG	MG/KG	MG/KG	
6175	PYRENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6296	PYRIDINE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6802	QUINUCLIDINOL, 3-	UG/L	UG/KG	MG/KG	MG/KG	
1112	RADIUM-226, DISSOLVED	PC/L	PC/G			
1110	RADIUM-226, TOTAL	PC/L	PC/G			
1113	RADIUM-228, DISSOLVED	PC/L	PC/G			
1111	RADIUM-228, TOTAL	PC/L				
6797	RDX (EXPLOSIVE)	UG/L	UG/KG	MG/KG	MG/KG	
1049	REACTIVITY PARAMETERS					
5826	ROZOL	UG/L	UG/KG	MG/KG	MG/KG	
9998	SAMPLE WT					
5500	SCAN, DDT					
400	SCAN, DIOXIN	NG/L	NG/KG	NG/KG	NG/KG	
300	SCAN, EP-TOX					
6000	SCAN, EXTRACTABLES					
5800	SCAN, HERBICIDES					
2000	SCAN, METALS					
3000	SCAN, NUTRIENTS					
5001	SCAN, PCB					
5000	SCAN, PESTICIDES					
7000	SCAN, VOLATILES					UG/M3
2070	SELENIUM	UG/L	MG/KG	MG/KG	MG/KG	
5891	SENCOR (METRIBUZIN)	UG/L	UG/KG	MG/KG	MG/KG	
5893	SEVIN (CARBARYL)	UG/L	UG/KG	MG/KG	MG/KG	
3046	SILICA (SIO2)	MG/L	MG/KG	MG/KG	MG/KG	
3045	SILICON (SI)	UG/L	MG/KG	MG/KG	MG/KG	
2005	SILVER	UG/L	MG/KG	MG/KG	MG/KG	
5210	SILVEX (2,4,5-TP)	UG/L	UG/KG	MG/KG	MG/KG	
5827	SIMAZINE	UG/L	UG/KG	MG/KG	MG/KG	
2150	SODIUM*	MG/L	MG/KG	MG/KG	MG/KG	
2151	SODIUM (LOW LEVEL)	UG/L	MG/KG	MG/KG	MG/KG	
1053	SOLIDS, % FIXED	%	%			
1051	SOLIDS, % TOTAL	%	%			
1052	SOLIDS, % VOLATILE	%	%			
1040	SOLIDS, SETTLEABLE	MG/L				
1045	SOLIDS, TOTAL	MG/L	MG/KG		MG/KG	
1050	SOLIDS, TOTAL (VOLATILE)	MG/L	MG/KG		MG/KG	

Table 7-3

TEST #	TEST DESCRIPTION	UNITS WATER	UNITS SOLIDS	UNITS TISSUE	UNITS WASTE	UNITS AIR
1065	SOLIDS, TOTAL DISSOLVED (105 DEGREE C)	MG/L				
1066	SOLIDS, TOTAL DISSOLVED (180 DEGREE C)	MG/L				
1070	SOLIDS, TOTAL DISSOLVED (VOLATILE)	MG/L				
1055	SOLIDS, TOTAL SUSPENDED	MG/L				
1060	SOLIDS, TOTAL SUSPENDED (VOLATILE)	MG/L				
1105	STANDARD PLATE COUNT, 35C, 48HR/ML					
2080	STRONTIUM	UG/L	MG/KG	MG/KG	MG/KG	
7158	STYRENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
3050	SULFATE	MG/L	MG/KG		MG/KG	
3057	SULFIDES (ION SELECTIVE ELECTRODE)	MG/L	MG/KG	MG/KG	MG/KG	
3055	SULFIDES (METHYLENE BLUE METHOD)	MG/L	MG/KG		MG/KG	
3054	SULFIDES, REACTIVE(AS H2S)	MG/L	MG/KG		MG/KG	
3056	SULFITE	MG/L				
5832	SULFOTEPP	UG/L	UG/KG	MG/KG	MG/KG	
3024	SULFUR				%	
4040	TANNIN AND LIGNIN	MG/L				
6780	TCDD, 2,3,7,8- (DIOXIN)	NG/L	NG/KG	NG/KG	NG/KG	
6786	TCDF, 2,3,7,8- (DIBENZOFURAN)	NG/L	NG/KG	NG/KG	NG/KG	
1074	TCLP (INORGANIC)					
1073	TCLP (ORGANIC)					
600	TCLP SCAN, EXTRACTABLES	MG/L	MG/L	MG/L	MG/L	
800	TCLP SCAN, HERBICIDES	MG/L	MG/L	MG/L	MG/L	
200	TCLP SCAN, METALS	MG/L	MG/L	MG/L	MG/L	
500	TCLP SCAN, PESTICIDES	MG/L	MG/L	MG/L	MG/L	
700	TCLP SCAN, VOLATILES	MG/L	MG/L	MG/L	MG/L	
2732	TECHNETIUM					
2085	TELLURIUM	UG/L	MG/KG	MG/KG	MG/KG	
8025	TEMPERATURE	DEG C			DEG C	
1017	TEQ (TOXIC EQUIVALENT VALUE, TCDD)	PPQ	PPT	PPT	PPT	
426	TEQ (TOXIC. EQUIV. VALUE, FROM I-TEF/89)	NG/L	NG/KG	NG/KG	NG/KG	
6792	TEQ (TOXICITY EQUIVALENT VALUE)	NG/L	NG/KG	NG/KG	NG/KG	
5799	TERBUFOS	UG/L	UG/KG	MG/KG	MG/KG	
5820	TERBUTRYN	UG/L	UG/KG	MG/KG	MG/KG	
6820	TETRACHLOROBENZENE, 1,2,4,5-	UG/L	UG/KG	MG/KG	MG/KG	
402	TETRACHLORODIBENZODIOXIN (TOTAL)	NG/L	NG/KG	NG/KG	NG/KG	
6781	TETRACHLORODIBENZODIOXIN (TOTAL)	NG/L	NG/KG	NG/KG	NG/KG	
401	TETRACHLORODIBENZODIOXIN, 2,3,7,8-	NG/L	NG/KG	NG/KG	NG/KG	
413	TETRACHLORODIBENZOFURAN (TOTAL)	NG/L	NG/KG	NG/KG	NG/KG	

TEST #	TEST DESCRIPTION	UNITS WATER	UNITS SOLIDS	UNITS TISSUE	UNITS WASTE	UNITS AIR
6787	TETRACHLORODIBENZOFURAN (TOTAL)	NG/L	NG/KG	NG/KG	NG/KG	
412	TETRACHLORODIBENZOFURAN, 2,3,7,8-	NG/L	NG/KG	NG/KG	NG/KG	
7151	TETRACHLOROETHANE, 1,1,1,2-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7135	TETRACHLOROETHANE, 1,1,2,2-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7140	TETRACHLOROETHENE (TETRACHLOROETHYLENE)	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6291	TETRACHLOROPHENOL, 2,3,4,6-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6766	TETRACHLOROPHENOL, 2,3,5,6-	UG/L	UG/KG	MG/KG	MG/KG	
7735	TETRAHYDROFURAN	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6799	TETRYL (EXPLOSIVE)	UG/L	UG/KG	MG/KG	MG/KG	
2095	THALLIUM	UG/L	MG/KG	MG/KG	MG/KG	
3029	THIOCYANATE	MG/L	MG/KG	MG/KG	MG/KG	
2075	TIN	UG/L	MG/KG	MG/KG	MG/KG	
2090	TITANIUM	UG/L	MG/KG	MG/KG	MG/KG	
6798	TNT (EXPLOSIVE)	UG/L	UG/KG	MG/KG	MG/KG	
7145	TOLUENE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
5120	TOXAPHENE	UG/L	UG/KG	MG/KG	MG/KG	
1075	TOXICITY (EP)	MG/L				
5892	TREFLAN (TRIFLURALIN)	UG/L	UG/KG	MG/KG	MG/KG	
5823	TRIALLATE	UG/L	UG/KG	MG/KG	MG/KG	
6773	TRIBUTYL TIN	UG/L	UG/KG	MG/KG	MG/KG	
6809	TRICHLOROACETIC ACID	UG/L	UG/KG	MG/KG	MG/KG	
6814	TRICHLOROACETONITRILE	UG/L	UG/KG	MG/KG	MG/KG	
7912	TRICHLOROBENZENE, 1,2,3-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6710	TRICHLOROBENZENE, 1,2,3-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7909	TRICHLOROBENZENE, 1,2,4-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6070	TRICHLOROBENZENE, 1,2,4-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6705	TRICHLOROBENZENE, 1,3,5-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7075	TRICHLOROETHANE, 1,1,1-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7115	TRICHLOROETHANE, 1,1,2-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7100	TRICHLOROETHENE (TRICHLOROETHYLENE)	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7010	TRICHLOROFLUOROMETHANE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6266	TRICHLOROPHENOL, 2,4,5-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6265	TRICHLOROPHENOL, 2,4,6-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7175	TRICHLOROPROPANE, 1,2,3-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6815	TRICHLOROPROPANONE, 1,1,1-	UG/L	UG/KG	MG/KG	MG/KG	
5798	TRIDEMORPH (CALIXIN)	UG/L	UG/KG	MG/KG	MG/KG	
7904	TRIMETHYLBENZENE, 1,2,4-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7902	TRIMETHYLBENZENE, 1,3,5-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
6801	TRINITROBENZENE, 1,3,5-	UG/L	UG/KG	MG/KG	MG/KG	

TEST #	TEST DESCRIPTION	UNITS WATER	UNITS SOLIDS	UNITS TISSUE	UNITS WASTE	UNITS AIR
6776	TRIPHENYL TIN	UG/L	UG/KG	MG/KG	MG/KG	
1080	TURBIDITY	NTU				
2731	URANIUM 234					
2725	URANIUM, TOTAL METAL	UG/L	MG/KG	MG/KG	MG/KG	
2100	VANADIUM	UG/L	MG/KG	MG/KG	MG/KG	
5814	VERNAM (VERNOLATE)	UG/L	UG/KG	MG/KG	MG/KG	
7054	VINYL ACETATE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7035	VINYL CHLORIDE	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7156	XYLENE, (M- AND/OR P-)	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7160	XYLENE, M-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7157	XYLENE, O-	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
7165	XYLENE, O- (MIXED)					UG/M3
7170	XYLENES, TOTAL	UG/L	UG/KG	MG/KG	MG/KG	UG/M3
2105	YTTRIUM	UG/L	MG/KG	MG/KG	MG/KG	
1084	ZEAXANTHIN A (HPLC)	UG/L	UG/KG	MG/KG	MG/KG	
2110	ZINC	UG/L	MG/KG	MG/KG	MG/KG	
2115	ZIRCONIUM	UG/L	MG/KG	MG/KG	MG/KG	

8. SAMPLE COLLECTION AND HANDLING

8.1. Sample Collection - Water

8.1.1. Water samples should be collected using standard field sampling techniques consistent with the parameter being determined. Sampling procedures are followed that minimize the possibility of sample adulteration by either the sample collector or sampling device. Field sample collection procedures are detailed in the Environmental Investigations Branch, Standard Operating Procedures and Quality Assurance Manual.

8.1.2. Sample Containers and Sample Preservation: Containers and preservation techniques used must be consistent with the recommendations contained in Table 8-1.

8.1.2.1. Selection of sample container types and preservation techniques are further guided by the method being applied. Additional guidance is available in references, e.g., Standard Methods for the Examination of Water and Wastewater, ASTM, Book of Standards, Volume 11.01 and 11.02 and EPA Methods for Chemical Analyses of Water and Waste.

8.1.2.2. Samples must be accompanied by proper identification, e.g., tags, labels, and chain-of-custody forms. Sample source, date of collection, time of collection, and analysis required must be provided.

8.1.2.3. Laboratory pure water blanks are prepared containing the preservative for each type of sample collected, such as metals, nutrients, phenols, etc. The same preservative is used for both blanks and samples. The blanks are then analyzed along with the samples for the constituents of interest.

8.2. Sample Handling - Water

8.2.1. Handling of samples must be done in a manner that both insures the integrity of the sample and minimizes sample alteration. Sample custody is handled according to the procedures outlined in Section 3 of this document.

8.2.2. When samples are not analyzed within the recommended holding time, a notation of this will be made in the final data report.

8.2.3. Intralaboratory sample control and handling is the responsibility of a project analyst.

8.3. Sample Collection and Handling - Other Substrates

8.3.1. Air, sediment, sludge, plant, and animal tissue samples, should be collected using techniques consistent with the parameter being determined and with the recommendations contained in Table 8-1. Sampling procedures are followed that minimize the possibility of sample adulteration either by the sample collector or sampling device.

8.3.2. Sediment and sludge samples for organic analyses must be collected in glass containers with Teflon or aluminum-foil-lined caps. Samples must be maintained at $4^{o}C$ and analyzed as soon as possible after collection. Sediment samples for extractable organic analyses

must be in 4 oz or 8 oz glass bottles. Samples for VOA analyses must be in 40 ml VOA vials or 4 oz wide mouth jars.

8.3.3. Sediment and sludge samples for nutrient and metal analyses should be collected in glass or plastic jars and cooled to 4°C.

8.3.4. Tissues from specific organs of fish or whole fish specimens should be frozen immediately after collection.

8.3.4.1. If organic analyses are to be performed on fish tissue, the tissue should be wrapped in aluminum foil (shiny side out) prior to freezing.

8.3.4.2. For metal analyses, fish are wrapped in aluminum foil and then placed in plastic bags. Past studies have indicated little or no problems due to aluminum contamination.

RECOMMENDED SAMPLE CONTAINERS, SAMPLE PRESERVATION, SAMPLE HOLDING TIMES, AND PERMISSIBLE SAMPLE TYPE

Parameter	Container	Permissible Preservative	Holding Time	Sample Type	Reference
Concentrated Waste Samples					
Organic Compounds-Extractable and Pesticide/PCBs	8-oz. widemouth glass with Teflon liner	None	14 days	G or C	A
Organic Compounds-Purgeable (VOA)	2-oz.(60-mL) VOA container with Teflon lined Septum sealed caps	None	14 days	G or C	A
Metals and Other Inorganic Compounds	8-oz. widemouth glass with Teflon liner	None	Not Specified	G or C	A
EP Toxicity	8-oz. widemouth glass with Teflon liner	None	Not Specified	G or C	B
TCLP Purgeable Organics (VOA)	2-oz.(60-mL) VOA container with Teflon lined Septum sealed caps[1]	None	28 days[2]	G or C	A
TCLP Extractable Organics, Herbicides and Pesticide/PCBs	8-oz. widemouth glass with Teflon liner[1]	None	54 days[2]	G or C	A
TCLP Mercury	8-oz. widemouth glass with Teflon liner[1]	None	56 days[2]	G or C	A
TCLP Metals except mercury	Sample will be taken from TCLP Mercury container[1]	None	360 days[2]	G or C	A
Flash Point and/or Heat Content	8-oz. widemouth glass with Teflon liner	None	Not Specified	G	B

RECOMMENDED SAMPLE CONTAINERS, SAMPLE PRESERVATION, SAMPLE HOLDING TIMES, AND PERMISSIBLE SAMPLE TYPE

Parameter	Container	Permissible Preservative	Holding Time	Sample Type	Reference
Fish Samples					
Organic Compounds	Wrap in aluminum foil (Shiney side out)	Freeze	Not Specified	G or C	
Metals and Other Inorganic Compounds	Place in plastic zip-lock bag	Freeze	Not Specified	G or C	
Water - Low to Medium Concentration Samples					
Alkalinity	500-ml or 1-liter poly-ethylene with polyethylene or polyethylene lined closure	Cool, 4°	14 days	G or C	C
Acidity	500-ml or 1-liter poly-ethylene with polyethylene or polyethylene lined closure	Cool, 4°C	14 days	G or C	C
Bacteriological	250-ml glass with glass closure or plastic capable of being autoclaved	Cool, 4°C	6 hrs.	G	C
Static Bioassay	1-gal. amber glass (not solvent rinsed)	Cool, 4°C	48 hrs.	G or C	D

RECOMMENDED SAMPLE CONTAINERS, SAMPLE PRESERVATION, SAMPLE HOLDING TIMES, AND PERMISSIBLE SAMPLE TYPE

Parameter	Container	Permissible Preservative	Holding Time	Sample Type	Reference
Water - Low to Medium Concentration Samples (contd)					
Biochemical Oxygen Demand (BOD)	1/2-gal. polyethylene[3] with polyethylene closure	Cool, 4°C	48 hrs.	G or C	C
Chloride	500-ml or 1-liter poly-[3] ethylene with polyethylene or polyethylene lined closure	None	28 days	G or C	C
Chlorine Residual	In-situ, beaker or bucket	None	Analyze Immediately	G	C
Color	500-ml or 1-liter poly-[3] ethylene with polyethy-lene or polyethylene lined closure	Cool, 4°C	48 hrs.	G or C	C
Conductivity	500-ml or 1-liter poly-[3] ethylene with polyethy-lene or polyethylene lined closure	Cool, 4°C	28 days (determine on site if possible)	G or C	C
Chromium, Hexavalent	1-liter polyethylene with polyethylene closure	Cool, 4°C	24 hrs.	G	C
Cyanide	1-liter or 1/2-gallon polyethylene with poly-ethylene or polyethylene lined closure	Ascorbic Acid[4,5] sodium Hydroxide, pH >12 Cool, 4°C	14 days	G	C

RECOMMENDED SAMPLE CONTAINERS, SAMPLE PRESERVATION, SAMPLE HOLDING TIMES, AND PERMISSIBLE SAMPLE TYPE

Parameter	Container	Permissible Preservative	Holding Time	Sample Type	Reference
Water - Low to Medium Concentration Samples (Continued)					
Dissolved Oxygen (Probe)	In-situ, beaker or bucket	None	Determine On Site	G	C
Dissolved Oxygen (Winkler)	300-ml glass, BOD bottle	Fix on site, store in dark	8 hrs. (determine on site if possible)	G	C
EP Toxicity	1-gal. glass (amber) with Teflon liner	Cool, 4°C	Not Specified	G or C	B
Fluoride	1-liter polyethylene or[3] 1/2-gal. polyethylene with polyethylene or polyethylene lined closure	None	28 days	G or C	C
Hardness	500-ml or 1-liter polyethylene with polyethylene or polyethylene lined closure	50% Nitric[4] Acid, pH <2	6 months	G or C	C
LAS	500-ml or 1-liter poly-[3] ethylene with polyethylene or polyethylene lined closure	Cool, 4°C	48 hrs.	G or C	C

RECOMMENDED SAMPLE CONTAINERS, SAMPLE PRESERVATION, SAMPLE HOLDING TIMES, AND PERMISSIBLE SAMPLE TYPE

Parameter	Container	Permissible Preservative	Holding Time	Sample Type	Reference
Water - Low to Medium Concentration Samples (Continued)					
Metals	1-liter polyethylene with polyethylene lined closure	50% Nitric[4] Acid, pH <2	6 months	G or C	C
Metals, Dissolved	1-liter polyethylene with polyethylene lined closure	Filter-on-site[4] 50% Nitric Acid, pH <2	6 months	G	C
Nutrients[6]	1-liter polyethylene or 1/2-gal. polyethylene with polyethylene or polyethylene lined closure	50% Sulfuric[4] Acid, pH <2 Cool, 4°C	28 days	G or C	C
Oil and grease	1-liter widemouth glass with Teflon lined cap	50% Sulfuric[4] Acid, pH <2 Cool, 4°C	28 days	G	C
Organic Compounds -					
Extractable and Pesticide Scan					
No Residual Chlorine Present	1-gal. amber glass or 2 1/2-gal. amber glass with Teflon lined cap	Cool, 4°C	47 days[7]	G or C	A or C

RECOMMENDED SAMPLE CONTAINERS, SAMPLE PRESERVATION, SAMPLE HOLDING TIMES, AND PERMISSIBLE SAMPLE TYPE

Parameter	Container	Permissible Preservative	Holding Time	Sample Type	Reference
Water - Low to Medium Concentration Samples (Continued)					
Residual Chlorine Present	1-gal. amber glass or 2 1/2-gal. amber glass with Teflon lined cap	Add 3 ml 10% sodium thiosulfate per gallon Cool, 4°C	47 days[7]	G or C	A or C
Organic Compounds - Purgeable (VOA)					
No Residual Chlorine Present	3 40-ml vials with Teflon lined septum sealed caps	4 drops 1+1 hydrochloric acid, Cool, 4°C	14 days	G	A or C
No Residual Chlorine Present	3 40-ml vials with Teflon lined septum sealed caps	Cool, 4°C	7 days	G	A or C
Residual Chlorine Present	3 40-ml vials with Teflon lined septum sealed caps	Footnote 8	14 days	G	A or C
Organic Compounds - Specified and Pesticides (Non-Priority Pollutants such as Herbicides)	1-gal. glass (amber) or 2 1/2-gal. glass (amber) with Teflon lined closure	Footnote 9	47 days[7]	G or C	A or C

RECOMMENDED SAMPLE CONTAINERS, SAMPLE PRESERVATION, SAMPLE HOLDING TIMES, AND PERMISSIBLE SAMPLE TYPE

Parameter	Permissible Container	Holding Preservative	Sample Time	Type	Reference
Water - Low to Medium Concentration Samples (Continued)					
Organic Halides - Total (TOX)	250-ml amber glass with Teflon lined septum closure	Cool, 4°C H_2SO_4 to pH<2	28 days	G	A or E
pH	In-situ, beaker or bucket	None	Analyze Immediately	G	C
Phenols	1-liter amber glass with Teflon lined closure	50% Sulfuric Acid, pH <2 Cool, 4°C	28 days	G	C
Phosphate-Ortho	500-ml or 1-liter polyethylene with polyethylene or polyethylene lined closure	Filter-on-site Cool, 4°C	48 hrs.	G	C
Phosphorus, Total Dissolved	500-ml or 1-liter polyethylene with polyethylene or polyethylene lined closure	Filter-on-site 50% Sulfuric Acid, pH <2 Cool, 4°C	28 days	G	C
Solids, Settleable	1/2-gal. polyethylene with polyethylene closure	Cool, 4°C	48 hrs.	G or C	C

RECOMMENDED SAMPLE CONTAINERS, SAMPLE PRESERVATION, SAMPLE HOLDING TIMES, AND PERMISSIBLE SAMPLE TYPE

Parameter	Container	Permissible Preservative	Holding Time	Sample Type	Reference
Water - Low to Medium Concentration Samples (Continued)					
Organic Halides - Total (TOX)	250-ml amber glass with Teflon lined septum closure	Cool, 4°C H_2SO_4 to pH<2	28 days	G	A or E
pH	In-situ, beaker or bucket	None	Analyze Immediately	G	C
Phenols	1-liter amber glass with Teflon lined closure	50% Sulfuric Acid, pH <2 Cool, 4°C	28 days	G	C
Phosphate-Ortho	500-ml or 1-liter polyethylene with polyethylene or polyethylene lined closure	Filter-on-site Cool, 4°C	48 hrs.	G	C
Phosphorus, Total Dissolved	500-ml or 1-liter polyethylene with polyethylene or polyethylene lined closure	Filter-on-site 50% Sulfuric Acid, pH <2 Cool, 4°C	28 days	G	C
Solids, Settleable	1/2-gal. polyethylene with polyethylene closure	Cool, 4°C	48 hrs.	G or C	C

RECOMMENDED SAMPLE CONTAINERS, SAMPLE PRESERVATION, SAMPLE HOLDING TIMES, AND PERMISSIBLE SAMPLE TYPE

Parameter	Permissible Container	Preservative	Holding Time	Sample Type	Reference
Soil, Sediment or Sludge Samples - Low to Medium Concentrations					
EP Toxicity	8-oz. widemouth glass with Teflon\ lined closure	Cool, 4°C	Not Specified	G or C	B
TCLP Purgeable Organics (VOA)	Two 2-oz. (60-mL) VOA container with Teflon lined Septum sealed caps[1]	None	28 days[2]	G or C	A
TCLP Extractable Organics, Herbicides and Pesticide/PCBs	8-oz. widemouth glass with Teflon liner[1]	None	54 days[2]	G or C	A
TCLP Mercury	8-oz. widemouth glass with Teflon liner[1]	None	56 days[2]	G or C	A
TCLP Metals except mercury	Sample will be taken from TCLP Mercury container	None	360 days[2]	G or C	A
Metals	8-oz. widemouth glass with Teflon lined closure	Cool, 4°C	6 months	G or C	A
Nutrients Including: Nitrogen, Phosphorus, Chemical Oxygen Demand	500-ml polyethylene with polyethylene closure or 8 oz. widemouth glass with Teflon lined closure	Cool, 4°C	Not Specified	G or C	A
Organics Extractable	8-oz. widemouth glass with Teflon liner	Cool, 4°C	ASAP	G or C	A

RECOMMENDED SAMPLE CONTAINERS, SAMPLE PRESERVATION, SAMPLE HOLDING TIMES, AND PERMISSIBLE SAMPLE TYPE

Parameter	Container	Permissible Preservative	Holding Time	Sample Type	Reference
Soil, Sediment or Sludge Samples - Low to Medium Concentrations					
Organics - Purgeable (VOA)	2-oz. (60 ml) VOA vial w/Teflon lined septum seal	Cool, 4°C	14 days	G or C	A
Other Inorganic Compounds - Including Cyanide	500-ml polyethylene with polyethylene closure or 8-oz. wide mouth glass with Teflon lined closure	Cool, 4°C	Not Specified	G or C	A
Municipal Sludge - Low to Medium Concentrations					
Organics - Extractable & Pesticide/PCBs	0 - 30% Solids 1- gal. amber glass or 4 qt wide mouth bottle (depending on consistency) with Teflon lined cap	Cool, 4°C	47 days[7]	G or C	F
	> 30% Solids 8-oz widemouth glass jar with Teflon lined cap	Cool, 4°C	47 days[7]	G or C	F
Organics - Purgeables (VOA)	0 - 1% Solids 3 40-mL VOA vials with Teflon lined septum sealed caps	4 drops 1+1 HCl acid, Cool, 4°C	14 days	G or C	F
	> 1% Solids 2 2-oz (60 mL) VOA vials w/ Teflon lined septum sealed cap	Cool, 4°C	14 days	G or C	F

NOTE: The Analytical Support Branch should be consulted prior to making any changes to any of the above sampling protocols.

RECOMMENDED SAMPLE CONTAINERS, SAMPLE PRESERVATION, SAMPLE HOLDING TIMES, AND PERMISSIBLE SAMPLE TYPE

Abbreviations: **G** = Grab **C** = Composite
NS = Not Specified **ASAP** = As Soon As Possible

Footnotes:

1. The TCLP method requires the leaching of 25 gm of solid for volatile organics and 100 gm of solids for all other parameters. If the sample is low in solids, additional sample containers may be required to provide sufficient sample for the TCLP leach extraction.

2. These are total holding times for TCLP that cover sampling through analysis. The holding times are broken down as follows: TCLP volatile organics - 14 days from collection to TCLP extraction plus 14 days from leach extraction to analysis; extractable organics, pesticides & herbicides - 7 days from collection to TCLP extraction plus 7 days to solvent extraction of leachate plus 40 days to analysis of extract; mercury - 28 days from collection to TCLP extraction plus 28 days to analysis; metals except mercury - 180 days from collection to TCLP extraction plus 180 days to analysis.

3. Use indicated container for single parameter requests or 1/2-gallon polyethylene container for multiple parameter requests except those including BOD. Use a 1-gallon polyethylene container for multiple parameter requests which include BOD.

4. Must be preserved in the field at time of collection.

5. Use ascorbic acid only if the sample contains residual chlorine. Test a drop of sample with potassium iodide-starch test paper; a blue color indicates need for treatment. Add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper. Then add an additional 0.6 g of ascorbic acid for each liter of sample volume.

6. May include nitrogen series (ammonia, total Kjeldahl nitrogen, nitrate-nitrite), total phosphorus, chemical oxygen demand and total organic carbon.

7. Samples must be extracted within seven days and extract must be analyzed within 40 days.

8. Collect the sample in a 4 oz. soil VOA container which has been pre-preserved with four drops of 25 percent ascorbic acid solution. Gently mix the sample and transfer to a 40 ml VOA vial that has been prepreserved with four drops 1+1 HCl, cool to 4°C.

9. See Organic Compounds - Extractable (page 8 & 9 of 15). The Analytical Support Branch should be consulted for any special organic compound analyses in order to check on special preservation requirements and or extra sample volume.

RECOMMENDED SAMPLE CONTAINERS, SAMPLE PRESERVATION, SAMPLE HOLDING TIMES, AND PERMISSIBLE SAMPLE TYPE

References

A. US-EPA, Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, Office of Solid Waste and Emergency Response, Washington,DC, Nov. 1986.

B. US-EPA, Test Methods for Evaluating Solid Waste, SW-846, Office of Solid Wastes, Washington, DC, 1982.

C. 40 CFR Part 136, Federal Register, Vol. 49, No. 209, October 26, 1984.

D. US-EPA, Region IV, Environmental Services Division, "Ecological Support Branch, Standard Operating Procedures Manual," latest version.

E. EPA Interim Method 450.1, "Total Organic Halide," US-EPA, ORD, EMSL, Physical and Chemical Methods Branch, Cincinnati, Ohio, November 1980.

F. US-EPA, Analytical Methods for the National Sewage Sludge Survey, Office of Water Regulations and Standards, Washington, DC, Aug. 1989.

9. SAMPLE RECORDS AND DATA HANDLING

9.1. Sample accountability through the analytical process can be divided into three major elements: (l) initial sample logging; (2) data acquisition, and (3) documentation/storage. The laboratory location, i.e., field or central, and the analyses requested will dictate the nature and location of the sample and data records. In addition to the procedure discussed in Section 3 of this manual, the following sections outline current sample and data documentation procedures.

9.2. Sample Logging

9.2.1. Field Laboratory Sample Logging

9.2.1.1. Samples received at a field laboratory with accompanying identification are logged into the field sample logbook. Samples are assigned sample ID and station ID.

9.3. SESD Laboratory Sample Logging

9.3.1. Samples received at the SESD laboratory with accompanying identification are logged into the Region 4 Laboratory Information Management System (R4LIMS). Samples are assigned consecutive log numbers and logged as described above.

9.3.2. Also contained in the R4LIMS is a description of the disposition of every log number used, whether in the field or SESD laboratory.

9.4. Analytical Data Handling

9.4.1. General

9.4.1.1. All raw analytical and instrument control data generated in the laboratory are entered into bound data books or kept as strip charts, or in instrument computer hardcopy, tape, or disk.

9.4.1.2. Information contained in these data logbooks includes the following: project number, sample log number, parameter, date of analysis, analyst, and all pertinent instrument identification with analytical conditions. For non-computerized instruments all calibration data, all readout data, calculation, final concentration, and quality control data should also be recorded in the log.

9.4.1.3. Final results of all analyses are provided in a standard computerized report format and forwarded to the requester with cover memorandum. Remarks should be used with reported data to alert the user to some specific condition that affects the data.

9.4.2. More specific information on data handling is contained in Sections 10 and 11.

9.5. Computerized Analytical Data System

9.5.1. Introduction

9.5.1.1. The "Region 4 Laboratory Information Management System" (R4LIMS) is a computerized data storage and laboratory information management system. R4LIMS is utilized to store project information as well as analytical results for specific samples.

9.5.1.2. R4LIMS is structured using the Oracle data base management system. R4LIMS is a very flexible, interactive system that integrates a variety of data processing functions within the structure of one high level language.

9.5.1.3. R4LIMS is located on an IBM compatible computer at the EPA, Region 4, Science and Ecosystems Technology Center in Athens, GA. All communication with R4LIMS is through the EPA Local Area Network (LAN) or Wide Area Network (WAN).

9.5.1.4. The sample custodian or field engineers are responsible for logging new projects into the system. The sample custodian logs sample related information into the system. Individual analysts are responsible for entering results and verifying their accuracy. The analysts are also responsible for reporting these results to the requestor.

9.5.2. System Description

9.5.2.1. Project Logging

9.5.2.1.1. All Analytical projects, when initiated by the requester, are logged into R4LIMS by either the project leader or the sample custodian. Four digit project numbers (prefixed by the two digit Fiscal Year) are assigned consecutively by R4LIMS starting with FY-000l at the beginning of each fiscal year (e.g.,FY=89, 90, etc). This includes all identification information for the project such as: project number, name of project, location, date project to be conducted, requester and program element, account number, time accounting information, etc.

9.5.2.1.2. If the samples from the project are to be analyzed by the Contract Laboratory Program (CLP), the project is flagged as contract and pertinent information recorded such as the contract laboratory name, case number, etc. are identified.

9.5.2.1.3. Project log also stores non-sample related project information from field investigations.

9.5.3. Sample Logging

9.5.3.1. All samples to be analyzed or tracked by R4LIMS are logged into the system when received. The samples are numbered in chronological order.

9.5.3.2. Data entered identifies and describes each sample, the tests required, and the test numbers. Test numbers are maintained in a file. A copy of the sample data log printout is filed in the project file and another copy is sent to the requester along with his copy of the custody record.

9.5.4. Analytical Data Processing

9.5.4.1. All analysis results are entered into the analytical results data bases.

9.5.4.2. The majority of all data is transmitted electronically to the Computer system.

9.5.5. Other ADP Operations

9.5.5.1. Quality Control Data. Data bases are available, or will be developed, for entering, storing and summarizing precision and accuracy data generated during sample analysis. This may include percent RSD, matrix spike recovery data, surrogate spike recovery data, results of reference sample analyses, etc. Entry and verification programs are available for this QC operation. Summary programs are available for QC reports as required.

9.5.5.2. Sample Custody Information. Sample custody information, such as custody room check-out and check-in information, sample disposal information, etc., is kept in a custody log. Information in this log and the sample log can be combined to give a complete documentation of chain-of-custody for all samples. A module to store this information is under development.

9.5.5.3. Time Accounting Information. A data base named ASBTIME, divided into fiscal years, is maintained for storage and manipulation of personnel time. All personnel time is entered by employee, pay period, activity, account number and program element. Summary reports can be generated based on the specific elements required.

9.5.5.4. Accounting Reports. Various reporting modules are available sample tracking and counting.

9.5.5.4.1. Sample Counter - Listing of number of samples received by type, program element, and whether analyses were performed by EPA or a contract lab.

9.5.5.4.2. Analysis Counter - Listing of analyses by parameter, sample type, and whether analyses were conducted by EPA or contract laboratory.

9.5.5.4.3. Total Accounting Report - Listing of analyses within each program element by parameter, sample type and whether analyses were conducted by EPA or contract laboratory.

9.5.5.5. Analytical Backlogs. Several types of backlogs can be produced that give information on completed projects, incomplete projects, and projects scheduled for the future. These are used for tracking progress of samples being analyzed and for planning analysis of samples not yet received. Tailor made requests can be submitted to report as much information as needed, or as specific as needed.

9.5.5.5.1. Analytical Backlog/Inhouse Samples: List projects in chronological order with name, project number, program element, requester, receipt data (actual or projected), projected completion date, number of samples scheduled to be received or number of samples received broken down into analytical categories (inorganics, VOA, extractable organics, pesticides, metals, and EP).

9.5.5.5.2. Analytical Backlog/Contractor Samples: Same information as above except for contract samples.

9.5.5.5.3. Detail Backlog: Listing of all required analyses, by parameter test code, sample type, project number and sample number. The listing separates in-house analyses from contractor analyses if requested.

10. ORGANIC ANALYSIS, PERFORMANCE QUALITY CONTROL AND ANALYTICAL OPERATION

10.1. Every element of environmental data acquisition, from sample collection to final data reporting, has associated with it degrees of error. The primary purpose of a total quality assurance program is the optimization of conditions whereby the introduction of error can be either precluded or substantially reduced. The operating procedures and quality control checks practiced in this laboratory and outlined in this manual are implemented to minimize the total error associated with data generation. No number can be affixed to total error; however, analytical performance is measurable and thus definable. Analyses are performed in support of EPA Programs such as RCRA, Superfund, NPDES, Drinking Water, Air Toxics, CERCLA, and other initiatives. The methods used in organic analysis are based primarily on RCRA guidance. Modifications have been made to increase quality, efficency, and to support specific requests of the various programs.

10.2. General

10.2.1. It is the policy of this Branch to apply the best laboratory practices, use approved methodology when mandated by regulation and use standardized methodology to meet quality requirements designated in the following paragraphs. When approved methodology is not applicable, fully document all operations associated with the generation of data.

10.2.2. Safety precautions associated with the safe handling of toxic chemicals, reagents, solutions and samples will be observed and regarded as a first order responsibility of the analyst. The analyst will take the necessary precautions to prevent exposure or harm to any employee.

10.3. Organic Methodology

10.3.1. Section 7 contains a listing of individual analytical methods used. Table 10-1 contains a listing of current analytical descriptors associated with these methods. These descriptors are used in sample vial labeling and file naming conventions in GC and GC/MS computer systems as appropriate.

10.4. Sample Preparation of Semivolatile fraction and Pesticide fraction

10.4.1. General Quality Control Requirements

10.4.1.1. All glassware and glass wool is rinsed sequentially with methanol, acetone, and the sample solvent, just prior to use.

10.4.1.2. A reagent blank is set up with each set of 20 or less samples when an extraction is performed or when simply putting a chemical waste sample in solution. Include all glassware and extraction equipment.

10.4.1.2.1. Water sample - Use reagent grade water and all solvents.

10.4.1.2.2. Soil/Sediment/Tissue sample - Use the appropriate amount of anhydrous sodium sulfate and all solvents and reagents.

10.4.1.2.3. Waste sample - Use anhydrous sodium sulfate (if used in the samples) and all solvents and reagents.

10.4.1.3. Duplicate matrix spikes (spike two portions of a sample expected to contain no organics or low levels) and/or duplicate method spikes (media known to be organic free, i.e., reagent grade water or for solids, clean sand and anhydrous sodium sulfate). Duplicate spikes are included with each set of 20 or less samples.

10.4.1.4. A gel permeation chromatograph (GPC) calibration standard consisting of corn oil, bis(2-ethylhexyl)phthalate, 4-nitrophenol, perylene, and sulfur must be passed through the GPC system prior to beginning cleanup of samples, once/month. This must be done more frequently after repacking the column. Adjust the collection volume to recover $\geq$ 85% of the bis(2-ethylhexyl)phthalate.

10.4.2. General Extraction Protocols

10.4.2.1. Determination of percent moisture

10.4.2.1.1. Sediment/Soil - Percent moisture must be determined on all samples unless otherwise specified.

10.4.2.1.2. Waste - Determine percent moisture if the sample is primarily heavily contaminated soil or a dry solid. This must be done in an oven located in a hood. Waste that is primarily a non-aqueous liquid does not require a percent moisture determination. See Section 3.5.6.2 for additional handling guidance.

10.4.2.2. Chlorinated water samples must be dechlorinated with sodium thiosulfate prior to extraction.

10.4.2.3. All water samples extracted for pesticide analysis from compliance sampling inspections (CSI) and toxic compliance sampling inspections (XCSI), and all water extracts with color must be passed through the alumina microcolumn.

10.4.2.4. If the final extract volume is greater than l mL, transfer at least l mL to a GC vial. The remainder is discarded. Never leave any extracts in volumetric flasks.

10.4.2.5. Labeling Laboratory Sample Containers

10.4.2.5.1. One or more of the analytical descriptors should be used as a suffix after each sample number recorded on the sample vial and in the extraction logbook (i.e. 10234SLS for a semivolatile low soil extract of sample 10234). See Table 10-1.

10.4.2.5.2. Record on the blank, spikes, and surrogate included with the set the inclusive numbers of the samples that were extracted together.

10.4.2.5.2.1. B - Blank, include inclusive sample numbers after B (e.g. B05440-0546lSLW)

10.4.2.5.2.2. S -Spike, include sample designation and sample number spiked after S (e.g. Sl2440P for pesticide spike of sample l2440)

10.4.2.5.2.3. X and Y - To designate duplicate extractions.

10.4.2.5.2.4. R, R2, R3, etc. - To designate when re-extractions are required; designate them with an "RX" depending on the number of re-extractions required.

10.4.2.5.2.5. A mark is placed on each sample vial to indicate the bottom of the meniscus when vialed.

10.4.2.5.2.6. The final extract volume is recorded on all vials and in the extraction logbook.

10.4.3. Extraction Logbook

10.4.3.1. All pertinent information requested on the sheet will be properly recorded prior to submittal to the GC and or GC/MS chemists. See Forms 10-1, 10-2, and 10-3.

10.4.3.2. List the blank and spike in the sample number column. Record the range of sample numbers that the blank and spike represent (example: blank 0l4ll-25; spike 0l4ll-25).

10.4.3.3. Record the extract volume on the sheet.

10.4.3.4. Record the designation for extract type after the sample number.

10.4.3.5. Record unusual occurrences during sample preparation, e.g., unusual appearance of sample, problems during extraction, losses of extract, precipitation and/or increase in viscosity during final evaporation, etc.

10.4.3.6. All calculations must be checked by a second person and the extraction sheet initialed by both analyst and checker.

10.4.3.7. Do not erase or use "Liquid Paper" to correct any error. Put one line through the error with initials and date.

10.4.4. Sample Vial Handling

10.4.4.1. Put all vials on one board or container that pertain to a set of samples that were extracted together, and label the board with the projects' names. The chemist in charge of the extraction laboratory should check the labeling of all vials. Do not put two separate extraction batches on one board.

10.4.4.2. Sample vials and copies of the extraction sheet should be given to the chemist in charge of the pesticide or semivolatile analysis.

10.4.4.3. Include a surrogate standard solution with each set of samples. This solution should be at the same concentration as in the sample extracts.

10.5. Surrogate Standards

10.5.1. A surrogate standard, a chemically inert compound not expected to occur in an environmental sample, is added to each sample just prior to extraction or purging. The recovery of the surrogate standard is used to monitor for unusual matrix effects, gross sample processing errors, etc. Surrogate recovery is evaluated by determining whether the measured concentration falls within the statistical acceptance limits.

10.5.2. Following are the surrogate standards and the corresponding spike solution concentrations currently used by ASB:

Semivolatile-Base/Neutral	Sol'n Conc.	Spike Amt per Final Extract Volume
Nitrobenzene - d5	1000ng/uL	50uL/1mL
Terphenyl - d13	1000ng/uL	

Semivolatile-Acid	Sol'n Conc.	Spike Amt per Final Extract Volume
2,4,6-tribromophenol	1000ng/uL	50uL/1mL
phenol - d6	1000ng/uL	

Volatiles-Water/Soil/Sed	Sol'n Conc.	Spike Amt per Final Purge Volume
toluene - d8	Method 8260	Method 8260
p-bromofluorobenzene	modifications	modifications
dibromofluoromethane		

Volatiles-Air Canister	Sol'n Conc.	Spike Amt per Final Canister Volume
toluene - d8	TO-14	TO-14
p-bromofluorobenzene	modifications	modifications
dibromofluoromethane		

Organo-chlorine Pesticides and PCBS	Sol'n Conc.	Spike Amt per Final Extract Volume
dibutylchlorendate (DBC)	40ng/uL	25uL/1mL
2,4,5,6 tetrachloro-meta-xylene (TCMX)	20ng/uL	

Phenoxy Herbicides	Sol'n Conc.	Spike Amt per Final Extract Volume
DCAA (2,4 -Dichlorophenyl-Acetic Acid)	20ng/uL	100uL/10mL

Organonitrogen/phosphate Pesticide	Sol'n Conc.	Spike Amt per Final Extract/Purge Volume
2-Nitro-m-xylene (NMX)	250ng/uL	50uL/1mL

10.5.3. Calculation of Acceptance Limits (All calculations are performed by the laboratory's computer data system).

10.5.3.1. Calculate average recovery (R) and standard deviation (S), in percent recovery, for each surrogate standard using the entire data base over a set period of time, e.g. annually.

10.5.3.2. Values greater than 3 standard deviations are eliminated from the data base as outliers. The limits are then re-calculated as above.

10.5.3.3. Calculate method performance criteria and define the performance of the laboratory for each surrogate standard being used.

10.5.3.4. Calculate upper and lower control limits for method performance and surrogate standard recovery:

Semivolatiles and Pesticides:

Upper Control Limit (UCL) = R + 2 S

Lower Control Limit (LCL) = R - 2 S

Volatiles:

Upper Control Limit (UCL) = R + 3 S

Lower Control Limit (LCL) = R - 3 S

10.5.3.5. Surrogate limits are calculated annually.

10.5.4. Analysis of Surrogates

10.5.4.1. Purgeable and Extractable Organics - All samples and blanks are to be analyzed by GC/MS. The GC/MS analyst is responsible for calculating recovery, recording the data in the GC/MS logbook and transferring it to the PC data base using appropriate software.

10.5.4.2. Pesticides/PCBs - Most samples and blanks will be analyzed by GC/EC or GC/NP for pesticides/PCBs. The analyst is responsible for keeping a hardcopy of the pesticide surrogate data in the project file as well as transferring the data to the computer data system. See Form 10-9.

$$\text{Percent Surrogate Recovery} = \frac{Qd \times 100}{Qa}$$

Where Qd = Quantity determined by analysis
Qa = Quantity added to the sample

10.5.5. Evaluation of Surrogate QC Data

10.5.5.1. Purgeable Organics - If surrogate standard recovery of any one surrogate is out of limits in a blank or a sample, proceed with corrective action.

10.5.5.2. Extractable Organics - If recovery of two surrogates from the same sample pH fraction are out of limits, proceed with corrective action. See below for action on blanks and matrix or method spikes.

10.5.5.3. Pesticide/PCB - Since GC/EC data is much more subject to interference than GC/MS, two surrogate standards are added to each sample: Dibutylchlorendate (DBC) and 2,4,5,6-tetrachloro-meta-xylene (TCMX). DBC is the primary surrogate and should be used whenever possible. However, DBC is subject to acid and base degradation so, if DBC recovery is low or compounds interfere with DBC, then the TCMX should be evaluated for acceptance. Proceed with corrective action when both surrogates are out of limits for a sample. See below for action on blanks and matrix or method spikes.

10.5.5.4. At present there are no QC limits for the herbicide and organo-nitrogen/phosphorus surrogates.

10.5.5.5. Corrective Action

10.5.5.5.1. Check for instrumental problems and make any necessary corrections. Redilute the extract (if necessary), and then rerun the sample. This also applies to blanks and matrix or method spikes.

10.5.5.5.2. If no instrumental problems exist, the sample should be re-extracted and re-analyzed. However, if the sample data from the first analysis has to be reported, report the data from the first analysis and flag it with a "J". If surrogates from extractable or pesticide blanks exceed the above criteria, but one or more samples in the set have acceptable surrogate limits, evaluate the blanks carefully to see if they still provide sufficient information to determine the presence of contaminants in the samples. For matrix or method spikes which are already prepared in duplicate, no re-extraction is required. If both duplicates are out, indicating a matrix effect, record matrix surrogate recovery data for both.

10.5.5.5.3. If the surrogates are still outside the acceptance limits after repurging or re-extraction, the data should be reported and flagged with a "J".

10.5.6. Reporting Surrogate Data

10.5.6.1. All surrogate data must be transferred to the computer data system except for surrogate data that is known to be in error; i.e., acid was not added to water prior to water extraction, valve on GPC instrument was leaking caused cross-contamination, purge and trap system contamination, etc. DBC data whose pH is outside neutral range during extraction or cleanup should also not be recorded.

10.6. Internal Standards

10.6.1. Internal standards, compounds not expected to occur in an environmental sample, are added to each sample just prior to instrumental analysis.

10.6.2. Following are the internal standards and the corresponding spike solution concentrations currently used by ASB:

Semivolatile	Sol'n Conc.	Amt per Final Extract Volume
1,4-Dichlorobenzene-d4	1000ng/uL	10uL/1mL
Naphthalene-d8	1000ng/uL	
Acenaphthene-d10	1000ng/uL	
Phenanthrene-d10	1000ng/uL	
Chrysene-d12	1000ng/uL	
Perylene-d12	1000ng/uL	

Volatiles-Water/Soil/Sed	Sol'n Conc.	Spike Amt Per Final Volume
Difluorobenzene Chlorobenzene-d5 1,4-Dichlorobenzene-d4	Method 8260 modifications	Method 8260 modifications

Volatiles-Air Canister	Sol'n Conc.	Spike Amt Per Final Volume
Difluorobenzene Chlorobenzene-d5 1,4-Dichlorobenzene-d4	TO-14 modifications	TO-14 modifications

10.7. GC Analysis

10.7.1 GC Screening

10.7.1.1 It is suggested that a GC Screening of all samples be conducted before the GC Analytical run. The following set-up is an example for screening all types of matrices.

10.7.1.1.1 Begin with an Evaluation Mix and a 100x dilution of the Surrogate standard or the dilution that is required for the surrogate to be within the standard curve range.

10.7.1.1.2 Make a 100X dilution of all extracts and run them next including the blank and spike. Include a standard and an Evaluation Mix after each 20 samples.

10.7.1.1.3 Repeat the 100X dilution of the Surrogate standard at the end of the screening run.

10.7.1.2 This run of 100X dilutions may be used to calculate the Surrogate recovery if the following procedures were done:

10.7.1.2.1 An Evaluation Mix is run both at the beginning and at the end of the run.

10.7.1.2.2 A QC curve of the Surrogate standard is run before or immediately after the screening run.

10.7.2. GC Logbook

10.7.2.1. Be sure all pertinent information requested on the sheet is properly recorded. See Form 10-8. An analyst should keep track of projects on a master log sheet. See Form 10-10.

10.7.2.2. All analysts that participated in making dilutions and/or loading the auto-sampler must record their names. This includes analysts that add extracts at the end of the run to verify or check on samples from other sets of samples.

10.7.2.3. Record inclusive sample numbers for each blank and spike.

10.7.2.4. Record the level of concentration of standard and the name of the standard (e.g., Red Pest Mix VI).

10.7.2.5. Record all information that is needed to identify the sample vial (see Table 10-1).

10.7.2.6. Record all dilutions with dilution factor, times sign, and original volume (example: 10 X 1 mL or 10,000 X of 25 mL).

10.7.2.7. A copy of the logbook page should be kept in the project file.

10.7.3. Follow the procedure for setting up instruments for data analysis, i.e., for collecting, processing, analyzing, and reporting data, using a PC with a pesticide analysis software program. Make sure that the correct time and date are on all QC runs and reports. This can be done by making sure that the processing PC and the acquiring GC are set to the correct time and date.

10.7.3.1. Build a new method or edit an existing one that is suitable for the analysis as required by the software program being used. This will include developing or updating the instrument, processing and calibrating parameters for the method.

10.7.3.2. Create a report format for the method.

10.7.3.3. Create a new Sequence file for each GC run. The sequence should include information to identify the sample, vial, and method used.

10.7.3.3.1. Give the sequence file a singular name associated with the project name.

10.7.3.4. Download a sequence or method file to the interface.

10.7.3.5. Set the GC conditions for the run and then start the GC which will begin data collection.

10.7.3.6. After collection, process the data using a pesticide analysis software protocol.

10.7.3.7. Build summary reports for QC linear curve, surrogate, and sample results.

10.7.3.8. Archive and back up all files associated with an analytical run.

10.7.4. Dilutions and Sample Vials.

10.7.4.1. The GC chemist is responsible for all sample extract vials received from the extraction lab. The chemist is responsible for the vials until GC analysis is complete, and the vials have been stored in proper order or have been discarded.

10.7.4.2. Re-mark all vials at the meniscus after dilutions or GC analysis. Do not allow original vials to remain in auto-samplers over the weekend.

10.7.4.3. One sample from every set of samples requiring dilutions will be analyzed in duplicate. Select the sample requiring the greatest dilution that has usable data. If the original dilution was made using the auto-diluter, then its duplicate should be made manually, or by a different auto-dilute, or by another analyst.

10.7.4.4. Record the QC dilutions in the GC Logbook.

10.7.4.5. The duplicate shall be made and analyzed as soon as possible after the initial dilutions are analyzed.

10.7.4.6. Data from duplicate of the greatest dilution containing usable peak(s) shall agree within 10% RSD.

10.7.4.7. If data difference is greater than 10% RSD, resolve the problem before continuing by:

10.7.4.7.1. Re-diluting the sample extract in question.

10.7.4.7.2. If unsatisfactory results are obtained, then all samples shall be re-diluted from the original extracts and analyzed again.

10.7.4.8. The auto-diluter must be rinsed at least 5 times when diluting sample extracts known or suspected of containing high compound concentrations. Rinsing 3 times is satisfactory for most routine samples.

10.7.5. Labeling Chromatograms and/or Data Packet.

10.7.5.1. The data packet should contain the following information: Logbook number and page, project name, who calculated the data, who checked the calculation, and when and who recorded the data and QC.

10.7.5.2. Individual chromatograms should contain information to identify the sample analyzed, volume and dilutions, and calculations used.

10.7.5.3. Do not erase or use "Liquid Paper" to correct any errors. Put one line through the error with initials and date.

10.7.6. Retention Time (RT) Windows

10.7.6.1. Retention time window size

10.7.6.1.1. Make a minimum of 1 injection of all single component mixtures, multi-response pesticides, and PCBs at 24-hour intervals throughout the course of a 72-hour (3 days) period. However, 1 injection at 24-hour intervals throughout the course of a 120-hour (5 day) period is preferred.

10.7.6.1.2. Calculate the standard deviation of three (preferably five) absolute retention times for each single component pesticide. For multi-response pesticides/PCBs, choose one major peak from the group of peaks and calculate the standard deviation of the retention time of that peak.

10.7.6.1.3. Three times the standard deviation of the retention time for each pesticide/PCB will be used to establish the retention time window or, (+/-) 0.03 min for capillary columns; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms. For multi-response pesticides/PCBs, the analyst should utilize the retention time window but should primarily rely on pattern recognition. If the standard deviation of any compound is zero, use the standard deviation of any compound near the same retention time.

10.7.6.1.4. The laboratory must calculate retention time windows for each pesticide/PCB on each GC column used at the beginning of any new GC instrument setup or whenever a new GC column is installed.

10.7.6.2. Daily retention time windows

10.7.6.2.1. Inject all individual standard mixes and all multi-response pesticides/PCBs. To establish the RT window for the pesticides/PCBs of interest, use the absolute RT from the above chromatograms as the midpoint, and (+/-) three times the standard deviation calculated in Section 10.7.5.1.3. as the range or (+/-) 0.03 min for capillary columns.

10.7.6.2.2. Intersperse a standard mixture after every 20 samples but no less than every 12 hours as a minimum to verify that standard retention times are falling within the windows. Any pesticide outside of its established time window requires immediate investigation and correction before continuing the analysis. New absolute retention time windows must be established, unless instrument maintenance corrects the problem. Then re-inject all samples following the last standard meeting the criteria. If no target compounds are present in the samples, and the surrogate recovery is within limits, no re-injection is necessary and MQLS may be calculated.

10.7.7. Calibration

10.7.7.1. The gas chromatographic system should be calibrated using the external standard technique for all columns used for quantitation and after a new column is installed.

10.7.7.1.1. Prepare calibration standards at a minimum of three concentration levels (preferably five) for each compound of interest. One level of the external standards should be at a concentration near, but above, the MDL and the other concentrations should define the working range of the detector. This should be done on each quantitation column, each new instrument, and whenever the new calibration verification standard falls outside of accepted criteria. See SW-846, 8000 methods. See Table 10-2.

10.7.7.1.2. Using injections of 1 to 5 uL of each calibration standard, tabulate peak height or area responses against amount injected. The results can be used to prepare a calibration curve for each compound.

10.7.7.1.3. If the run is for confirmation (no quantitation) or for MQLs, the linearity check is not required. For MQLs, however, a standard at the MQL level of QC-1 is required.

10.7.7.1.4. The %RSD is calculated on representative compounds of interest (e.g., Lindane, Endrin, p,p'-DDT, and Methoxychlor). If the %RSD is (</=) 20 %RSD for the representative compounds, all compounds are assumed to be linear. When any compound is greater than 20% RSD, take the average %RSD of all target compounds. If the average %RSD is less than or equal to 20% RSD, the instrument is passes the linearity criteria. See SW-846, 8000 methods. Calculate the %RSD for the representative compounds as follows: Determine the response factor for each concentration by dividing the area or peak height by the amount injected. Calculate the standard deviations of the 5 response factors using:

$$s = (+/-) \sqrt{\frac{nE\,X^2 - (E\,X)^2}{n(n-1)}}$$

and then %RSD:

$$\%\text{Relative Standard Deviation} = \frac{\text{Standard Deviation} \times 100}{\text{Mean}}$$

%RSD may also be calculated using the S factor table.

10.7.7.1.5. If the linearity criteria is exceeded see Section 10.7.9 for suggested maintenance.

10.7.7.1.6. The %RSD may be calculated using 3, 4, or 5 concentration levels. However, any peaks quantitated must fall within the selected concentration range.

10.7.7.1.7. The calculation for %RSD for the representative compounds must be included in the chromatogram package.

10.7.8. GC Analytical Performance Criteria

10.7.8.1. As a guideline adjust the carrier flow rate or head pressure and oven temperature so that the standards will be eluted within 30 minutes on capillary columns.

10.7.8.2. Inject a GC/EC column performance mix consisting of:

	ng/uL
lindane	0.010
aldrin	0.010
endrin	0.025
p,p'-DDT	0.030

at the beginning of each run and after each set of 20 samples but no less than every 12 hours. Calculate the percent breakdown (BD) as follows:

$$\text{Percent BD for 4,4'-DDT} = \frac{\text{Total DDT degradation peak area (DDE + DDD) x 100}}{\text{Total DDT peak area (DDT + DDE + DDD)}}$$

Percent BD for Endrin =

$$\frac{\text{Total Endrin degradation peak areas (E. Ald. + E. Ketone) x 100}}{\text{Total Endrin Peak Area (Endrin + E. Aldehyde + E. Ketone)}}$$

See suggested maintenance in Section 10.7.8. if degradation exceeds 20%.

10.7.8.3. All calculations for percent breakdown must be part of the data package.

10.7.8.4. For calibration verification target analytes required in the project plan must be injected at the beginning of each 12 hour period with the following exception for the Aroclors. For sites that require PCB analysis include only the Aroclors that are expected to be found at the site. If PCBs are required but it is unknown which Aroclors may be present, the mid-concentration Aroclors 1242/1260 mixture only need be injected. However, if specific Aroclors are found at the site during the initial screening, it is required that the samples containing Aroclors be reinjected with the proper mid-concentration Aroclor standards. See SW-846, 8000 methods.

10.7.8.5. Intersperse a mid-point calibration standard after every 20 samples but no less than every 12 hours as a minimum. It is recommended that a calibration standard be included after every 10 samples for highly contaminated samples to minimize the number of repeat injections. The calibration factor of a specific standard compound shall not exceed a 20% difference from the initial response when screening samples or more than (+/-) 15% for any standard used for quantitating. When one or more of the compounds are greater than +/- 15%, take the average % of all compounds. If the average % is less than +/- 15%, the calibration verification is considered acceptable. See SW-846, 8000 methods.

$$\text{Calibration Factor} = \frac{\text{Total Response of Peak*}}{\text{Amount injected (in nanograms)}}$$

*For multi-response pesticides/PCBs use the response of the major peaks used for quantitation.

$$\text{Percent Difference} = \frac{R1-R2}{R1} \times 100$$

Where R1 = Calibration Factor from first analysis and
R2 = Calibration Factor from succeeding analysis.

10.7.8.5.1. All calculations for percent difference must be included in the data package.

10.7.8.6. Check retention time windows by analyzing a calibration standard after every 20 samples but no less than every 12 hours and compare it to the standard at the beginning of the 12 hour shift. If retention time is outside of calibrated window (see 10.7.6.2.) (+/- .03min or 3 standard deviations) check the GC for problem (i.e., septum and/or column leaks, bad syringe, etc.)

10.7.8.7. Check for peak tailing and take corrective action if necessary.

10.7.8.8. The %RSD may be calculated using 3 to 5 concentration levels. However, any peaks quantitated must fall within the linear range and the required minimum quantitation limits must be met. The calculation for % RSD for the representative compounds must be included in the chromatogram package.

10.7.9. Suggested Maintenance

10.7.9.1. Corrective measures may require any one or more of the following remedial actions:

10.7.9.1.1. Capillary columns-Turn off both oven and injection ports. Clean and deactivate the glass injector port insert or replace with a cleaned and deactivated insert. Remove the analytical column when the oven has cooled. Break off the first few inches of the column (up to one foot) on the injector port side and then reconnect the column. If these procedures fail to eliminate the degradation problem, it may be necessary to deactivate the metal injector body and/or replace the column.

10.7.9.1.2. Metal Injector Port-Turn off the oven and injection port heaters and remove the analytical column when the oven and the injection port heaters have cooled. Remove the glass injection port insert (in instruments with off-column injection). Inspect the injection port and removed any noticeable foreign material.

10.7.9.1.2.1. Place a beaker beneath the injector port inside the GC oven. Using a wash bottle, serially rinse the entire inside of the injector port with acetone, toluene and then iso-octane, catching the rinsate in the beaker.

10.7.9.1.2.2. Use a solution of deactivating agent (Sylon-CT or its equivalent) following manufacturer’s directions. After all metal surfaces inside the injector body have been thoroughly coated with the deactivation solution, serially rinse the injector body with toluene, methanol, acetone, and hexane. Reassemble the injector and reconnect the column.

10.7.10. Qualitative Analysis

10.7.10.1. Identification of compounds by retention times must be performed by experienced gas chromatographers because slight shifts in retention times require judgment decisions. Observe retention time shifts of standards throughout a day's run to evaluate retention time shifts in samples. Utilize the daily retention time windows for compound identification.

10.7.10.2. Confirm all compounds (pesticides/PCBs) on a second different column, or different detector (other than FID), unless the compound has been confirmed by GC/MS.

10.7.10.3. It is suggested that at least one sample from a set be confirmed by GC/MS, if concentration permits. It is the responsibility of the GC analyst to report any pesticides/PCBs confirmed by GC/MS. This must be properly noted on the data sheet. Confirmation by GC/MS is shown by adding the letter C to the amount of the compound being reported. Alpha-BHC, gamma-BHC, Endosulfan I and II, and Endrin must be confirmed on the pesticide extract from water rather than the BNA extract (these compounds are unstable at the basic pH).

10.7.10.4. Reporting Chlordane-Weathering and/or different fomulations of chlordane may modify the technical chlordane pattern. If the chlordane pattern in a sample is similar to technical chlordane, use a technical chlordane standard for quantitation. ("similar" means all constituents are present, including heptachlor, in about the same ratio as a standard of technical chlordane.) If the pattern is different but gamma and alpha chlordane and other chlordane constituents are present, use the individual chlordane constituent standards for calculation. Report the individual constituents on the data reporting sheet. Report a total of all constituents listed on the data sheet, except heptachlor, when the total is requested. Heptachlor is reported separately in these situations.

10.7.11. Calculation and Project Wrap-up (Also see Section 10.11 on Data Reporting)

10.7.11.1. For calculation of components in a sample two options are available:

10.7.11.1.1. Use a one-point mid-level red mix standard either manually or by acceptable computer program; or,

10.7.11.1.2. Use a three (preferably five)-point linear curve by acceptable computer program.

10.7.11.1.3. For samples with no analytes found use the MQL guidelines for different matrices. See Form 10-16.

10.7.11.2. To simplify the checking of calculations, everyone may use the formulas for calculating concentrations:

For response factor (K):

$$\frac{\text{uL injected}}{\text{(Volume extract in uL) (dilution)}} \times \text{(mL, mg or gm extracted)} = K$$

For amount in sample:

$$\frac{\text{Pk ht or area of sample}}{\text{Pk ht or area of std.}} \times \frac{\text{(ul inj)(conc.of std.,ng/ul)}}{K} = \text{Concentration}$$

10.7.11.3. Calculation of off-scale peaks using peak height or area is allowable if it has been shown that response is linear in the concentration range of the off-scale peak and no interfering or rising baseline exists.

10.7.11.4. All calculations must be checked by someone other than the person who performed the original calculation. The chromatogram with the appropriate standards and QC showing the calculations for the reported data should be given to the checker. A hardcopy of the chromatogram should be put in the project file.

10.7.11.5. The checker should check for accuracy of the transcription of data to the data report sheets.

10.7.11.6. Diluted samples and all standards should be discarded at the completion of each project.

10.7.11.7. All vials that are ready for disposal should be placed in a waste safety can, keeping vials with PCBs in a separate waste safety can. These vials must be treated as hazardous waste and disposed of accordingly. (See Section 4.6.)

10.7.11.8. All original sample vials should be stored in vial storage boxes in a refrigerator and placed in a secure area for permanent storage after completion of analysis. See Section 10.9. for instructions on vial storage.

10.7.11.9. The project chemist is responsible for calculating surrogate and matrix spike recoveries and recording the results on the appropriate data sheets and/or transmitting all results to the proper computer data system. Unusual results on QC data should be reported to a pesticides' senior staff specialist. See Forms 10-11, 10-12, 10-13, and 10-14.

10.7.11.10. Samples having greater than or equal to 50 ppm PCBs should be reported to the extraction lab Senior staff specialist.

10.8. GC/MS ANALYSIS

10.8.1. GC Screen and GC/MS Logbook

10.8.1.1. Record all pertinent information requested on the logbook sheet. The electronic version of these forms are available from the forms custodian. The designated person at this time is Sallie Hale. See Forms 10-4 and 10-5.

10.8.1.2. Record file name under sample number column as it exists on the disk.

10.8.2. GC Screen

10.8.2.1. Volatile Organics

10.8.2.1.1. All samples may be screened by GC/PID/ELCD to determine the approximate concentration level prior to GC/MS analysis. Dilutions for GC/MS analysis are to be determined from this screen analysis.

10.8.2.2. Semivolatile Organics

10.8.2.2.1. All samples may be screened by GC/FID, GC/ELCD, GC/PID, or any combination of these necessary to determine the approximate concentration level prior to GC/MS analysis. Dilutions for GC/MS analysis are to be determined from this screen analysis.

10.8.3. File Name Labeling

10.8.3.1. Use the following format for file names for volatile blanks and standards.

10.8.3.1.1. S0128R1 - R1 (or R2, R3, ect.) represents the standard run number, B for blanks, followed by date of analysis.

10.8.3.2. Use the following format for file names for semivolatile blanks.

10.8.3.2.1. B00736SLW - B for blank, followed by ASB log number for first sample in the set that blank applies to, followed by appropriate analysis designations.

10.8.3.3. Use the following format for file names for semivolatile standards.

10.8.3.3.1. S01997SLW - Surrogate standard. S for standard, followed by ASB log number for first sample in the set that standard applies to, followed by appropriate analysis designations, followed by the day of the month if the instrument allows that length for file names.

10.8.3.3.2. S093020 - First four digits date designations, followed by concentration level in ng/ul. Surrogate compounds are normally included in the daily standard.

10.8.3.4. Sample file name. Use the ASB log number followed by the proper analytical descriptor if the instrument allows that length for file names (ie 42361SLW). See Table 10-1.

10.8.3.5. Current instrument designations are:

50 - INCOS 50 - EPA
52 - INCOS 50 - EPA
53 - INCOS 500 - EPA
71 - HP5971 - EPA - VOA
72 - HP5972 - EPA - VOA
73S - HP5973 - ESAT - BNA
73B - HP5973 - ESAT - VOA
73A - HP5973 - EPA - BNA

10.8.3.6. Add the following designations between the SESD number and the analytical descriptor (ie. 40849XDSLS):

10.8.3.6.1. X and Y - for duplicates.

10.8.3.6.2. D - Dilution (Indicate D2, D3, etc. for subsequent dilutions)

10.8.3.6.3. R, RS, R3, etc. - Designates a re-extraction of a sample or reinjection or a purging of a replicate VOA sample.

10.8.3.6.4. If other designations are needed, record their meaning in logbook.

10.8.3.7. NOTE: Some software may limit the file name to eight characters.

10.8.4. Title Information as Follows:

10.8.4.1. File name.

10.8.4.2. Instrument designation.

10.8.4.3. Sample volume information (including dilution information).

10.8.4.4. GC Column type and conditions as 50-210 X 8, I2 Fl2 where 50-210 are initial and final temperatures, X8 is program rate, I2 is initial hold time, Fl2 is final hold time.

10.8.5. Mass Scale Calibration Using FC43

10.8.5.1. Tune instrument using the following guidance:

10.8.5.1.1. Admit FC43 with carrier flow entering source as appropriate for the individual instrument.

10.8.5.1.2. Adjust resolution to achieve the desired parameters.

10.8.5.1.3. Make appropriate tuning adjustments to achieve the following ion intensity ratios as nearly as possible.

Mass 2l9 15-40% of Mass 69

Mass 220 $\geq$ Mass of 70

Mass 4l4 50-l25% of Mass 220 (for semi-volatiles)

Mass l3l $\pm$ 80-120%of Mass 2l9

10.8.5.2. Acquire at least 5 scans of FC43 data scanning a mass range of 20-650 amu (or as appropriate).

10.8.5.3. Run calibration routine.

10.8.5.4. Instrument should calibrate from at least 28 - 502 amu.

10.8.6. Zero the Instrument

10.8.6.1. Set instrument zero consistent with manufacturer's specifications and/or to a proven, reliable setting (if this is necessary).

10.8.7. Instrument Tuning Performance Test

10.8.7.1. A tune performance check must be performed every 12 hours during analysis.

10.8.7.2. Analyze 50 ng of Decafluorotriphenylphosphine (DFTPP) for extractables or 50 ng of p-Bromofluorobenzene (BFB) for volatiles.

10.8.7.3. Other concentrations or compounds may be used as required by the analytical protocols.

10.8.7.4. Operating Conditions

10.8.7.4.0.1. Mass Spectrometer parameters same as analysis planned for the twelve hour shift.

10.8.7.4.0.2. The reference compound should elute so that compounds of interest are resolved.

10.8.7.4.1. The mass spectrum must be acquired in the following manner: Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan within 10 scans prior to the beginning of elution of the performance compound.

10.8.7.4.2. Compare the ion intensity ratio of those of published criteria.

10.8.7.4.3. If the required criteria are not met, the instrument must be retuned until the spectra meets the specified criteria.

10.8.7.4.4. Check retention time and peak shape of reference compound to determine if they are consistent with prior results.

10.8.7.4.5. Check the peak intensity (by peak height or area) to determine if the sensitivity is adequate.

10.8.7.4.6. Print a list of masses and intensities, a copy of the chromatogram with areas of each peak printed, and maintain in a folder.

10.8.8. GC/MS Linearity Check

10.8.8.1. Initial Calibration

10.8.8.1.1. The GC/MS system must be initially calibrated with all compounds of interest at a minimum of three concentrations (5 levels are recommended). Using the response factors (RF) from the initial calibration, calculate the percent relative standard deviations (% RSD) for all compounds.

10.8.8.1.1.1. A system performance check must be met for all compounds. A minimum response factor of 0.100 for the volatile compounds and 0.05 for the semivolatile compounds is required. If this criteria is not met, corrective action must be taken.

10.8.8.1.1.2. The % RSD for each compound must be less than 15 percent. If this criteria is not met, corrective action must be taken. This might require instrument maintenance, new standards preparation, and/or repeating the analysis of the curve. If after corrective action some compounds exceed 15 percent, the analyst may proceed, but any positive results for these compounds must be reported with a J flag.

10.8.8.1.1.2.1. Thirty percent RSD is acceptable for the following semivolatile compounds: 4-nitrophenol, 4-chloro-3-methylphenol, 2,4-dinitrophenol, 2-methyl,4,6-dinitrophenol, pentachlorophenol, 3,3-dichlorobenzidine, 2-nitroaniline, 3-nitroaniline, 4-nitroaniline. Thirty percent RSD is acceptable for

the following purgeable compounds: vinylchloride, 1,1-dichlorothene, chloroform, 1,2-dichloropropane,

toluene, and ethylbenzene. If these analytes are of specific importance to the project, corrective action may be necessary. Otherwise, these compounds must be flagged as estimated (J) if the %RSD is greater than 15%.

10.8.8.1.1.2.2. The RF for each compound in each concentration level of the curve must be compared to the average RF of the curve to determine if any individual point on the curve is an outlier. Calculate the percent difference between the average response factor from the curve and the response factor from the individual concentration level in the curve. If the percent difference for any compound is greater than 25%, corrective action may be necessary. This usually means re-analyzing the bad point on the curve.

10.8.8.2. Daily Calibration Check

10.8.8.2.1. A standard mixture containing all volatile or semivolatile compounds of interest must be analyzed every l2 hours of operation.

10.8.8.2.1.1. A system performance check must be met for all compounds. A minimum response factor of 0.100 for the volatile compounds and 0.05 for the semivolatile compounds is required. If this criteria is not met, corrective action must be taken.

10.8.8.2.1.2. A calibration check of the initial calibration curve is made for each target compound. Calculate the percent difference between the average response factor from the initial calibration and the response factor from the current standard. If the percent difference for any compound is greater than 25%, corrective action may be necessary. The analyst must immediately judge the impact on the data generated for that day. Any compounds with %D greater than 25% should be flagged as estimated (J). If more than 25% of the compounds are greater than 25%D, corrective action must be taken. This may require generation of a new curve.

$$\%\ \text{Difference (\%D)} = \frac{\overline{RF}_I - RFc}{\overline{RF}_I} \times 100$$

$\overline{RF}_I$ - Average response factor for initial curve

RFc - Response factor from current standard mixture

10.8.8.2.1.3. Continuing without corrective action may be prudent if the outlier compounds are not of interest to the project. The Senior Staff Specialist or Organic Section Coach must be consulted before continuing without corrective action. In this case, the corrective action may be to report these compounds as not analyzed or with an estimated flag.

10.8.8.2.1.4. A file of the results from the initial and continuing calibration checks must be maintained. Continuing calibration files are part of the daily standard chromatograms and are to be filed with the appropriate project.

10.8.9. Analyze Standard Mixture

10.8.9.1. Analyze standard mixtures and performance compounds at least every 12 hours (purgeable standards should be at room temperature before analysis).

10.8.9.2. Use GC conditions and MS parameters consistent with sensitivity requirements and equal to those planned for the shift's operations.

10.8.9.3. Incorporate internal standards where feasible.

10.8.9.4. Perform system performance check and daily calibration check.

10.8.9.5. Record area count of the quantitation ion for at least one of the internal standards.

10.8.9.6. The surrogate standard is normally part of the BNA standard.

10.8.10. Analyze Laboratory Blank

10.8.10.1. Utilize internal standards where feasible.

10.8.10.2. Record integrations for the same internal standards recorded in standard.

10.8.10.2.1. If the area count is not within - 50% to + 100% of those in Standard Mixture, rerun.

10.8.10.2.2. Internal standard retention times must be within ± 10 scans or 10 seconds of standard, whichever is greater.

10.8.10.3. Check for carryover from standard injection.

10.8.10.4. Compute surrogate recovery.

10.8.10.5. Section 10.11.3 gives further guidance on use of blanks.

10.8.11. Analyze Samples

10.8.11.1. If area count of internal standard is not within - 50% to +100% of the standard, rerun.

10.8.11.2. Internal standard retention times must be within ± 10 scans or 30 seconds of standard, whichever is greater.

10.8.11.3. Disperse field or lab blanks throughout the day as necessary.

10.8.11.4. Disperse standard mixtures between at least every l2 hours of analysis.

10.8.11.5. Utilize internal standard where feasible.

10.8.11.6. Compute surrogate recovery and record in GC/MS Log.

10.8.12. Analyze at least one check sample monthly.

10.8.13. Drinking water samples with positive results should be verified by analyzing a replicate sample whenever possible. The Senior Staff Specialist or Organic Section Chief should be contacted if deviations from this policy are necessary.

10.8.14. TCLP analysis: The GC/MS data generated for VOA, BNA, and Pesticide analysis is reviewed with the Extraction Laboratory Chemist and a decision made whether any samples could fail the TCLP test. If it potentially could fail, then the TCLP test is performed and the results reported. If the sample cannot fail the test, this information is reported.

10.8.15. Data Processing

10.8.15.1. Plot total ion current profiles.

10.8.15.2. Using the peak finding algorithm and the total ion current profile, place scan numbers in scan list. The parameters should be set to find all peaks at approximately 10% of instrument MQL (This is usually set to 10% of the area of an internal standard response.)

10.8.15.3. Print a copy of spectra and library search (best 3 match graph, ranked on purity). Use both NIST and Wiley library in search if available.

10.8.15.3.1. If peaks are asymmetrical, print a spectra with the background manually subtracted.

10.8.15.4. Compare the spectra of the unknown with the 3 best matches and see if one is a logical match.

10.8.15.5. Check for presence of molecular ion and isotopic clusters.

10.8.15.6. Check the data printed with the best entries from the library search as an aid to the visual comparison of an unknown spectra to the library spectra.

10.8.15.7. If no reasonable match, check other published data bases, as needed.

10.8.16. Qualitative Analysis

10.8.16.1. Target compounds shall be identified by comparison of the sample mass spectrum to the mass spectrum of a standard of a reference spectra of suspected compound. Two criteria must be satisfied to verify the identifications: (l) elution of the sample component at the same GC relative retention time as the standard component, and (2) correspondence of the sample component and standard component mass spectra.

10.8.16.1.1. For establishing correspondence of the GC relative retention time (RRT), the sample component RRT must compare within ± 0.06 RRT units of the RRT of the standard component. For reference, the standard must be run within l2 hours of the sample. The RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

10.8.16.1.2. The requirements for qualitative verification by comparison of mass spectra are as follows:

10.8.16.1.2.1. All ions present in the standards mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

10.8.16.1.2.2. The relative intensities of ions specified above must agree within plus or minus 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample ion abundance must be between 30 and 70 percent.)

10.8.16.1.2.3. Ions greater than 10% intensity in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. Do not report any compounds with a calculated value below 0.1 of the MQL.

10.8.16.2. A library search shall be executed for Non-Target sample components for tentative identification. The most recent available version of the NIST and Wiley Mass Spectral Libraries should be used.

10.8.16.2.1. Do not report any compounds with a calculated value below the MQL. Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

10.8.16.2.2. Guidelines for making tentative identification:

10.8.16.2.2.1. Relative intensities of major ions of the reference spectrum (ions greater than 10% intensity of the most abundant ion) should be present in the sample spectrum.

10.8.16.2.2.2. The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample ion abundance must be between 30 and 70 percent.

10.8.16.2.2.3. Molecular ions present in reference spectrum should be present in sample spectrum.

10.8.16.2.2.4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

10.8.16.2.2.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination of coeluting compounds. Data system library reduction programs can sometimes create these discrepancies.

10.8.16.2.2.6. If in the opinion of the mass spectral specialist, no valid tentative identification can be made, the compound should be reported as unidentified compound. The mass spectral specialist may give additional classification of the unknown compound, if possible (i.e. unknown phthalate, unknown hydrocarbon, unknown acid type, unknown chlorinated compound).

10.8.16.2.2.7. Non-Target compounds identified in samples will be reported using the NIST and Wiley Libraries name of the best probable match. The best probable match is selected by the mass spectroscopist from the best matches as chosen by the library search routine ranked by purity. The analysts interpretation may supersede the computer matching algorithm.

10.8.16.2.2.8. The NIST or Wiley Library nomenclature should be stripped of numbers or letters that would make the reported compound a specific isomer (e.g. 1,2-dibromoethane should be reported as dibromoethane).

10.8.16.2.2.9. Where more than one isomer of a compound is identified, they should be reported under one name. The total concentration should be reported with this one name and the number of isomers should be reported in parenthesis. The isomer name chosen for one sample of a project should be used in all samples for the project, where no distinguishable spectral differences are present (e.g. If the best match for C_3alkyl benzenes is methyl ethyl benzene instead of trimethyl benzene, or propyl benzene, report as methyl ethyl benzene in all samples of the project where this is true).

10.8.16.2.2.10. Name alkyl substituted analogs of Target compound isomers using the earlier eluting of the isomers(e.g. methylfluoranthene, not methylpyrene).

10.8.17. Quantitation

10.8.17.1. Target components identified shall be quantified by the internal standard method. The internal standard used shall be the one nearest the retention time to that of a given analyte. The EICP area of characteristic ions of analytes are used. The response factor (RF) from the daily standard analysis is used to calculate the concentration in the sample. Secondary ions may be used if interferences are present. The area of a secondary ion cannot be substituted for the area of a primary ion unless a response factor is calculated using the secondary ion.

10.8.17.1.1. Any compound that had a %RSD in RF of greater than 30 in the initial calibration curve must be reported with an estimated value flag (J). Similarly, any compound that had a % difference in RF of greater than 25 between the RF from daily standard mixture and the average RF from the initial curve must be reported with an estimated value flag (J).

10.8.17.2. An estimated concentration for Non-Target components tentatively identified shall be quantified by comparison to an internal standard free of interferences. The following order of preference for internal standards to use as a reference for extractables is D_{10}Phenanthrene, D_8Naphthalene, D_{10}Acenaphthylene, D_{12}Chrysene, D_{12}Perylene, and D_4 Dichlorobenzene. The internal standard nearest in retention time to the Non-Target compound may be used to estimate concentration. Total area counts or peak

heights from the total ion chromatograms are to be used for both the compound to be measured and the internal standard. A RF of one

(l) is to be assumed. The value from this quantitation shall be qualified as estimated. This estimated concentration should be calculated for all tentatively identified compounds as well as those identified as unknowns.

10.8.18. GC/MS Data Transfer

10.8.18.1. Initial Reports - After the data is processed by the GC/MS data system, it is transferred to another computer. The data is then adjusted taking into account sample dilution, amount purged or extracted, and dry weight, when applicable. Hard-copies are then produced. After corrections or additions are made to the data based on further analysis of the chromatograms and mass spectra, the final data product is transferred to the R4LIMS computer.

10.8.18.2. Final Reports

10.8.18.2.1. When no more alterations are necessary, the final data can be transferred to the R4LIMS system.

10.8.18.2.2. The data is then printed out in final production format and proofed for errors. Any corrections are made and the corrected data sheet is printed. A memo is also printed, the appropriate number of copies (include one file copy) are made and the report is signed by the project chemist and given to the Organic Section Chief for review.

10.8.19. Archiving Data

10.8.19.1. All samples and standards must be archived by copying to nine-track mag tapes using the EPA program or using other electronic storage devices.

10.8.20. General Responsibilities

10.8.20.1. The GC/MS chemist is responsible for verifying that all sample extract vials were received from the extraction lab or GC analyst. The chemist is then responsible for the vials until GC/MS analysis is complete, and the vials have been stored or have been discarded. The extract vials should be stored in the refrigerator designated for semivolatile extracts when not in use.

10.8.20.2. Recap all vials that are to be retained as soon as possible after puncturing the septum. Remark the volume on the vial label after injection or dilution.

10.8.20.3. Diluted samples and standards should be discarded immediately following injection to avoid unnecessarily cluttering up the lab and extract boards.

10.8.20.4. All vials that are ready for disposal should be placed in the "Oily Waste Safety Can." Vials must be disposed of according to the procedures outlined in Section 4.6. Dispose of standard and sample vials containing PCB's and other listed compounds in a separate waste container.

10.8.20.5. All original sample vials are placed in boxes and are to be stored in a locked custody room upon completion of analysis. See Section 10.9 for instructions on vial storage.

10.8.20.6. The project chemist is responsible for calculating surrogate and matrix spike recoveries and recording the results on the appropriate computer data sheet. Unusual results on QC data should be reported to the Senior Staff Specialist and/or the Organic Section Chief.

10.8.20.7. GC/MS Files - Chromatograms should be filed numerically according to sample numbers. Files should be labeled with the series of sample numbers on first line. Project name(s) should be listed under this. Chromatograms and each file should be arranged as follows:

10.8.20.7.1. Extraction sheets and data sheets.

10.8.20.7.2. Standards analyzed in order of date run.

10.8.20.7.3. Blanks analyzed in order of date run or sample # of blank series.

10.8.20.7.4. Samples analyzed in numerical order.

10.8.20.7.5. Pertinent GC screening chromatograms.

10.8.20.8. Any pesticides/PCBs confirmed by GC/MS must be reported to the Pesticides Senior Staff Specialist to be noted on the pesticide/PCB data sheet. Chromatograms from Pesticide and PCB confirmation are sent to the GC unit to file with their chromatograms.

10.8.20.9. Keep GC screen chromatograms for samples that did not require GC/MS analysis. All other GC screen chromatograms should be discarded.

10.9. Extract Storage

10.9.1. Sample extracts are to be stored in storage containers after final reporting of data. These containers will be kept in their respective areas of the GC Lab and the GC/MS Lab in a refrigerator, based on the type of sample. As soon as possible, the containers should be disposed of. Some criminal and other samples may need to be stored for extended periods of time. The sample storage custodian will furnish information on disposition of samples in a timely manner. Record sample extracts placed in storage containers on Form 10-17.

10.10. Preparation, Storage, and Use of Organic Analytical Standards

10.10.1. Standard Sources

10.10.1.1. Primary Standards: Commercial sources are available, request the purest grade available.

10.10.1.1.1. Prepared standards: Commercial sources.

10.10.1.1.2. QC Standards: second source other than primary standards.

10.10.1.1.3. Prepared standards: commercial. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source. If the purity of these standards is questionable, report the data based on these standards as estimated.

10.10.2. Glassware, Equipment, and Solvents:

10.10.2.1. Analytical balance, capable of an accuracy of $\pm$ 0.l mg.

10.10.2.2. Spatula, stainless steel.

10.10.2.3. Transfer class "A" pipets and Pasteur disposable pipets or suitable syringes.

10.10.2.4. Flasks, volumetric, 25, 50, 100 and 200 mL.

10.10.2.5. Bottles, Teflon-lined caps 60 mL.

10.10.2.6. Small glass funnels, and bent paper clip.

10.10.2.7. Refrigerator, explosion-proof.

10.10.2.8. Pesticide grade solvents: ethyl acetate, toluene, acetone, isooctane, hexane, methanol, and carbon disulfide.

10.10.3. Safety Precautions and Operating Procedures

10.10.3.1. Gloves should be used when handling reference materials.

10.10.3.2. Standards used for quantitating samples are to be made by a chemist.

10.10.3.3. Hoods should be used when weighing toxic standards or diluting with organic solvents.

10.10.3.4. Rinse all glassware prior to use with methanol, acetone, and isooctane and let air dry in hood.

10.10.3.5. Always perform a balance check with Class-S weights each day the balance is used. Record the balance check on the Standard Sheet. Check calculations on solutions to be made up.

10.10.3.6. Do not store any standards in volumetric glassware. Transfer to a 60-mL screwcap bottle with Teflon^{R} liner if the solution is to be stored. Use phosphate tubes or vials, with Teflon^{R} liners, for short term storage. All standards must be properly labeled.

10.10.3.7. Always rinse used glassware with acetone before washing in dishwasher with other glassware. Rinse pipets out with acetone immediately after use.

10.10.3.8. Keep all standards in refrigerator or freezer when not in use.

10.10.3.9. Always let standards and solutions come to room temperature before opening.

10.10.3.10. Check new working standard against old standard. Old standard may be slightly more concentrated due to evaporation of solvent from repeated openings.

10.10.3.11. Transfer waste standards to a waste bottle. Rinse the empty bottle several times with acetone. Add the rinsate to the waste bottle and discard the standard bottle.

10.10.3.12. Provide a large waste beaker located in a hood for rinsing all used glassware and pipets before washing with soap and water. Transfer the wash solvent to a waste bottle.

10.10.3.13. Volumetric flasks and storage bottles used for standards must be rinsed several times with distilled water to remove any alkaline residue. Alkaline residues cause degradation of certain organics and pesticides.

10.10.4. Standards:

10.10.4.1. Replace stock standards and “non-working” standards every year.

10.10.4.1.1. Suggested procedures for preparation of stock standards follow. We also suggest procedures found in SW-846, 8000 methods.

10.10.4.1.1.1. Weigh 50.0 mg of primary standard into a 50 mL beaker using a small spatula for solids or a disposable pasteur pipet for liquids. It may be necessary to aid dissolution by adding as small amount of solvent (e. g. ethyl acetate or toluene).

10.10.4.1.1.2. Some standards may require placing the beaker in an ultrasonic cleaning device or on a steam bath for complete dissolution.

10.10.4.1.1.3. Transfer through a glass funnel into a 50 mL volumetric flask, washing with an appropriate solvent. Dilute to volume with the least volatile of the appropriate solvents and mix. Calculate the concentration in micrograms per microliter. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. If the purity is less than 96%, the weight must be adjusted for purity.

10.10.4.1.1.4. Transfer to 60 mL screw capped bottles with orange labels. Old bottles that contain the same standard may be reused if rinsed with isooctane.

10.10.4.1.2. Calibration Standards - This consists of a set of five standards with concentrations covering the linearity range for each detector used for quantitative analysis.

10.10.4.1.3. Intermediate and working standards, (blue, yellow, silver, and red) are diluted with a high boiling solvent such as isooctane. Working standards are diluted to give even numbered concentrations if possible, from intermediate and working standards (i.e. 10 ng/uL vs 11 ng/uL). Discard working standards after six months. Some standards are unstable and must be made up more frequently. Working solutions should be checked at least quarterly against available Cincinnati QC samples or a check standard. New working solutions should be checked against the old standard. Percent difference must not exceed 10% for each compound checked.

$$\text{Percent Difference} = \frac{R_1 - R_2}{R_2} \times 100$$

$$R = \frac{\text{Total area of peak*}}{\text{Amount injected (in nanograms)}}$$

10.10.4.1.4. For multicomponent pesticides/PCB's, use the total area of all peaks used for quantitation.

R_1 = relative response from working standard

R_2 = relative response from (second vendor) QC standard

Mixes Used	Detector	Concentrations (ng/uL)
Blue	FID/MS	5 - 100
Yellow	Hall, N/P	0.2 - 10
Silver	Hall	0.05 - 2.5
Red	EC	0.005 - 0.25
Green	(Spike solutions for all parameters)	

10.10.4.1.4.1. All standards must be stored in refrigerator when not in use.

10.10.4.2. Spike Solution - All spike solutions are made from stock or intermediate solutions and diluted with acetone or methanol.

10.10.4.3. Volatile standard solutions - Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol using assayed liquids or gases as appropriate. Because of the toxicity of some of the organohalides, primary dilutions of these materials should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be used when the analyst handles high concentrations of such materials.

10.10.4.3.1. Place about 9.8 mL of methanol into a 10 mL ground glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 minutes or until all alcohol wetted surfaces have dried. Weigh the flask to the nearest 0.l mg.

10.10.4.3.2. Add the assayed reference materials as described below:

10.10.4.3.2.1. Liquids - Using a 100 uL syringe, immediately add 2 or more drops of assayed reference material to the flask, then re-weigh. The liquid must fall directly into the alcohol without contacting the neck of the flask.

10.10.4.3.2.2. Gases - Introduced from lecture bottle. Flow rate is controlled with a valve through a teflon tube to top of the meniscus.

10.10.4.3.3. Re-weigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in micrograms per microliter. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard.

10.10.4.3.4. Transfer the stock standard solution into a Teflon sealed screw-cap bottle. Store, with minimal head-space, at -10^{o}C to -20^{o}C and protect from light.

10.10.4.3.5. When stored under the above conditions, those standards should be replaced if comparison with QC check samples indicates a problem. Gases are replaced after 3 months and all others after 6 months.

10.10.4.3.6. Intermediate standards - Using stock standard solutions, prepare intermediate standards in methanol that contain the compounds of interest, either singly or mixed together. The intermediate standards should be prepared at concentrations such that the aqueous calibration standards will bracket the working range of analytical system. Intermediate standards should be diluted to give calibration standards of approximately 30 ng/uL.

10.10.4.3.7. Intermediate standards are prepared as needed and should be stored in a freezer with minimal headspace. They should be checked frequently for signs of degradation or evaporation, especially prior to preparation of calibration standards. Quality control check standards used to determine the accuracy of calibration standards may be obtained from commercial sources.

10.10.4.3.8. Internal/Surrogate standard spiking solution - The solution may be prepared as described above or with a dilution of a commercial standard. The intermediate surrogate solution is prepared as needed. The stock standard is good for a minimum of 6 months. The first compound is a good barometer of the solution; if it appears to be much smaller in size than the other two in a GC/MS run, the stock solution should be discarded and another one made.

10.10.5. Records

10.10.5.1. Stock Standards. Enter the weight of the primary standard and other requested information on the stock standard sheet in the Quality Control Standards Logbook (See Form 10-6). Also, record the requested information on the summary log sheet at the beginning of the section (See Form 10-7), and on the label of the standard bottle.

10.10.5.2. Each stock solution is assigned a discrete number that identifies that particular stock solution. Whenever a new stock solution of the same compound is made up, the original number should be retained.

10.10.5.3. Blue, Yellow, and Red Mixes. Record the parent color, dilution number and date of preparation on the standard sheet. See Form 10-15.

10.10.5.4. A new standard sheet must be prepared when one or more ingredients or the concentrations in a mixture changes. Retain the old mix bottle number on the new mix. Select a new number when a completely new mix or standard is made up. Retire the number when a mix or standard is no longer needed.

10.10.5.5. In GC and GC/MS Logbook, enter name of the standard plus the color code if applicable.

10.10.5.6. There are three Quality Control Standards Logbooks. Volume l is for stock solutions (orange label). Volume 2 is for intermediate, working, and spike standards. Volume 3 is for outdated sheets.

10.11. Data Reporting

10.11.1. General

10.11.1.1. No data will be reported until all QC data has been evaluated and data determined to be valid.

10.11.1.2. In certain situations where % moisture is not determined or used, the data should be reported on a wet weight basis.

10.11.1.3. Report parameter concentration in units as in Table 7-4.

10.11.1.4. Waste samples and % moisture - Calculate and report waste samples on a dry weight basis if they are primarily heavily contaminated soil or dry solid. Waste that is primarily nonaqueous liquid should be reported using the weight as received or wet weight basis.

10.11.1.5. Use the following designations on the data sheet:

10.11.1.5.1. U - The analyte was analyzed for but not detected. The value preceding the "U" is the "minimum quantitation limit (MQL)".

10.11.1.5.1.1. Minimum Quantitation Limit (MQL) -- Every sample has a concentration level below which the variance of the results for a particular analyte (element or compound) exceeds the acceptable quality control criteria. This level is the MQL and is reported as the value preceding the "U". The MQL is based on the lowest quantitative data point of the instrument calibration curve. The MQL is derived using this data point and other factors such as: sample size, dilution required, sample % moisture, and sample interferences. The value often varies from analyte to analyte within a sample. Analytes are often detected at levels below the MQL and are reported as estimated values (J). Generally, analytes identified below the MQL will only be reported if the concentration is greater than one tenth of the MQL.

10.11.1.5.2. J - The identification of the analyte is acceptable, but the quantitative value is an estimate. The value preceding the "J" is the "estimated value".

10.11.1.5.2.1. Estimated Value--Every sample analysis has quality control criteria associated with the quantitative data which have been established based on similar analyses. When these criteria are exceeded, the value for that analyte or similar analytes is reported as an estimated value. Examples are:

10.11.1.5.2.1.1. Calculated values are below or above an appropriate linear range

10.11.1.5.2.1.2. Calculated values are below the MQL of an analyte.

10.11.1.5.2.1.3. Analytical holding times for analysis are exceeded.

10.11.1.5.2.1.4. Surrogate recovery limits are exceeded.

10.11.1.5.2.1.5. There are no known quality control criteria for an analyte.

10.11.1.5.3. N - There is presumptive evidence that the analyte is present but it has not been confirmed. The analyte is "tentatively identified".

10.11.1.5.3.1. Tentative Identification--There is an indication that the analyte reported is present. The quality control requirements necessary for confirmation were not met. Examples are:

10.11.1.5.3.1.1. A specific list of compounds is analyzed for in every organic analysis by gas chromatography/mass spectrometry (GC/MS). Other compounds are often present and their spectra are compared to published mass spectral data. If a qualitative determination is made, the compound is reported as tentatively identified.

10.11.1.5.3.1.2. The presence of analytes is often indicated, but there is evidence of possible interferences. There is presumptive evidence that the analyte is present, therefore, it is reported as tentatively identified.

10.11.1.5.4. C - The analyte is determined to be present. The presence of the analyte was "confirmed by GC/MS".

10.11.1.5.4.1. Confirmed by GC/MS - Pesticides are routinely analyzed by gas chromatography with an electron capture detector (GC/EC). When identified by GC/EC analysis in sufficient concentrations, pesticides are confirmed on the mass spectrometer by comparing the spectra of the analyte with the spectra of a particular pesticide. If a good spectral match is obtained, the pesticide identification is considered to be confirmed. The concentration is quantitated by GC/EC.

10.11.1.5.5. A - The analyte was analyzed in replicate. The value preceding the "A" is an "average value" of the replicates.

10.11.1.5.5.1. Average Value--Samples are often analyzed in replicate (usually in duplicate). Aliquots of the same sample are analyzed and the values are averaged. Sometimes replicate samples are analyzed and the values are reported as an average.

10.11.1.5.6. K - The analyte is determined to be present. The actual value is known to be "less than" the value preceding the "K".

10.11.1.5.6.1. Less Than Values--The analyte is present, but the amount of the analyte is determined to be below an acceptable level for quantitation. The concentration can not be calculated, but is determined to be less than the value given. Example: 10K means that the analyst has determined that the analyte is present at some undetermined amount less than 10.

10.11.1.5.7. L - The analyte is determined to be present. The actual value is known to be "greater than" the value preceding the "L".

10.11.1.5.7.1. Greater Than Values--The analyte is present, but the amount of the analyte is determined to be above an acceptable level for quantitation. Example: 500L means that the analyte is present at some undetermined amount greater than 500.

10.11.1.5.8. R - Data is "rejected" and should not be used.

10.11.1.5.8.1. Rejected Data - Some or all of the quality control data for the analyte were outside criteria. The presence or absence of the analyte can not be determined from the data. Resampling and reanalysis are necessary to confirm or deny the presence of the analyte.

10.11.1.5.9. UJ - This is a combination of the "U" and "J" codes. The analyte is not present and the value preceding "UJ" is an estimated MQL.

10.11.1.5.10. JN - This is a combination of the "J" and "N" codes. The analyte is tentatively identified and the value preceding the "JN" is estimated.

10.11.1.5.11. JR - This is a combination of the "J" and "R" codes. The analysis indicated the presence of the analyte. The data is rejected and the value preceding "JR" is estimated. Resampling and reanalysis are necessary to confirm or deny the presence of the analyte.

10.11.1.5.12. UR - This is a combination of the "U" and "R" codes. The analysis did not indicate the presence of the analyte. The data is rejected and the value preceding "UR" is the MQL. Resampling and reanalysis are necessary to confirm or deny the presence of the analyte.

10.11.1.5.13. NAI - Not analyzed due to interference.

10.11.1.5.14. NA - Not analyzed for.

10.11.1.6. The number precedes the code letter in all cases.

10.11.1.7. When adding one or more J values to real values (e.g., for DDTR), J the total if the J values are equal to $\geq$ 10% of the total value.

10.11.1.8. Rules of rounding. Round off by dropping digits that are not significant. If the digit 6,7,8, or 9 is dropped, increase preceding digit by one unit; if the digit 0,l,2,3, or 4 is dropped, do not alter preceding digit. If the digit 5 is dropped, round off preceding digit to the nearest even number: thus 2.25 becomes 2.2 and 2.35 become 2.4.

10.11.1.9. Reporting estimated minimum quantitation limits (MQL) - The MQL is reported to 2 significant figures.

10.11.1.10. Reporting target compounds below the MQL - Report the actual calculated value (to 2 significant figures) with a J for any concentration below the MQL. Anything below l/10 of the MQL and/or less than a 3mm peak height is reported as not detected.

10.11.1.11. If dilutions of the sample extract are required, the MQL is raised by the same factor as the dilution.

10.11.1.12. Determine from the analytical request if a specific limit of quantitation is required. This is especially important when analyzing samples for compliance monitoring, spill investigations, and drinking water investigations.

10.11.1.13. Calculate the MQL for the blank using the lowest sample weight or sample volume.

10.11.1.14. Report all non-target (library search compounds) data to 1 significant figure.

10.11.1.15. Inorganic compounds. Do not report sulfur, H_2S, SO_2, etc.

10.11.1.16. Reporting duplicate data - Calculate and report the average with an A flag. If a compound is detected on one duplicate and not the other, do not report the compound.

10.11.2. Nomenclature for Library Search Compounds

10.11.2.1. Spaces in chemical names - Be careful where spaces are used in nomenclature especially when rearranging CAS names. Where there are dashes, always attach the words. Where there are spaces, always leave them.

10.11.2.1.1. Caution! Location of spaces, brackets, and parentheses are very important when entering names and should be entered consistently.

10.11.2.2. Names should be entered in all caps.

10.11.2.3. Isomers. Report as METHYLBIPHENYL (2 ISOMERS). Make sure spacing is strictly adhered to.

10.11.2.4. Isomers of Target Analytes: TRICHLOROBENZENE (NOT 1,2,4-) (2 ISOMERS).

10.11.2.5. Specified compounds: Enter as 6275*SPECIFIED COMPOUND NAME.

10.11.2.6. ESAT should send a copy of final edited data to the work assignment monitor.

10.11.2.7. Acids. Always precede acids with a space, same for esters, acetates, oxides, etc. (i.e. BENZOIC ACID).

10.11.2.8. Isomers and/or rearranged names, 2-HEXANONE, 5-METHYL-3-METHYLENE -, rearrange and combine to METHYLMETHYLENEHEXANONE. Dashes indicate no space when the name is rearranged.

10.11.2.9. Alkyls. No space.

10.11.2.10. Esters. Do not rearrange esters (i.e., DODECANOIC ACID, METHYL ESTER not METHYL ESTER OF DODECANOIC ACID).

10.11.2.11. Truncated names in data system. The current GC/MS data system library only stores the first 70 characters. Long names with odd looking endings should be looked up to verify the complete name. The HS library display truncates if the name extends beyond where the scan number is printed.

10.11.2.12. Unidentified compounds. Report as 5 UNIDENTIFIED COMPOUNDS, 5 equals the number of compounds.

10.11.2.13. Hydrocarbon series. Report homologous hydrocarbon series as "PETROLEUM PRODUCT" with an "N" flag in the result field.

10.11.2.14. Common names vs CAS names. The following commonly found compounds are changed from CAS names to common names:

All pesticides recognized as such
2-PROPANONE TO ACETONE
2-PROPANOL TO ISOPROPANOL
2- BUTANONE TO METHYL ETHYL KETONE
2-PENTANONE TO METHYL PROPYL KETONE
3 METHYL-2-BUTANONE TO METHYL ISOPROPYL KETONE
4 METHYL-2-PENTANONE TO METHYL ISOBUTYL KETONE
THIOBISMETHANE TO METHYL SULFIDE
ACETIC ACID ETHYL ESTER TO ETHYL ACETATE
1,1′-OXYBISETHANE TO ETHYL ETHER
2,2′-OXYBISPROPANE TO ISOPROPYL ETHER
1,2-BENZENEDICARBOXYLIC ACID TO PHTHALIC ACID
ETHENYLBENZENE TO STYRENE

10.11.3. Laboratory Contaminants

10.11.3.1. The following organics are frequently detected in blanks in trace concentrations. Therefore, special precautions need to be taken when reporting positive findings of these compounds. Do not subtract blank values from sample values unless specified by the method.

10.11.3.1.1. Volatile Organic Analysis

10.11.3.1.1.1. Do not report compounds unless they are 5 times the blank value.

10.11.3.1.1.2. Background contamination by methylene chloride, methylethylketone, and acetone may yield higher than normal values. Therefore, do not report these compounds unless they exceed 10 times the blank value.

10.11.3.1.2. Extractable Organic Analysis

10.11.3.1.2.1. Report phthalates only if above the MQL.

10.11.3.1.2.2. Do not report these compounds unless they are present at 5 times the blank value: bis(2-ethylhexyl) phthalate, diethyl phthalate, dibutyl phthalate, n-nitrosodiphenylamine, the xylenes, silicones, and phthalic acid.

10.11.3.1.2.3. Butoxyethoxyethanol and related compounds are common contaminants of tubing used in automatic samplers. Therefore, if these compounds are detected in the sampler blank, report them in sampler blank and all samples where identified.

10.11.3.2. Reporting of Data when Considering Other Possible Contaminants

10.11.3.2.1. Use the following criteria when evaluating the validity of a positive identification (see aslo SW-846, 8000 methods):

10.11.3.2.1.1. If the compound in question is in the blank and in the sample but the concentration in both is <MQL, report the compound as --U.

10.11.3.2.1.2. If a compound in question is detected in the blank at greater than the usual MQL and is in a sample at $\leq$ the blank, report the sample value as the MQL for the sample.

10.11.3.2.1.3. If the compound in question is in the blank at <MQL but in the sample at >MQL, then the analyst must use professional judgement in determining its validity.

10.11.3.2.1.4. In general, it should be >2 times the blank value before reporting. The same is true if the compound in question is present in both the blank and sample, and >MQL. If the compound is reported with a U, the MQL should be adjusted to the level found in the sample.

10.11.3.2.1.5. Natural organics in fish - Do not report cholestanol or related compounds.

ANALYTICAL DESCRIPTORS

Method#	Descriptor	Sample Type
46A	VLW	Volatile low water
43D	VLS	Volatile low soil/sed
54B	VMS	Volatile medium soil/sed
54B	VMW	Volatile medium waste
56	VAC	Volatile air canister
56B	VAC	Volatile air canister
60	VTC	Volatile by TCLP Extraction
47	SLW	Semivolatile low water - separatory funnel
47A	SLW	Semivolatile low water - cont. liquid ext.
43	SLG	Semivolatile low soil/sed w/GPC
43A	SLS	Semivolatile low soil/sed wo/GPC
54	SMS	Semivolatile medium soil/sed
54A	SHW	Semivolatile high waste wo/son.
54C	SMW	Semivolatile medium waste w/son.
44	SLT	Semivolatile low tissue
50B	SAP	Semivolatile air PUF
58	SCW	Semivolatile Cartridge ext. water
60	STC	Semivolatiles by TCLP extraction
31C	SSS	Semivolatiles low soil with soxhlet
55	PLW	Pesticide low water - separatory funnel
55A	PLW	Pesticide low water - cont. liquid ext.
43	PLG	Pesticide low soil/sed w/GPC
43A	PLS	Pesticide low soil/sed wo/GPC
43B	PSA	Pesticide low soil/sed w/acid cleanup
43C	PSH	Pesticide low soil/sed w/hex/acetone
44	PLT	Pesticide low tissue
44A	PTH	Pesticide low tissue w/hexane
44B	PTA	Pesticide low tissue w/acid cleanup
50B	PAP	Pesticide air PUF
54	PMS	Pesticide medium soil/sed
54A	PHW	Pesticide high waste wo/son.
54C	PMW	Pesticide medium waste w/son.
52B	PCW	Pesticide Cartridge ext. water
57	PNP	Pesticide low water nitrogen/phosphorous
60	PTC	Pesticides by TCLP extraction
35	PWO	PCBs waste oil
31A	PCS	Pesticide low soil/sed w/sohxlet
31C	PSS	Pesticide/PCB low soil/sed w/sohxlet
38	HLW	Herbicides low water
51	HLS	Herbicides low soil/sed
60	HTC	Herbicides by TCLP extraction
48	FLW	Formaldehyde low water
48	FLS	Formaldehyde low soil/sed
59	FAC	Formaldehyde air cartridge
61	CLW	Carbamates low water w/HPLC
61	CLS	Carbamates low soil/sed w/HPLC

10.11.3.2.1.6. Do not report chlorinated and brominated cyclohexenes, cyclohexanes, and cyclohexanols if chlorinated water was extracted with methylene chloride.

The sampler should verify that it was chlorinated water and write a memo to the Chemistry Section, so stating.

Standard Levels and Concentrations

	Red Level	Linear Curve Concentration				
CDI Components	Concentrations	Level QC-1	Level QC-2	Level QC-3	Level QC-4	Level QC-5
TCMX		0	0.0025	0.005	0.01	0.02
g- BHC (Lindane)	0.005	0.001	0.002	0.004	0.008	0.016
Aldrin	0.010	0.002	0.004	0.008	0.016	0.032
Heptachlor	0.008	0.0015	0.0030	0.0060	0.0120	0.0240
Hept. Epoxide	0.010	0.002	0.004	0.008	0.016	0.032
Endosulfan I	0.010	0.002	0.004	0.008	0.016	0.032
Endosulfan II	0.020	0.004	0.008	0.016	0.032	0.064
Dieldrin	0.010	0.002	0.004	0.008	0.016	0.032
p,p'-DDT	0.025	0.005	0.010	0.020	0.040	0.080
Endrin	0.020	0.004	0.008	0.016	0.032	0.064
Methoxychlor	0.050	0.010	0.020	0.040	0.080	0.160
DBC		0.00313	0.0063	0.013	0.025	0.050
CDII Components						
TCMX		0.0013	0.0025	0.0050	0.0100	0.0200
a- BHC	0.005	0.001	0.002	0.004	0.008	0.016
b- BHC	0.010	0.002	0.004	0.008	0.016	0.032
d- BHC	0.010	0.002	0.004	0.008	0.016	0.032
Aldrin	0.010	0.002	0.004	0.008	0.016	0.032
p,p'-DDT	0.025	0.005	0.010	0.020	0.040	0.080
p,p'-DDD	0.020	0.004	0.008	0.016	0.032	0.064
p,p'-DDE	0.010	0.002	0.004	0.008	0.016	0.032
Endrin Aldehyde	0.025	0.005	0.010	0.020	0.040	0.080
Endrin Ketone	0.025	0.005	0.010	0.020	0.040	0.080
Endosulfan Sulfate	0.025	0.005	0.010	0.020	0.040	0.080
DBC		0.00313	0.0063	0.013	0.025	0.050
Multi Component Compounds						
Toxaphene	0.500	0.200	0.400	0.600	0.800	1.000
Technical Chlordane	0.075	0.0125	0.025	0.050	0.100	0.200
Chlordane Constituents I						
a-Chlordane	0.010	0.0025	0.005	0.010	0.020	0.040
b-Chlordene	0.010	0.0025	0.005	0.010	0.020	0.040
g-Chlordane	0.010	0.0025	0.005	0.010	0.020	0.040
Chlordane Constituents II						
Chlordene	0.005	0.0013	0.0025	0.005	0.01	0.02
a-Chlordene	0.010	0.0025	0.005	0.010	0.020	0.040
g-Chlordene	0.010	0.0025	0.005	0.010	0.020	0.040
Oxy chlordane	0.010	0.0025	0.005	0.010	0.020	0.040
Cis Nonachlor	0.010	0.0025	0.005	0.010	0.020	0.040
Trans Nonachlor	0.010	0.0025	0.005	0.010	0.020	0.040
PCB's						
AR 1242	0.15	0.025	0.050	0.100	0.200	0.300
AR 1248	0.15					

Standard Levels and Concentrations						
	Red Level	Linear Curve Concentration				
CDI Components	Concentrations	Level QC-1	Level QC-2	Level QC-3	Level QC-4	Level QC-5
TCMX		0	0.0025	0.005	0.01	0.02
g- BHC (Lindane)	0.005	0.001	0.002	0.004	0.008	0.016
Aldrin	0.010	0.002	0.004	0.008	0.016	0.032
Heptachlor	0.008	0.0015	0.0030	0.0060	0.0120	0.0240
Hept. Epoxide	0.010	0.002	0.004	0.008	0.016	0.032
Endosulfan I	0.010	0.002	0.004	0.008	0.016	0.032
Endosulfan II	0.020	0.004	0.008	0.016	0.032	0.064
Dieldrin	0.010	0.002	0.004	0.008	0.016	0.032
p,p'-DDT	0.025	0.005	0.010	0.020	0.040	0.080
Endrin	0.020	0.004	0.008	0.016	0.032	0.064
Methoxychlor	0.050	0.010	0.020	0.040	0.080	0.160
DBC		0.00313	0.0063	0.013	0.025	0.050
CDII Components						
TCMX		0.0013	0.0025	0.0050	0.0100	0.0200
a- BHC	0.005	0.001	0.002	0.004	0.008	0.016
b- BHC	0.010	0.002	0.004	0.008	0.016	0.032
d- BHC	0.010	0.002	0.004	0.008	0.016	0.032
Aldrin	0.010	0.002	0.004	0.008	0.016	0.032
p,p'-DDT	0.025	0.005	0.010	0.020	0.040	0.080
p,p'-DDD	0.020	0.004	0.008	0.016	0.032	0.064
p,p'-DDE	0.010	0.002	0.004	0.008	0.016	0.032
Endrin Aldehyde	0.025	0.005	0.010	0.020	0.040	0.080
Endrin Ketone	0.025	0.005	0.010	0.020	0.040	0.080
Endosulfan Sulfate	0.025	0.005	0.010	0.020	0.040	0.080
DBC		0.00313	0.0063	0.013	0.025	0.050
Multi Component Compounds						
AR 1254	0.15					
AR 1260	0.25	0.025	0.050	0.100	0.200	0.300
AR 1268	0.25					

Table 10-2

BOOK _____

EXTRACTION OF WATER

Project _____
Analysts_____

Extraction Solvent & Volume
Pesticide _____ **Surrogate**
Acid _____ **Spike**
Base/Neutral _____ **BNA** _____
Herbicide _____ **Pest**_____
Comments: _____

Spike Solution, Volume & Solvent Method/Matri
Pesticide _____|_____|_____
Acid _____|_____|_____
Base/Neutral _____|_____|_____
Herbicide _____|_____|_____

Method No: _____

Start date:								
Start time:								
SAD #	BNA VOL SAMPLE	FINAL VOL	PESTICIDES VOL SAMPLE	FINAL VOL	HERBICIDE VOL SAMPLE	FINAL VOL	_____ __ VOL SAMPLE	FINAL VOL
STOP DATE:								
TIME:								
FINAL PROG CHECK								

Form 10-1

BOOK_____
EXTRACTION OF SOLIDS/WASTE

Project_____DATE_____
Analysts_____

Extraction Method#_____B/N Acid VOA Pest. Herb._____

Extraction Device:_____Soxhlet_____Sonicator_____

Surrogate Spike Added BNA_____ Florisil _____5% _____ 15% _____ 50%
Pest_____ Spike Solution

Cleanup/Separation	Extraction Solvent & Volume	Volume/Solvent	Method/Matrix
Acetonitrile_____	Pesticide_____	Pesticide_____	_____
Sulfuric Acid_____	Acid_____	Acid_____	_____
Auto Prep _____	Base/Neutral_____	B/N_____	_____
_____	Herbicide_____	Herbicide_____	_____
	_____	PCB's_____	_____

Esterification:_____Diazomethane_____

COMMENTS:_____

SAD NUM.	CRUC SURR ADD / SURR	GROSS WT.	DRY WT.	TARE WT.	DRY WT / WET WT	% SOLID	WET WT EXTRACT	DRY WT EXTRACT	FINAL VOL

Calculations _____ Completed Date _____
Checked _____ Final Project Check _____

Form 10-2

BOOK_____
EXTRACTION OF BIOLOGICAL TISSUE

Project_____DATE_____
Analysts_____

Extraction Method#_____B/N Acid Pest. PCB _____

Extraction Device: Sonicator_____
_____

Surrogate Spike Added BNA_____ Pest_____

Cleanup/Separation	Extraction Solvent & Volume	Volume/Solvent	Method/Matrix
GPC _____	Pesticide_____	Pesticide_____	_____
Alumina _____	Acid_____	Acid_____	_____
Sulfuric Acid_____	Base/Neutral_____	B/N_____	_____
	_____	PCB's_____	_____

COMMENTS:_____
_____

SAD NUM	GROSS WT	TARE WT	WEIGHT OF OIL	% LIPID	WET WEIGHT	FINAL VOL	COMMENTS

Calculations _____ Completed Date _____
Checked _____ Final Project Check _____

Form 10-3

BOOK/PAGE 105 /____

GC ANALYSIS

INSTRUMENT DETECTOR COLUMN TYPE

____ HP 5890 Hall/PID DB624 30m x 0.53mm ID x 3um FT
RESTEK 30m X 0.53mm ID x 3um FT

____ HP 5890 Hall/PID ___DB5 30m x 0.32mm ID x 1um FT
FID ___Other_____

Sweep: _____ min _____ ml/min Split: _____ min _____ ml/min Makeup: _____ml/m

Init:_____°C Final:_____°C Rate:_____ °C/min Init. Hold:_____ Final Hold_____

Atten:____ Hall _____PID _____FID _____ Other

PSI: _____ Volume Inj: _____

RUN #	PROJ #	SAMPLE	VOLUME	DIL	COMMENTS	DATE	ANAL

Form 10-4

GC/MS ANALYSIS

BOOK/PAGE 135/__
DATE _____
ANALYST _____
TRANSFERRED_____

PROJECT(S) _____

INSTRUMENT: __5971 __5972 __5973S __5973V ___5973B _X 5200 __5300 METHOD#: _____
COLUMN: _X_ DB5MS-FSCC ___ DB 624 FSCC ____ Other (Specify) _____
Meters: 30 ID: .25 mm FT: .25 um Purge Temp/Flow: ___/___ ACQU TIME: ____ EMV: _____V
Pulse Splitless: .5 min 35 PSI Vent: .6 min 60 ml/min EPC/He: 1.2 ml/min Makeup: _____ ml/min
Init. l °C/Hld: 40 / 2 Rate l: 35 Init. 2 °C/H: 130 / 0 Rate 2: 12 Final °C/H: 300 / 8
SS#l D5Phenol SS#2 D5Nitrobenzene SS#3 Tribromophenol SS#4 D14Terphenyl

SLOT# TIME	SS#1	SS#2	SS#3	SS#4	M	C	Q	MAG TAPE	FILE NAME	VOL INJ.	COMMENTS	INT. STD. AREA

Form 10-5

PREPARATION OF STOCK STANDARD

BOTTLE NO._____
COMPOUND_____

SOL. NO.	DATE	INT	COMPOUND SOURCE	LOT NO.	PURITY	NET WEIGHT	ADJUSTED NET WEIGHT*	SOLVENT	DILUTION VOLUME ml	FINAL CONC. ng/ul	BALANCE CHECK ACTUAL/-FOUND	COMMENTS
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												

Form 10-6

SUMMARY LOG SHEET

SECTION_______________

B - Bottle Number
D - Dilution Number

Name of Standard or Mix	B	D	Date	B	D	Date	B	D	Date	B	D	Date

Form 10-7

GC ANALYSIS BOOK-PAGE _____

PROJECT(S) _____

ANALYST(S) _____ DATE _____

ANALYSES:
___ Pesticide Quantitation
___ Organic Quantitation
___ PCB Quantitation
___ Herbicide Quantitation
___ Confirmation
___ _____

INSTRUMENT:
___ HP5890-1
___ HP5890-2
___ HP5890-3
___ HP5890-4
___ HP5890 ESAT
___ HPLC/Fluorescence
___ HPLC/PDA

DETECTOR:
___ Electron Capture NI[63]
___ Nitrogen/Phosphorus Thermionic
___ Fluorescence
___ PDA

GC COLUMN TYPE:
___ C_{18} HPLC
___ DB5-FSCC
___ DB608-FSCC
___ DB1701-FSCC
___ DB1301-FSCC
___ DB210-FSCC
___ DB1-FSCC ___ _____

COLUMN INFORMATION:
1. Capillary Length ____m ID ____ Film Thickness ____um
2. Capillary Length ____m ID ____ Film Thickness ____um
____ _____

STANDARD MIX:
___ Red _____ Zip File: _____
___ Yellow _____ _____

Temperature: Iso _____°C. for ____ min. Program: Initial _____ ∘C. for _____ min.
Attenuation: A _____ 1. Vol. Inj. ____ ul. Level 1 ____ ∘C. for ____ min. @ ___ ∘C/min.
B _____ 2. Vol. Inj. ____ ul. Post Value _____ ∘C. for _____ min.

			SAD # TYPE ANALYSIS	DILUTION	FINAL VOLUME	QC	REMARKS
1	24	47					
2	25	48					
3	26	49					
4	27	50					
5	28	51					
6	29	52					
7	30	53					
8	31	54					
9	32	55					
10	33	56					
11	34	57					
12	35	58					
13	36	59					
14	37	60					
15	38	61					
16	39	62					
17	40	63					
18	41	64					
19	42	65					
20	43	66					
21	44	67					
22	45	68					
23	46	69					

Form 10-8

PESTICIDE / HERBICIDE **SURROGATE RECOVERIES (%)**

DATE ANALYZED_____

SAMPLE TYPE_____

			Ext. org.				Pest.*		VOA			Pest
SAMPLE NO.	**METHOD**	**INSTRUMENT**	**1**	**2**	**3**	**4**	**5**	**6**	**10**	**11**	**12**	**13**

1) Phenol-D5
2) 2,4,6-Tribromophenol
3) Nitrobenzene-D5
4) Terphenyl-D14
5) 1,2,3,4-TCDD
6) Dibutylchlorendate
10) D_4-1,2-Dichloroethane
11) p-Difluorobenzene
12) p-Bromofluorobenzene
13) 2,4,5,6 Tetrachloro-m-xylene

*** DCAA = 2,4-Dichlorophenyl Acetic Acid**
*** NMX = 2-Nitro-m-xylene**

Form 10-9

MASTER LOG-GC ANALYSIS

Sample Type

W-Water	**WA-Waste**
S-Sediment	**AP-Air Puffs**
F-Fish	**O-Oil**
B-Biological Material	**MLW-Midlevel Waste**
P-Plant	**X-Others**

Project	Ext Sht	Project #	Sample Type	Date Received (GC Lab)	Projected Completion Date	Analysis Type				Analysis Progress					Project File Progress		
						Pest	PCB	Herb	Other	GC Screen	Calc	Calc Check	Date	Data Recorded	Project Archive Name		

Form 10-10

PESTICIDE QC DATA REPORTING SHEET
H2O PEST MATRIX A
P27

Sample #_____
Std QC Types
___Reference Ref#_____
___PE Sample PE#_____ **Chemist_____** **Date_____**
___NEIC PRP **Instrument_____** **Method_____**
___Method Spk **Vol/Wt._____** **Vol/Wt._____**
Other_____ **Remarks_____**

Test#	Compound	Dup 1	Dup 2	Spike Added	%Rec 1	%Rec 2
5030	Gamma-BHC			500		
5010	Heptachlor			1000		
5005	Aldrin			1000		
5015	Heptachlor Epoxide			1000		
5075	Alpha-Endosulfan			1000		
5045	Dieldrin			1000		
5175	Methoxychlor			1000		
5050	P, P-DDT			2000		
5070	Beta-Endosulfan			2000		
5125	Endrin Aldehyde			2500		

PESTICIDE QC DATA REPORTING SHEET
H_2O PEST MATRIX B
P28

SAMPLE #_____
Std QC Types
___Reference Ref#_____
___PE Sample PE#_____ **Chemist_____** **Date_____**
___NEIC PRP **Instrument_____** **Method_____**
___Method Spk **Vol/Wt._____** **Vol/Wt._____**
Other_____ **Remarks_____**

Test#	Compound	Dup 1	Dup 2	Spike Added	%Rec 1	%Rec 2
5020	Alpha-BHC			500		
5025	Beta-BHC			1000		
5035	Delta-BHC			1000		
5005	Aldrin			1000		
5055	P, P-DDE			1000		
5065	Endrin			1000		
5165	Alpha-Chlordane			1000		
5155	Gamma-Chlordane			1000		
5060	P, P-DDD			2000		
5075	Endosulfan Sulfate			2000		

PESTICIDE QC DATA REPORTING SHEET
SOIL PEST MATRIX A
P29

Sample #_____
Std QC Types
___Reference Ref#_____
___PE Sample PE#_____ **Chemist_____** **Date_____**
___NEIC PRP **Instrument_____** **Method_____**
___Method Spk **Vol/Wt._____** **Vol/Wt._____**
Other_____ **Remarks_____**

Test#	Compound	Dup 1	Dup 2	Spike Added	%Rec 1	%Rec 2
5030	Gamma-BHC			1250		
5010	Heptachlor			2500		
5005	Aldrin			2500		
5015	Heptachlor Epoxide			2500		
5075	Alpha-Endosulfan			2500		
5045	Dieldrin			2500		
5175	Methoxychlor			2500		
5050	P, P-DDT			5000		
5070	Beta-Endosulfan			5000		
5125	Endrin Aldehyde			6250		

PESTICIDE QC DATA REPORTING SHEET
SOIL PEST MATRIX B
P30

Sample #_____
Std QC Types
___Reference Ref#_____
___PE Sample PE#_____ **Chemist_____** **Date_____**
___NEIC PRP **Instrument_____** **Method_____**
___Method Spk **Vol/Wt._____** **Vol/Wt._____**
Other_____ **Remarks_____**

Test#	Compound	Dup 1	Dup 2	Spike Added	%Rec 1	%Rec 2
5020	Alpha-BHC			1250		
5025	Beta-BHC			2500		
5035	Delta-BHC			2500		
5005	Aldrin			2500		
5055	P, P-DDE			2500		
5065	Endrin			2500		
5165	Alpha-Chlordane			2500		
5155	Gamma-Chlordane			2500		
5060	P, P-DDD			5000		
5075	Endosulfan Sulfate			5000		

A=COLOR CODE B=BOTTLE NUMBER C=DILUTION NUMBER

NAME_____ SOLVENT_____

BOTTLE#___ COLOR CODE___ COMPOUND	Parent Sol. ID A-B-C	Conc. Parent ng/ul	Ali. Vol. ml	Fin. Conc. ng/ul	Fin. Vol. ml

DILUTION	DATE	INITIALS

	BOTTLE#___ COLOR CODE___	Parent Sol. ID A-B-C	Conc. Parent ng/ul	Ali. Vol. ml	Fin Conc. ng/ul	Fin Vol. ml
1.	COMPOUND					
2.						
3.						
4.						
5.						

Dilution Date	#1	2	3	4	5
Initials					

	BOTTLE#___ COLOR CODE___	Parent Sol. ID A-B-C	Conc. Parent ng/ul	Ali. Vol. ml	Fin. Conc. ng/ul	Fin. Vol. ml
1.	COMPOUND					
2.						
3.						
4.						
5.						
6.						
7.						
8.						
9.						
10.						

Dilution #	2	3	4	5	6	7	8	9	10
1 Date									
Initials									

MQL[1] GUIDELINES FOR DIFFERENT MATRICES					
COMPONENT	**DW ug/l (ppb)[2]**	**WATERS (other) ug/l (ppb)[2]**	**SED ug/kg (ppb)[3]**	**WASTE mg/kg (ppm)[4]**	**FISH mg/kg (ppm)[5]**
Aldrin	**0.10 U**	**0.50 U**	**50 U**	**0.20 U**	**0.050 U**
Heptachlor	**0.10 U**	**0.50 U**	**50 U**	**0.20 U**	**0.050 U**
Hept. Epoxide	**0.10 U**	**0.50 U**	**50 U**	**0.20 U**	**0.050 U**
alpha-BHC	**0.10 U**	**0.50 U**	**50 U**	**0.20 U**	**0.050 U**
beta-BHC	**0.10 U**	**0.50 U**	**50 U**	**0.20 U**	**0.050 U**
gamma-BHC	**0.10 U**	**0.50 U**	**50 U**	**0.20 U**	**0.050 U**
delta-BHC	**0.10 U**	**0.50 U**	**50 U**	**0.20 U**	**0.050 U**
Endosulfan- I	**0.10 U**	**0.50 U**	**50 U**	**0.20 U**	**0.050 U**
Dieldrin	**0.10 U**	**0.50 U**	**50 U**	**0.20 U**	**0.050 U**
pp-DDT	**0.10 U**	**0.50 U**	**50 U**	**0.20 U**	**0.050 U**
pp-DDE	**0.10 U**	**0.50 U**	**50 U**	**0.20 U**	**0.050 U**
pp-DDD	**0.10 U**	**0.50 U**	**50 U**	**0.20 U**	**0.050 U**
Endrin	**0.10 U**	**0.50 U**	**50 U**	**0.20 U**	**0.050 U**

[2] Based on an extraction of 1L and a final concentration vol. of 1.0ml.

[3] Based on an extraction of approx. 25gm(dry weight) and a concentration vol. of 4.0ml.

[4] Based on an extraction of 1.0gm(wet weight) and a final concentration vol. of 10ml.

[5] Based on an extaction of 25gm(wet weight) and a final concentration vol. of 4.0ml.

DW = drinking water SED = sediments

MQL[1] GUIDELINES FOR DIFFERENT MATRICES					
Endosulfan -II	**0.10 U**	**0.50 U**	**50 U**	**0.20 U**	**0.050 U**
Endosulfan- SO4	**0.10 U**	**0.50 U**	**50 U**	**0.20 U**	**0.050 U**
Endrin Ketone	**0.10 U**	**0.50 U**	**50 U**	**0.20 U**	**0.050 U**
Methoxychlor	**0.25 U**	**1.0 U**	**200 U**	**0.50 U**	**0.20 U**
Tech. Chlordane	**0.25 U**	**1.0 U**	**200 U**	**0.50 U**	**0.20 U**
PCB	**0.50 U**	**2.0 U**	**500 U**	**1.0 U**	**0.50 U**
Toxaphene	**5.0 U**	**20 U**	**3000 U**	**8.0 U**	**3.0 U**

BOX N0._____ STORAGE LOCATION_____

	Sample #				
1			50		
2			51		
3			52		
4			53		
5			54		
6			55		
7			56		
8			57		
9			58		
10			59		
11			60		
12			61		
13			62		
14			63		
15			64		
16			65		
17			66		
18			67		
19			68		
20			69		
21			70		
22			71		
23			72		
24			73		
25			74		
26			75		
27			76		
28			77		
29			78		
30			79		
31			80		
32			81		
33			82		
34			83		
35			84		
36			85		
37			86		
38			87		
39			88		
40			89		
48			97		
49			98		

Form 10-17

11. INORGANIC ANALYSIS, PERFORMANCE QUALITY CONTROL AND ANALYTICAL OPERATION

11.1. Every element of environmental data acquisition, from sample collection to final data reporting, has associated with it degrees of error. The primary purpose of a quality assurance program is the optimization of conditions whereby the introduction of error can be either precluded or substantially reduced. The operating procedures and quality control checks practiced in this laboratory and outlined in this manual are implemented to minimize the total error associated with data generation. No number can be affixed to total error; however, analytical performance is measurable and thus definable. This section is limited to a discussion of the analytical operation and procedures used in this laboratory to measure and record analytical performance.

11.2. General

11.2.1. During the course of generating data on samples for inorganic parameters, it is the policy of the Analytical Support Branch (ASB) to apply the best laboratory practices, use approved methodology when mandated by regulation, use standardized methodology, if possible, when approved methodology is not applicable, fully document all operations associated with the generation of data and to meet certain quality requirements that will be designated in the following paragraphs. It should be noted, however, that occasionally certain matrices and samples present analytical challenges, or are not amenable to standardized methodology. In these instances modifications to standard protocols may have to be made to produce a high quality analysis. When this occurs, any deviations from standard operating procedures will be fully documented.

11.2.2. Safety precautions associated with the safe handling of toxic chemicals, reagents, solutions and samples will be observed and regarded as a first order responsibility of the analyst. The analyst will take the necessary precautions to prevent exposure or harm both to himself and his fellow workers.

11.2.3. Water used to prepare calibration standards, spike solutions, standard reference solutions or any sample dilutions or mixtures must meet or exceed the requirements for Type II grade water as specified by the American Society for Testing and Materials (ASTM); Standard Practice D 1193. This grade water is equivalent to Type II water as specified in Standard Methods 1080. The parameter measured to verify the quality of water is resistivity, with a requirement of 1 megohm-cm at 25^0C or better. See also section 2.2 of Handbook for Analytical Quality Control in Water and Wastewater Laboratories (EPA 600/4-79-019, March l979), and any future updates of the manual. Reagent water used for trace metals determinations must meet or exceed the requirements for Type I grade water as specified by ASTM. The parameter monitored to verify the quality of water is resistivity, with a requirement of 18 megohms-cm at 25^0C or better. This grade water is equivalent to Standard Methods Type I water.

11.2.4. Reagents must be ACS reagent grade quality or better. All reagents will be dated upon receipt, and will be properly disposed of when the shelf life has been reached.

11.3. Custody

11.3.1. The EPA Region IV Science and Ecosystem Technology Center is a "controlled access" facility. Entry to the facility is restricted to employees and is controlled by keycards. All visitors to the facility must enter through the guard station at the main entrance and be escorted by a host when in the facility. Additionally, access to the custody room is controlled by keycard. Only employees with legitimate reason for access to samples have keycard access to the custody room. All samples removed from the custody room must be signed out. When a sample is signed out, the signee is legally assuming custody of the sample and is responsible for its integrity and accountability during possession. Custody is relinquished only when the samples have been returned and signed back to the custody room. Aliquots taken from the original samples for analysis will be accounted for by entering sample ID in the proper log books during preparation and analysis.

11.4. Metals Metals analyses are performed in support of various agency programs. Some programs mandate methods (e.g. Drinking Water at 40CFR Part 141 ff. and NPDES at 40 CFR Part 136), while others publish methods strictly as guidance (e.g. RCRA except for the Characteristic Tests at 40CFR Subpart C Part 261.20 ff.) Subject to the restrictions in 11.2.1, mandated methodology will be used for those analyses requiring them. Guidance methods will be closely adhered to with the possibility of minor changes which do not change the chemistry of the procedure. In any event, all procedures will be fully documented. The following programs are supported by laboratory analyses:

11.4.1. Drinking Water

11.4.1.1. Regulatory Authority: National Primary Drinking Water Regulations are found at 40 CFR Part 141. National Secondary Drinking Water Regulations are found at 40 CFR Part 143. In general these regulations apply to Public Water Systems which are defined as "a system for the provision to the public of piped water for human consumption, if such system has at least fifteen service connections or regularly serves an average of at least twenty-five individuals at least 60 days out of the year." Historically, this laboratory has analyzed few samples from public water systems as the states have been delegated the authority for monitoring public water supplies within their boundaries. However, this laboratory does often analyze samples from individual private potable wells. While not legally obligated to adhere to the requirements of 40 CFR Part 141 for these samples, this lab has chosen to follow the requirements Part 141 whenever possible when analyzing private potable wells.

11.4.1.2. Identification of Samples: Drinking water samples from public water systems will be logged into the data system with the program element **HOH**. The requirements of 40 CFR Part 141 must be adhered to for the analysis of these samples. Samples from individual potable wells may be received under any program element, the most common being **RCRA** or Superfund (**SSF** or **NSF**). The samples will be identified on the sample log sheets as "Potable Well", code **PW**. Requirements of 40 CFR Part 141 will be met whenever practicable.

11.4.1.3 Preparation and Analysis of Drinking Water Samples: Any or all of the following methods in Table 11.1 may be used by this laboratory for the analysis of drinking water and potable well samples. Prior to analysis samples will be digested using the procedure in the approved method. The digestion step may be

omitted on those samples with a turbidity of less than 1 nephelometric turbidity unit (NTU) and a "direct analysis" may be performed. (Technical Notes on Drinking Water Methods, EPA 600/R-94-173, October 1994 as referenced in 40CFR 141.23.)

Table 11.1 Drinking Water Methods

ANALYTE	MCL (mg/L)	ICP[4]	Graphite Furnace[4]	Graphite Furnace[5]	ICP-MS[4]	CVAA[4,6]
Antimony	0.006[1]		200.9	3113B	200.8	
Arsenic	0.050[2]	200.7	200.9	3113B	200.8	
Barium	2.0[1]	200.7		3113B	200.8	
Beryllium	0.004[1]	200.7	200.9	3113B	200.8	
Cadmium	0.005[1]	200.7	200.9	3113B	200.8	
Chromium	0.10[1]	200.7	200.9	3113B	200.8	
Lead	0.015[3]		200.9	3113B	200.8	
Mercury	0.002[1]				200.8	245.1 245.2
Nickel	0.10[1]	200.7	200.9	3113B	200.8	
Selenium	0.050[1]		200.9	3113B	200.8	
Thallium	0.002[1]		200.9		200.8	

Footnotes:
1 40CFR 141.23
2 40CFR 141.11
3 40CFR 141.80
4 ICP method 200.7, Graphite Furnace Method 200.9, ICP-MS Method 200.8 and Mercury CVAA Method 245.1 are in "Methods for the Determination of Metals in Environmental Samples-Suppplement 1", EPA-600/R-94-111, May 1994. Available from NTIS, PB 94-184942; (800) 553-6847.
5 Graphite Furnace Method 3113B is in 18th Edition of Standard Methods for the Examination of Water and Wastewater, 1992, American Public Health Association. Available from American Public Health Association, 1015 Fifteenth Street NW, Washington DC.
6 Mercury CVAA Method 245.2 is available from US EPA, NERL, Cincinnati, OH 45268. The identical method was formerly in "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020, March 1983 which is available from NTIS, PB84-128677; (800) 553-6847.

11.4.1.4. NPDES Monitoring: The National Pollutant Discharge Elimination System (NPDES) is the national system for the issuance of permits under section 402 of the Clean Water Act (CWA) of 1977 as amended. Test procedures for the analysis of pollutants are found at 40CFR Part 136.

11.4.1.5. Identification of Samples: Samples received by this laboratory will be logged into the data system under one of three program elements; ICSI (Industrial Compliance Sampling Inspection), MCSI (Municipal Compliance Sampling Inspection) or XCSI (Toxic Compliance Sampling Inspection).

11.4.1.6. Preparation and analysis of NPDES samples: Samples received in support of the NPDES program will be prepared and analyzed in accordance with the requirements at 40CFR 136. Table 11.2 lists approved test procedures for metals analyses that may be used by this laboratory. Digestion is required prior to analysis for all metals.

Table 11.2 NPDES Methods

Analyte	ICP	GFAA	Other	CVAA-HG
Aluminum	200.7[1]	3113B[2]		
Antimony	200.7	3113B		
Arsenic	200.7	3113B		
Barium	200.7	3113B		
Beryllium	200.7	3113B		
Boron	200.7			
Cadmium	200.7	3113B		
Calcium	200.7			
Chromium VI			3500-Cr D[2]	
Chromium	200.7	3113B		
Cobalt	200.7	3113B		
Copper	200.7	3113B		
Hardness	200.7			
Iron	200.7	3113B		
Lead	200.7	3113B		
Magnesium	200.7			
Manganese	200.7	3113B		
Mercury				245.1[1] 245.2
Molybdenum	200.7	3113B		
Nickel	200.7	3113B		
Potassium	200.7			
Selenium	200.7	3113B		
Silica	200.7			
Silver	200.7	3113B		
Sodium	200.7			
Thallium	200.7	279.2[1]		
Tin	200.7	3113B[1]		
Titanium		283.2[1]		
Vanadium	200.7	286.2[1]		
Zinc	200.7	289.2[1]		

Footnotes:

1 Methods for the Determination of Metals in Environmental Samples-Suppplement 1, EPA-600/R-94-111, May 1994.
2 Standard Methods for the Examination of Water and Wastewater. American Public Health Association, 18th Edition, 1992.

11.4.1.7. Other Waters: Monitoring well, ambient water, effluents and other water samples are digested with nitric-hydrochloric acids according to Method 200.2. All digests are scanned by ICP. Where the detection/quantitation technique is specified by program requirements, positive elements from the ICP scan will be verified by atomic absorption to satisfy the appropriate requirement. Mercury analyses are performed according to MCAWW[1]-245.1 or 245.1-Region 4 Modification or 245.7 CVAF depending on detection limit requirements. (245.1-Region 4 Modification consists of an autoclave digestion instead of a water bath digestion. The autoclave digestion of waters has proven effective and allows for greater throughput than the water bath digestion. All other method parameters remain the same.)

11.4.1.8. Soil and Sediment: A 50 g aliquot (approximately) is taken from a well mixed sample and weighed in a crucible. The sample is dried overnight at 60°C for a % moisture determination. The dried sample is ground to fineness and a l g subsample is taken for analysis. Sample digestion is conducted according to Method 200.2 or Method 3050[2] for those samples containing large amounts of organic matter and made up to l00 mLs for analysis.

11.4.1.9. Final low level data for thirty elements is generated by ICP (Method 200.7), Graphite Furnace (Method 200.9 or 3113B) or ICP-MS (Method 200.8, 6020, or 1638).

11.4.1.10. Mercury analysis of sediments will be conducted according to method 245.5 - Either water bath digestion or Section 11.3 (autoclave digestion) in Methods for the Determination of Metals in Environmental Samples, or by CVAF Region 4 method for those samples requiring lower levels of detection.

11.4.1.11. Fish: Whole fish are initially prepared with dry ice grinding followed by preparation by Method 200.3 (Methods for the determination of Metals in Environmental Samples, Supplement 1: EPA/600/R-94/111.)

11.4.1.12. Requests for analysis of individual organs or tissue can be satisfied by using the sample in its entirety or sub-sampling to obtain the maximum weight required for the analysis. The tissue should be kept frozen during sub-sampling and weighing to prevent fluid migration or drainage.

11.4.1.13. Mercury analyses on fish tissue are performed according to Region 4 modification of Method 245.5.

11.4.1.14. Digestion of tissue for multielement analyses is conducted according to Method 200.3 (nitric/peroxide) followed by ICP detection. Detection limit requirements are satisfied by manipulation of sample weight, final volume of digestate and in certain instances, detection by HGAA graphite furnace.

11.4.1.15. Other Tissue: Generally, other tissues will be prepared and analyzed the same as fish tissue with the additional precaution during preparation to observe closely and add additional reagents as required to thoroughly digest the sample.

11.4.1.16. EP/TCLP Extracts: Waste samples for EP Toxicity determinations will be extracted according to Method l3l0A in SW-846 and subsequent clarifications of the methodology as received from Office of Solid Waste. The extract may be acid digested (Method 30l0A SW-846) and scanned by ICP for the usual 26 elements. If any of the drinking water parameters are above the fail level in EP extracts, the parameter(s) will be confirmed by the method of standard additions, Method 6010. (Figure 2-6, Chapter Two, SW-846.) TCLP extracts will be prepared per Method 1311, optionally digested by Method 3010A and analyzed by Method 6010.

11.4.1.17. Oil and Oily Samples: Oil emulsions and oil samples will be prepared in one of two ways on a wet weight basis. Those oil samples which are thin enough to disperse on heating, yet not cover the entire surface of the digestion fluid resulting in a superheated solution will be prepared by Method 3050A. Those samples not amenable to Method 3050A will be prepared by the following method: The oil phase is weighed (l g) into a small crucible. The crucible is transferred to a muffle furnace and brought up to l25^{o}C for l hr. Increase temperature l75^{o}C for l hr. Increase to 250^{o}C for l hr. NOTE: Do not open furnace during the procedure and until furnace has cooled to l00^{o}C. The sample is ashed overnight at 450^{o}C maximum temperature. One mL of concentrated nitric acid and l mL of concentrated hydrochloric acid is added to the ash and warmed until ash is in solution. This solution is diluted to volume and is ready for analysis by ICP.

11.4.1.18. High Volume Filters: Air filters are prepared with a digestion fluid as outlined in 40 CFR Part 50, Appendix G. The digestion fluid is prepared by combining 167 mL of HNO_3 and 77 mL of HCL and diluting to 1 Liter. The resulting digestion fluid is 2.6 M HNO_3 and 0.9M HCl. The Federal Register method uses an ultrasonic extraction; however, an oscillating hot plate extraction is also acceptable. Typically, a 1x8 inch strip of a "high vol" filter is digested with a final volume of 100 mL, or a 1x4 inch strip is digested to 50 mL final volume. For the preparation of "saturation" filters, typically the entire filter is digested because of its small size.

11.4.1.19. Special Samples: Samples received for analysis which are not amenable to the standard digestion techniques will be prepared according to the best judgement of the analyst. These cases will require additional documentation as to methodology, quality control, and justification of the method used.

11.4.2. QC Requirements for Metals:

11.4.2.1. Sample Preparation

11.4.2.1.1. A blank solution will be prepared with each group of samples to monitor for contamination of reagents, glassware and the laboratory. Detectable blank levels up to ten percent of sample concentration are permissable and are not an indication of an out of control sample.

11.4.2.1.2. A spike solution, prepared from standard reference materials (or laboratory standards that have been confirmed by SRM) will be prepared with each group of samples. (A group is defined as any batch of samples prepared together in the same hood at the same time and with the same reagents). This

solution verifies instrument calibration and monitors the digestion procedure.

11.4.2.1.3. All projects will have at least one sample duplicated and spiked. Projects with large numbers of samples will be duplicated and spiked at the rate of ten percent.

11.4.2.2. Calibration Standards: Commercial single element or multielement standard solutions will be used for the preparation of instrument calibration solutions. These standards will be dated when received and their concentration verified with standard reference materials from NIST, commercial sources where available, or reference samples from NERL-Cincinnati, QA Branch. All commercial standards will undergo additional examination for trace contamination of elements other than the specified element. Mixes of these single element standards are prepared according to the requirements of the instrument being used.

11.4.2.3. Instrument Calibration: All instruments will be calibrated with working standards diluted from commercial stock solutions that have been verified to contain their stated concentration. Instruments will be calibrated to cover the range of concentrations found in the samples or the samples may be diluted to fall within the calibration range. (The following acceptable alternate technique is used in multi-element analyses (ICP or ICP/MS) when an analyte exceeds the high standard: A high level single element standard may be run to demonstrate that the linear calibration range has not been exceeded and that no inter-element interferences are presented by the higher level of the analyte.) An initial calibration check solution should be run as specified in the method. Calibration must be verified during each set of samples at a frequency that will validate all data generated for that set. Reference samples can also be considered as calibration check samples.

11.4.2.4. Instrument Log Books: Will be maintained to record all service and maintenance records.

11.4.2.5. Sample Analysis Records: Log books will be maintained to record preparation of samples to include records of duplicates, spikes, sample numbers, dates, analyst, etc.

11.4.2.6. Log Books: Will be maintained at each instrument to record instrumental conditions and settings during the analysis of samples.

11.4.2.7. Data Records: All raw data from instrumentation will be retained for future reference in either hard copy or electronic storage.

11.4.2.8. QC Data: Data generated from sample duplicates, sample spikes, preparation blanks and SRM preparations will be compared with historical data for that particular sample type and, if found to be within acceptable limits, will be added to the QC data base. If the data are not within acceptable limits, the samples will be re-analyzed or will the data will be flagged. If, after a second analysis, the data still remains outside acceptable limits, data will be flagged and reported.

11.4.2.9. Glassware and Equipment: All glassware/teflon vessels will be placed into a detergent soak immediately after use and must not be allowed to dry while dirty. After thoroughly soaking,

all detergent is removed by rinsing, followed by a 20% nitric acid rinse and finally a thorough rinsing with DI water. Allow to drain on its side and seal with parafilm or a glass stopper before storage in an upright position. Pipets are rinsed immediately after use and placed in a detergent soak until moved to an automatic rinser with DI water. Labware used in ultra-trace analyses may require more rigorous specialize cleaning.

Footnotes:

1 MCAWW - Methods for Chemical Analysis of Water and Wastewater. EPA 600/4-79-020, March 1979. (Revised March 1983), and any future updates.

2 SW846 - Test Method for Evaluating Solid Waste, EPA 1982, and any future updates.

11.5. General Inorganic

11.5.1. Preparation and Analysis: A large portion of the samples that require analysis for nonmetal inorganic constituents (commonly referred to as Nutrients/Classicals) are NPDES Projects. These samples are analyzed according to acceptable methods listed in 40 CFR Part 136. In addition to these methods, other methods are used as appropriate for programs other than NPDES. The current approved versions of all methods are used for regulatory purposes. Guidance methods will be closely adhered to with the possibility of minor changes which do not change the chemistry of the procedure. In any event, all procedures will be fully documented. Table 11.3 lists parameters routinely analyzed in this laboratory and methods of analysis. In addition to these parameters, the lab is capable of or is in the process of developing capability for analysis of dermal corrosion, heat of combustion, and trace level analysis for several parameters.

11.6. QC Requirements for General Inorganic

11.6.1. Sample Preparation

11.6.1.0.1. A blank solution will be prepared with each group of samples to monitor for contamination of reagents, glassware and the laboratory.

11.6.1.0.2. All projects will have at least one sample duplicated and spiked. Projects with large numbers of samples will be duplicated and spiked at the rate of ten percent.

11.6.1.1. Instrument Calibration: All instruments will be calibrated with working standards diluted from commercial stock solutions that have been verified to contain their stated concentration when these commercial solutions are available and use is appropriate. Instruments will be calibrated to cover the range of concentrations found in the samples or the samples may be diluted to fall within the calibration range. An initial calibration check solution should be run as specified in the method. Calibration must be verified during each set of samples at a frequency that will validate all data generated for that set. Reference samples can also be considered as calibration check samples.

11.6.1.2. Instrument Log Books: Will be maintained to record all service and maintenance records.

11.6.1.3. Sample Analysis Records: Log books will be maintained to record preparation of samples to include records of duplicates, spikes, sample numbers, dates, analyst, etc.

11.6.1.4. Log Books: Will be maintained at each instrument to record instrumental conditions and settings during the analysis of samples.

11.6.1.5. Data Records: All raw data from instrumentation will be retained for future reference. Where readings are read directly from an instrument, these readings are considered raw data and are recorded in the appropriate log book.

11.6.1.6. QC Data: Data generated from sample duplicates, sample spikes, preparation blanks and SRM preparations will be compared with historical data for that particular sample type and, if found to be within acceptable limits, will be added to the QC data base.

If the data are not within acceptable limits, the samples will be re-analyzed. If, after a second analysis, the data still remains outside acceptable limits, data will be flagged and reported.

11.6.1.7. Reference materials: Sources outside the lab will be used for reference materials when available. As of November 1997, the following parameters do not have commercial sources known to this lab: color, settlable solids, acidity, and TVSS.

Table 11.3 Nutrients/Classicals Capabilities and Methods

Analyte	**Method Other than 40 CFR Part 136 and Comments**
Acidity	
Alkalinity	
Ammonia	Sedmt-EPA Region 4 method: 1 g sample distilled similarly to aqueous samples
BOD	
Chloride	NPDES Methods and Method 300[1]
Chlorine, Residual	
COD	
Color, ADMI	
Color, Apparent	
Color, Pt Co	
Conductivity	
Cyanide	Sedmt digested according to note in Std Mthds[2] p.4-19 2.b.
Fluoride	
Hardness	Summation of Ca+Mg carbonates (ICP)
Nitrate/Nitrite	Method 353.2M- formal request submitted for Alternate Test Procedure 11/97 Sedmt-EPA Region 4 method: 1 g sample leached into dilute acid
Nitrite	
Oil and Grease	Method 1664- interim approval 4/96, Document # EPA-821-B-94-0046
% Solids or Moisture	
pH	SW-846 Method 9040 or 9045
Phenols	
Phosphorus	Sedmt-EPA Region 4 method: 0.2 g sample digested similarly to aqueous samples
Phosphorus, Ortho	
Solids	
Sulfates	NPDES Methods and Method 300[1]
Sulfides	Sedmt distillation[3] followed by methylene blue color development

TKN	MCAWW 351.2-Cu compound substituted for Hg digestion compound Sedmt-Method 42[4], pp3-22
TOC	
Turbidity	

Footnotes:

1 Methods for the Determination of Inorganic Substances in Environmental Samples. EPA/600/R-93/100, August 1993.

2 Standard Methods for the Examination of Water and Wastewater. American Public Health Association, 18th Edition, 1992.

3 Chemistry Laboratory Manual "Bottom Sediments". Great Lakes Region Committee on Analytical Methods, EPA-FRQA, December 1969.

4 Procedures for Handling and Chemical Analysis of Sediment and Water Samples. USEPA/Corp of Engineers.

12. PERFORMANCE QUALITY CONTROL DATA HANDLING

12.1. All performance quality control data (Section 10, and 11) are transferred from the data books and forms to the appropriate quality control logs or data entry forms. Quality control logs or forms are maintained for inorganic parameters, organics and pesticides, metals and microbiological parameters.

12.2. The following subsections contain the techniques used to measure analytical performance:

12.2.1. Precision Data

12.2.1.1. Organic - Precision is expressed as percent relative standard deviation and is calculated by the formula:

$$\% \text{ RSD} = \frac{S}{\overline{X}} \times 100$$

Where: S = Standard Deviation
$\overline{X}$ = Mean

12.2.1.2. Inorganic - Precision is expressed as relative percent difference and is calculated by the formula:

$$\% \text{ RPD} = \frac{D}{\overline{X}} \times 100$$

Where: D = Difference between measurements
$\overline{X}$ = Mean

12.2.2. The estimated standard deviation may be calculated by the following equations fpr duplicate analysis:

$$S = \pm (X_1 - X_2)\ 0.89$$

Where: X_1 and X_2 = individual observations.

For replicate analysis (any number >2)

$$S = \sqrt{\frac{+n \sum X^2 - (\sum X)^2}{n(n-1)}}$$

NOTE: Automatic calculators may be used to determine S if this formula is used.

Where: X = individual observations.
n = number of observations.

Do not use this formula for n=2.

12.3. Accuracy Data

12.3.1. Accuracy is expressed as percent recovery and calculated by the formula:

$$\% \text{ Recovery} = \frac{Z - X}{T} (100)$$

Where: X = concentration in unspiked sample.
Z = concentration in spiked sample.
T - True concentration of spike added.

12.4. Annual Analytical Performance Summary

12.4.1. At the end of each fiscal year, a summary report of the Branch's analytical performance is prepared. Contained in this report are: the precision data (average percent RSD or RPD, upper warning and control limits), and accuracy data (average total percent recovery of spiked samples, AQC reference samples, and performance audit samples where possible). This summary will contain all parameters for which adequate quality control data have been generated during the year.

12.4.2. Participation in EPA Performance Evaluation Studies.

12.4.2.1. The Branch will participate in announced EPA performance Evaluation Studies. Performance on these studies further indicates the effectiveness of the laboratory's day-to-day quality control procedure.

13. ANALYTICAL CORRECTIVE ACTIONS

13.1. Corrective action will be taken at any time during the analytical process when deemed necessary based on analyst judgement or when quality control data indicate a need for action. Generally, corrective action will be triggered by such things as: poor analysis replication, poor recovery, instrument calibration problems, blank contamination, etc. (See previous sections for specifics).

13.2. Corrective actions will include, but not necessarily be limited to: reanalysis, calculation checks, instrument recalibration, preparation of new standards/blanks, re-extraction/digestion, dilution, application of another analysis method, additional analysts training, etc. Most frequently, these corrective actions will be initiated by the analyst at the time of analysis. However, some corrective actions are initiated subsequent to analysis based on evaluations performed by quality assurance or laboratory management personnel.

13.3. All data corrective actions will be noted on the appropriate log, chromatogram, strip chart or data report.

14. DATA QUALITY OBJECTIVES

14.1. During the planning phase of a project requiring laboratory support, the data user must establish the quality of data required from the investigation. Such statements of data quality are known as Data Quality Objectives (DQO's). The DQO's are qualitative and quantitative statements of the quality of data required to support specific decisions or regulatory actions. The laboratory is responsible for producing data of known quality and consistent with that prescribed in the DQO.

14.2. The laboratory will select analytical methods, instruments, parameter detection limits, etc. which are capable of producing data of the quality required by the DQO. The quality of a data set is defined in terms of: precision, accuracy, representativeness, completeness and comparability. The significance of each of these measures differs according to their applicability to the laboratory and to a particular data set. A brief explanation of the above measures are as follows:

14.2.1. Precision and accuracy. These are quantitative measures that characterize the amount of variability and bias inherent in a given data set. Precision refers to the level of agreement among repeated measurements of the same characteristic. Accuracy refers to the difference between an estimate based on the data and the true value of the parameter being estimated (See Section 13).

14.2.2. Representativeness. Refers to the degree to which the data collected accurately reflect the population, group or medium being sampled.

14.2.3. Completeness. Refers to the amount of data that is successfully collected with respect to that amount intended in the study design.

14.2.4. Comparability. Refers to the ability to compare data from different sources with a degree of confidence.

Attachment 5

Battelle Quality Assurance Management Plan

Battelle Marine Sciences Laboratory

Quality Assurance Management Plan

January 2000

Battelle
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Marine Sciences Laboratory

Quality Assurance Management Plan

Introduction

The purpose of the Battelle Marine Sciences Laboratory (MSL) Quality Assurance Management Plan is to describe the Quality Program implemented at the facility. This plan summarizes the elements of the quality assurance program and discusses the quality control activities routinely used for MSL work. The objective of the Quality Program is to obtain accurate and precise data consistent with project objectives. The MSL QA Program has evolved over time to meet client needs but its roots are from the U.S. Environmental Protection Agency's Quality Assurance Management Staff's requirements specified in QAMS 005/80 which has been updated to the "EPA requirements for Quality Management Plans", EPA QA/R-2. While this plan sets forth Quality Program requirements, project plans and work plans are used to define project-specific customer requirements.

This QA Management Plan is divided into three volumes:

Volume 1 MSL administrative and management requirements
Volume 2 Marine and Environmental Chemistry QC and technical requirements
Volume 3 Marine Ecological Processes QC and technical requirements

Implementation of the policies and requirements specified in the MSL Quality Assurance Management Plan and the associated MSL procedures will provide defensible and credible data enhancing the quality of MSL products and services.

_____
R.M. Ecker
MSL Manager

Battelle
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Marine Sciences Laboratory

QUALITY ASSURANCE MANAGEMENT PLAN

VOLUME 1

May 2000

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Battelle Marine Sciences Laboratory
QUALITY ASSURANCE MANAGEMENT PLAN
VOLUME 1

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Battelle Marine Sciences Laboratory
QUALITY ASSURANCE MANAGEMENT PLAN
VOLUME 1

Contents

1.0 INTRODUCTION

1.1 QUALITY ASSURANCE MANAGEMENT PLAN

The purpose of this Quality Assurance (QA) Management Plan (QAMP) is to describe Battelle's QA Program as implemented within the Marine Science Laboratory (MSL). This QAMP summarizes elements of quality assurance and the quality control (QC) activities routinely used to perform work by collecting accurate and precise data consistent with project objectives. This QAMP has been designed to meet the requirements of many of the MSL's clients and addresses elements of the Environmental Protection Agency's (EPA's) Quality Assurance Management Staff's (QAM's) "EPA Requirements for Quality Management Plans", EPA QA/R-2, the Navy QA Program and the requirements for the National Environmental Laboratory Accreditation Program (NELAP). While this plan establishes the quality assurance program requirements, Quality Assurance Project Plans (QAPjPs), sample analysis plans and "kits" assembled at the time of sample receipt, are used to define any project specific quality requirements not contained in this plan.

A copy of this QAMP is available to each staff member, who is expected to be aware of, and perform his or her assignments in accordance with, the QA requirements stated in this document. The signature page at the front of the QAMP indicates MSL management's review, consensus and approval.

To ensure that the QAMP remains current, it is reviewed annually and updated as needed. If major changes are needed, the entire document is re-issued; if only minor changes are needed, only the affected sections are updated. The document control header in the upper right hand corner of each page signifies that the document is controlled. Upon revision of the document (or selected sections), the effective date is updated and the revision number incremented by one. The revisions are reviewed and approved as described below and distributed to the staff. The QAMP will be issued by the MSL Quality Assurance Officer.

1.2 POLICY STATEMENT

The commitment of Battelle to quality assurance is reflected in the following statements from the Battelle Pacific Northwest National Laboratory (PNNL) policies:

- *We are committed to provide services and products of the highest quality consistent with the needs, expectations, and resources of our customers.*

- *We are committed to continuously improving our processes, systems and capabilities so that we can increase the technology-based value of products delivered to our customers.*

In accordance with these principles, the MSL has developed a QA Program to assure that all activities affecting the quality of data or products produced for clients are thoroughly planned and coordinated by project teams. The policy of the MSL is to ensure that all data generated, processed, or used in completing each task are scientifically valid, legally defensible, and of known and acceptable quality. As part of PNNL, the MSL is committed to the corporate policy of providing quality products and services and committed to their clients to ensure that sampling and analytical procedures are properly executed, sample integrity is not compromised, all QC procedures are implemented and recorded, and only valid data is reported. To attain this goal, the MSL has implemented the QA Program summarized in section 1.3.

1.3 OVERVIEW OF PROGRAM

The objective of the MSL's QA Program is to provide clients with quality products and services. A critical element in providing quality products is the maintenance of a QA Program that provides for conducting activities in a planned and controlled manner, thereby permitting the verification of quality performance. The consistent delivery of products of acceptable and documented quality requires commitment and adherence to QA and QC principles and procedures throughout the performance of each task. A commitment to quality is an integral part of every employee's job at the MSL. In addition, the MSL recognizes that formal functions are necessary to assure Battelle Management and it's clients that the work performed and the technical products produced meet client needs and conform with their specific data quality objectives and requirements. These formal functions are QA and QC. QA includes all systems designed to assure MSL management and the client that data were collected, processed, and interpreted in accordance with the requirements of the planning documents; that all aspects of work performance, including data generation and analysis are adequately documented; and that all data are accurate and fully traceable. For this system to be effective, each individual must understand his or her role in implementing the program. The responsibilities, authorities, and accountabilities with the MSL QA Program are defined in Section 2.0. QC functions include all activities that are designed to assess or control precision and accuracy of measurements and data. QC functions involve performance of procedures necessary to attain and document the prescribed standards of performance in all measurement and data collection processes.

One of the first steps of the planning process is the development of data quality objectives (DQOs) (refer to Section 5.4). DQOs provide the criteria needed to design a study, and once determined, become part of the project planning documents (Section 5.0). In addition to the objectives, the project planning documents define the methods, personnel, schedule, and deliverables associated with the project. The project planning documents are supported by standard operating procedures (SOPs), which are detailed documents that describe the approved methods for instrument calibration, data collection, processing, reduction and reporting (Section 6.0). Planning also involves ensuring that staff members are fully qualified and trained to perform their responsibilities (Section 2.0) and that facilities and equipment are adequate and appropriate for their use (Section 3.0). Procurement of qualified subcontractors (Section 4.0) is also a key consideration during the project planning stage.

A major component of the work performed by the MSL involves the collection and analysis of samples for chemical, biological, and physical parameters. A sample control system is essential to ensure that the history of each sample is documented and verifiable (Section 8.0). QC activities are implemented during the performance of the work to measure and control the quality of the product (Section 9.0). Additional methods of quality assessment are data validation and document reviews (Section 10.0) and QA verification activities (Section 11.0). Deficiencies noted during the assessment process are reported to management who take the necessary remedial action to bring the system into compliance (Section 11.3). Quality improvement processes are implemented to ensure that problems identified are solved, and do not recur (Section 12.0).

1.4 SCOPE

Battelle MSL comprises three technical groups: Marine and Environmental Chemistry, Ecotoxicology and Risk Assessment, and Ecosystem Processes and Restoration. These groups provide a wide range of contract research services related to environmental programs, primarily related to the marine environment. The QA program defined in this document applies to all projects performed by Battelle MSL, both for external clients and other components of Battelle.

The services and products provided by Battelle MSL are used by our clients for a variety of purposes, including defining baseline environmental conditions, assessing environmental effects, as evidence in litigation, and as the basis for regulatory decisions. The diversity of projects demands a flexible QA program that is cost-effective, yet meets the needs of the client and the standards of Battelle MSL. This document describes the framework of Battelle MSL's QA Program and defines the minimum standards that apply to all projects. This QAMP is supplemented by SOPs and project planning documents (i.e.,

QAPjPs, work plans, toxicity testing plans). MSL procedures provide detailed descriptions of QA activities, as well as the QC requirements for routine technical procedures. Project planning documents define the specific quality objectives for projects and describe the procedures necessary to attain those objectives.

This QA Management Plan is divided into three volumes:

Volume 1	MSL administrative and management requirements
Volume 2	Marine and Environmental Chemistry QC and technical requirements
Volume 3	Marine Ecological Processes QC and technical requirements

2.0 ORGANIZATION AND PERSONNEL

This section describes the organization of the MSL and defines the associated responsibilities, authorities, and accountabilities.

2.1 ORGANIZATION

QA at MSL is an interdisciplinary line management function. MSL's responsibility assignments are that 1) quality is achieved and maintained by those who have been assigned responsibility for performing work, and 2) quality achievement is verified by those not directly responsible for performing the work. The organization of Battelle MSL is illustrated in Figure 2.1.

The QA Officer has the authority and organizational freedom to identify quality problems, to initiate, recommend or provide solutions, and to verify implementation. All verification activity reports are made available to line and project management. Line and project management are responsible for identifying and assuring implementation of corrective action to all deficiencies.

Any MSL employee can initiate a stop work on the basis of a safety or quality concern. The immediate supervisor shall be immediately notified of the concern and the shall initiate investigative activities or initiate implementation of corrective actions.

2.2 RESPONSIBILITIES

Quality Assurance Officer

The QA Officer provides overall direction to, and management of, all Battelle MSL QA activities. Specific responsibilities include

- Developing the QAMP and updating it, as needed, to reflect Battelle MSL policies and procedures
- Developing project budgets for QA activities and reviewing proposals for adequate and appropriate QA requirements
- Assisting project managers in defining the QA/QC procedures to be used during a project
- Administering a training program related to QA policies and procedures
- Scheduling, planning, and conducting verification activities (assessments, data audits) of projects and facilities
- Preparing written reports summarizing the results of verification activities for distribution to project managers and MSL management
- Participating in, or coordinating, inspections and audits conducted by clients and regulatory agencies
- Preparing periodic status reports of QA activities and verification results for MSL management
- Reviewing and approving technical procedures, project planning documents, and reports
- Preparing SOPs of QA activities
- Scheduling annual SOP review, distributing SOPs, maintaining an SOP log, and archiving historical SOPs

- Conducting training sessions on QA functions and activities.

The MSL QA Officer is part of the Battelle Process Quality Department located in Richland, WA and reports to the supervisor of that Department. The MSL QA Officer does not report to anyone at the Sequim facility, thereby attaining independence.

Marine Sciences Laboratory Manager

The MSL Manager provides overall management of the MSL and has responsibility for all the laboratory's operations.

Technical Group Leader

Technical Group Leaders are responsible for ensuring the quality of products produced within their group. Specific responsibilities include

- Ensuring that all activities related to meeting the data quality objectives defined in the MSL QAMP are being performed
- Providing sufficient resources, including both time and staff, to meet project and laboratory objectives
- Ensuring that all products produced from their group are reviewed and approved according to Battelle MSL policy before being released
- Ensuring that all projects have adequate project planning documents prior to initiation
- Promptly and appropriately correcting deficiencies noted during QA verification activities
- Ensuring that any SOPs that are required within the group are written, reviewed, and revised accordingly
- Identifying and addressing training needs

Project Manager

The Project Manager has overall responsibility for the management of project activities. Specific responsibilities include

- Administering and supervising all project tasks to ensure that all project objectives are met, on time, within budget, and of appropriate quality
- Preparing project planning documents and ensuring that the plans are reviewed and approved according to MSL policies
- Ensuring that the project objectives are communicated to project personnel and that project personnel are trained to perform any procedures unique to the project
- Reviewing all project reports and deliverables
- Addressing project-specific deficiencies that are identified during verification activities

Laboratory Supervisor

Laboratory supervisors provide the day-to-day oversight activities of the laboratory. Specific responsibilities include

- Organizing equipment, staff, and materials
- Providing technical direction in the performance of tasks
- Resolving day-to-day problems, including instrument operation, calibration and use concerns and ES&H issues
- Reviewing records and data associated with the tasks under their direction for accuracy, validity and completeness
- Communicating with the project manager and advising him/her of problems, progress and needs

Staff Member

Each staff member has the following responsibilities:

- Performing work to the specified procedures in conformance with the project planning documents, applicable SOPs, and Battelle MSL policies, including ethical and legal responsibilities
- Identifying safety and quality concerns and informing the appropriate supervisor
- Communicating to the appropriate manager any deviation from established procedures or issues requiring corrective action
- Defining appropriate QA requirements for purchased items and services

Contracts/Business Representative

- Providing acquisition, contracts, and related business support to the MSL that assists in meeting the strategic goals and objectives of the MSL and its clients
- Assisting staff in ensuring that the proposal preparation process meets the goals of the MSL
- Ensuring that QA requirements are specified in procurement documentation

ES&H Representative

- Overseeing and implementing core ES&H support services (Environmental Compliance, Safety, Health, and Training) to ensure laboratories and staff compliance with regulations
- Ensuring and assessing that proper waste handling, safety measures, and training are being performed by and for staff in conjunction with work performed at the MSL.

Environmental and Safety Engineer and Radiation Safety Officer

- Managing laboratory water and ventilation supply and discharge systems in compliance with environmental and health regulations and in support of lab missions

- Managing lab radiation safety program in compliance with regulations and in support of lab missions

2.3 PERSONNEL QUALIFICATIONS AND EXPERIENCE

The quality of MSL products depend, in part, on the competence and expertise of the staff involved. It is MSL policy that all individuals involved in the conduct or supervision of projects (including laboratory technicians, field personnel, toxicologists, analysts, data-processing personnel, supervisors, project managers, and QA staff) must have the necessary education, training, or experience to perform their assigned tasks. This objective is achieved by hiring personnel with the appropriate qualifications and providing continual training and opportunities for professional growth.

A summary of experience and qualifications is documented on the Qualification and Training form and placed in the individual's training file. In addition, a list of personnel and an associated biosketch is maintained for each employee and are shown in Appendix B of this QAMP. Biosketches are revised as needed.

2.3.1 Responsibilities

The MSL Manager is ultimately responsible for ensuring that appropriately qualified personnel are hired, resources for training are allocated, and that appropriate training and professional growth are provided, and records of training are maintained. Within a group, these responsibilities rest with the Technical Group Leader.

Each individual's supervisor is responsible for identifying specific training needs, ensuring that the employee receives the necessary training to perform his/her assigned tasks, and assigning personnel to project tasks in accordance with their experience and skill.

Each individual is responsible for completing required training and submitting training records and certificates to their supervisor, for updating their biosketch as needed, and for identifying and completing additional training that may be required, but was not assigned.

2.3.2 Training

Specific training requirements are contained in procedure, MSL-A-006, *Marine Sciences Laboratory Training*. Training begins the first day of service and continues throughout a staff member's term of employment. Introductory seminars on Battelle policies and organization, QA, ethical and legal responsibilities, and Environment, Safety, and Health (ES&H) are presented during an orientation program. Technical training begins prior to work being performed, through reviews of procedural documents and demonstrations by experienced personnel. Introductory courses are augmented by general and project-specific training that is conducted periodically. All personnel assigned to projects receive training to acquire the necessary skills to perform their responsibilities. Technical training is accomplished through a variety of approaches, including

- Direct hands-on training. Training is accomplished by reviewing procedural documents (e.g., SOPs, project work plans), proficiency testing, and supervision by experienced personnel. Each MSL procedure includes the training requirements associated with that procedure, including any proficiency tests.

- Project kickoff meetings. Kickoff meetings ensure that all project personnel are aware of the project objectives and the methods to be used to accomplish the objectives. This also includes field safety training at the beginning of each sampling period.

- In-house technical seminars. These seminars, which are available to all personnel, are conducted by MSL staff or guest speakers and generally cover current projects or related research programs.

- Continuous education through a tuition reimbursement program.

- Attendance of professional meetings and outside workshops.

ES&H training is provided to each employee who works in the laboratory or whose responsibilities expose them to potential risk or hazard. Training includes chemical, physical, biological, radiological, and mechanical hazards. Training is conducted and coordinated by the MSL ES&H Representative.

QA training is conducted by the QA Officer. Briefings and one-on-one training on general or project-specific topics related to QA (e.g., sample custody, data validation, Good Laboratory Practices [GLPs]) are conducted as needed. A Battelle on-line training module titled, Quality Program Training (course #1366) is available. The employee completes the training activity and prints a training completion form that must be signed and submitted to the training department to obtain credit. The signed form is evidence that the employee has read; acknowledges, and understands their personal QA responsibilities.

2.3.3 Documentation

Records of training and qualifications include the following:

- MSL training assignments
- Certificates attesting to the attendance or completion of external courses
- Resumes and biosketches
- ES&H Records

Original records of training and qualifications are maintained by the Technical Group Managers. Copies of technical training records are forwarded to the QA Officer.

2.3.4 Improper, Unethical or illegal Actions

Training courses in ethical and legal responsibilities including the potential punishments & penalties for violations are provided by Battelle via on-line computer training. The applicable course title and number are Battelle Standard of Business Ethics and Conduct, course number 1062. The employee completes the training activity and must score 80% or better. The module allows the successful employee to print a training completion form that must be signed and submitted to the training department to obtain credit. The signed form is evidence that the employee has read; acknowledges, and understands their personal and legal responsibilities including potential punishments & penalties for violations; and provides the required training documentation.

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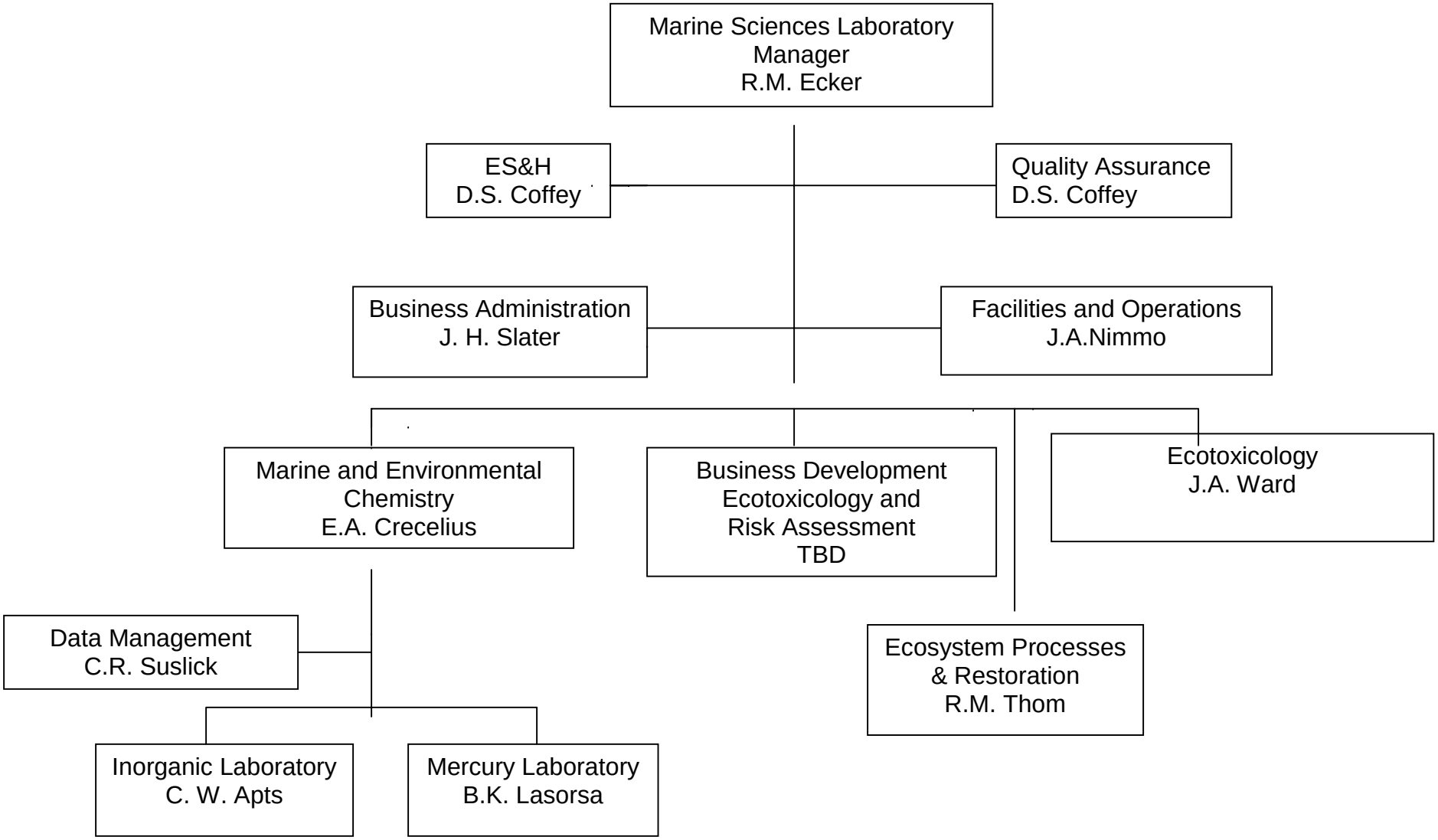
Marine Sciences Laboratory



3.0 FACILITIES AND EQUIPMENT

Pacific Northwest National Laboratory is a research and development laboratory operated by Battelle Memorial Institute, Pacific Northwest Division (Battelle), a non-profit organization. Among the entities that operate Department of Energy (DOE) National Laboratories, Battelle is the only entity that holds a Use Permit contract with the U. S. Department of Energy in addition to its operating contract. Battelle is the only DOE Management and Operating Contractor that actually owns a significant amount of our own land and buildings, rather than occupying exclusively government-owned property. The method of obtaining contract research business under the Use Permit is to prepare and submit technical or research proposals to potential clients. Battelle MSL is part of the PNNL, operated for the U.S. DOE by Battelle Memorial Institute under Contract DE-AC06-76RLO.

3.1 FACILITIES

Battelle MSL is located in Sequim, WA on 125 acres fronting Sequim Bay, and consists of 40,000 square feet of laboratory and office space housed in two buildings -- a beach facility containing bioassay laboratories, and an upland facility for analytical laboratories and office space. The facilities support approximately 40 scientists and support staff and about 15 on-site contractors and graduate research students.

Biological Laboratories

Two bioassay laboratories provide 5,300 sq ft of space for studies requiring flowing seawater. Four separate distribution systems supply seawater and/or freshwater to the laboratories. High quality, Class AA seawater is obtained from Sequim Bay through an all-PVC system with two independent intakes. A redundant system of three pumps provides a continuous supply of filtered and unfiltered seawater to experimental tanks. An emergency diesel generator ensures continuous seawater supply and other essential services in the event of electrical failure. Furthermore, the system is checked daily every 2 to 4 hours while experiments are in progress. A 16,000-gal reserve tank provides filtered seawater to the wet laboratories for up to 18 hours in the event of failure of all three pumps. Seawater at ambient temperature (9-11°C) can be provided at a rate of 250 GPM, and up to 20 GPM of seawater can be supplied at temperatures ranging from 0-38°C through a chiller and gas-fired heat exchange system. Fresh water is also supplied to the laboratories from uncontaminated groundwater reservoirs.

Holding and breeding facilities for a variety of fish, shellfish, and freshwater, estuarine, and marine plants are provided in these laboratories and in outdoor tanks. All water leaving the seawater laboratory is passed through a treatment system to ensure no impact is made on the receiving environment.

Analytical Chemistry Laboratories

Analytical laboratories in the beach facility consist of two general purpose bioassay preparation laboratories occupying approximately 1,000 sq ft. These laboratories provide space and equipment for conducting measurements supporting the bioassays, such as water quality parameters, pH, dissolved oxygen, temperature, and salinity. They also provide work stations for microscopy and space for sample storage and preservation.

Analytical laboratories in the upland facility consist of two banks of five fully-equipped chemistry laboratories, each occupying 600 sq ft. The chemistry laboratories are equipped with state-of-the-art instrumentation and supplies, including an array of mass spectrometers, chromatographs, analytical balances, rotary evaporators, freeze-driers, microwave digestion systems, sonicators, freezers, refrigerators, and drying ovens. The following are some of the specialized purposes that these laboratories serve:

General Laboratory for Rceipt of Smples and Peparation for Analysis:

General Organic Chemistry Laboratories for preparation of sample extracts for gas chromatography and mass spectroscopy, and analysis for physical properties of sediment. A high-performance liquid chromatography (HPLC) system, with variable-wavelength UV detector, fluorescence detector, auto injector, fraction collector, integrator, and data reduction system is available for specialized sample preparation.

Analytical Services

Gas Chromatography and Mass Spectroscopy Laboratory for conducting specialized cleanup procedures and analyses of oil and grease, polynuclear aromatic hydrocarbons, phenols, polychlorinated biphenyls by congener or Aroclor, pesticides, and organotin compounds. The laboratory contains three microprocessor-controlled high-resolution gas chromatographs: two Hewlett-Packard Model 5890A, and one Model 5890 Series 2 equipped with a mass spectrometer. Detectors available include flame-ionization detection (FID), flame photometric detection (FPD), and electron-capture detection (ECD). The laboratory also contains a VG Fison Model TRIO 1000 GC/LC/Mass Spectrometer.

Metals Chemistry Laboratory for preparation of samples for metals analyses and determination of basic chemical and physical properties of samples such as pH, salinity, grain size, total volatile solids, and percent dry weight, as well as instrumentation for counting radioisotopes used in age-dating sediments.

Inorganics Laboratory for metals analyses using a Perkin Elmer Elan 5000 Inductively Coupled Plasma Mass Spectrometer and two atomic absorption spectrophotometers: a Perkin-Elmer Model 5000 and a Perkin-Elmer 3030 equipped with a background corrector, graphite furnace or flame capability, autosampler, and printer and rapid-response recorder.

Specialized Mercury Analysis Laboratory for ultra-trace level (picogram/liter) analysis of mercury in water samples, and parts per trillion analysis of total and methylmercury in water, tissue, and sediment using analytical methods developed at Battelle MSL.

Specialized Sulfide Analysis Laboratory for trace level determination of inorganic sulfur compounds in water and sediment using gas generation, purge and trap, and gas chromatography coupled with flame photometric or photoionization detection.

Physical Oceanography Laboratory for gas exchange research, contains a whitecap simulator with two large tanks, a Dantec laser-doppler velocity and particle analyzer, electronics shop, computer-controlled data loggers, and extensive test equipment and chemical instrumentation.

Computer Facilities

Battelle MSL staff use PC, Macintosh, and UNIX-based computer systems connected via a local area network. The systems are linked to other on- and offsite hardware composed of some 6700 workstations and servers, minicomputers, database and file repositories, Web servers, and supercomputer facilities.

Battelle MSL has access to the numerous online databases accessible through Dialog Information Services. Commercial databases such as BIOSIS (Biological Abstracts), Chemical Abstracts, Oceanic Abstracts, Enviroline, and many others can all be accessed quickly by computer at MSL. Other databases such as Aquatic Sciences and Fisheries Abstracts, National Technical Information Service, ad ToxChem (Toxicology and Chemistry) are accessible through the University of Washington libraries. Through such access to information, literature searches can be conducted efficiently at MSL.

Safety and Security

The safety of MSL employees is of paramount importance. Therefore, the MSL buildings are equipped with structural safety features (e.g., fire doors and extinguishers, emergency lighting systems), alarm systems which serve to alert the staff in the event of emergencies (e.g., fire/smoke alarm), and engineering controls designed to minimize exposure to potential hazards (e.g., fume hoods).

The security of the facility is an important consideration because of the type of work performed by the MSL. Access to the MSL grounds and buildings is controlled through a card-access and lock and key system. During business hours, all visitors must enter through the main lobby and sign in with the receptionist. Selected areas within the facility are secured at all times and their access limited to authorized personnel. These areas include the walk-in cold room used for sample storage, the records storage area, the solvent shed, and the GLP data archives. Procedure, MSL-A-011, *MSL Access Control*, describes the process in detail.

Computer security is a function of the PNNL network and is administered from facilities located in Richland, WA. Staff have individual responsibility to back up files, instruments and data bases at regularly scheduled intervals which are described in the MSL procedure, MSL-D-004, *Data Reporting, Reduction, Back Up, and Archiving.*

3.2 EQUIPMENT

The quality of MSL products is directly related to the validity of the data produced. To produce valid data, equipment must be properly operated, maintained, and calibrated. Preventive maintenance and primary maintenance is provided through the Battelle Facilities and Operations staff located in Sequim, but located organizationally in Richland, WA. The MSL maintains a wide variety of equipment related to the collection and analysis of chemical, biological, and physical oceanographic parameters. To support the generation of data of known and acceptable quality, the following general guidelines are implemented

- The appropriate and necessary equipment, instruments, and supplies must be available in adequate quantities to perform the proposed work. Spare parts for critical components are maintained to minimize downtime.

- Measuring and testing equipment is properly handled and stored to maintain accuracy.

- All equipment involved in the collection and analysis of environmental data is operated, maintained, and calibrated according to approved procedures and specified schedules.

- Equipment is serviced regularly by qualified individuals, either trained in-house personnel or through service contracts with the manufacturer or an authorized representative. For example, balances are cleaned and calibrated by a Battelle Preferred-Supplier, and analytical instruments have service contracts with manufacturers such as Perkin-Elmer. Most support equipment (e.g., ovens, refrigerators, freezers, hoods) servicing is done internally by Battelle Facilities and Operations staff. When problems arise that can not be corrected internally, external contractors or manufacturer's representatives are contacted.

- Written records of all instrument maintenance, calibration, testing, and inspection are maintained. Maintenance records contain a description of the operation or problem, the remedial action taken (if necessary), date, and the person responsible.

- When equipment or instrument maintenance is required, equipment is monitored by facilities to ensure correct operation. Analytical instrument operation after maintenance is monitored by the responsible analyst by running a calibration curve and assessing results of standard reference materials (SRMs) .

- All calibrated equipment is suitably marked to indicate calibration status.

- Written direction on equipment operation (e.g., operating manual, manufacturer's instruction, and SOPs) are maintained with the equipment and are available to personnel using the equipment.

- All balances are calibrated annually or semi-annually by an approved metrology laboratory. All balances are checked daily prior to use with certified weights by a designated laboratory technician. These performance checks are documented in balance logbooks.

- All cold-storage facilities are monitored daily with a calibrated or certified thermometer. Acceptable temperature ranges for refrigerators is 4 ±2 ^{o}C and for freezers is -20 ±10 ^{o}C. The ultra-low freezer is maintained at –68 $\pm$ 5 ^{o}C.

Specific equipment lists for the Marine and Environmental Chemistry Group are contained in Volume 2.

4.0 PROCUREMENT AND CONTROL

4.1 MATERIAL PROCUREMENT AND CONTROL

Examples of items that generally have a significant influence on the quality of MSL work, and therefore generally need defined quality requirements are the following:

- Standards and reference materials
- Reagents, chemicals and solutions
- Animals and feed
- Computer software and hardware, and
- Some miscellaneous items such as designed equipment

Procurement activities at MSL are guided by procedure, MSL-A-012, *Procurement* , which should be consulted to determine appropriate QA requirements before initiating procurement actions.

Miscellaneous Procurements

Miscellaneous procurements of items that have a significant influence on the quality of MSL work, generally need defined quality requirements. When the purchaser does not know if quality requirements should be specified, the rule is to request the MSL Quality Assurance Officer or representative to make this determination and document it as a note, letter or email.

Material Receiving Inspection

When the MSL orders materials that require certification (i.e., standard or certified reference materials (SRMs, CRMs), standards, precleaned sample containers, etc.), a request for certifications shall be made on the purchase order. Standards and reference materials must be traceable to the National Institute of Standards and Technology (NIST; formerly the National Bureau of Standards or NBS) or other nationally-recognized standard (e.g., American Society for Testing Materials [ASTM]). The traceability must be documented by a certificate or label that verifies this link. The traceability documentation must be received and found to be acceptable by MSL staff before material use. Acceptance of these items and certifications shall consist of verifying that the lot numbers on the certifications and the jar and/or boxes are the same. Approval shall be indicated by a signature and date of signature on the certificate. Pending receipt of this documentation and its acceptance, affected material must be segregated to prevent inadvertent use. Certifications received will be maintained in the Project or Central files.

Reagent and Standard Inventory Procedures

The procurement of reagents, chemicals and solution should include requirements for shipping stocked inventory materials with the longest period to the expiration date (i.e., the freshest material) possible, with lot numbers specified. In some cases where extremely high purity material is requested, a request for purity documentation may be necessary.

Procurement procedures should require that a manufacturer's recommended expiration date be provided with every standard material. If manufacturer's expiration dates are not provided, the laboratory must assign an appropriate expiration date, based on professional judgement and in consideration of the shelf life for similar materials at similar concentrations. The technical basis for each such determination must be documented in the project file by the responsible analyst, and approved by the Project Manager.

MSL follows the Pacific Northwest National Laboratory's (PNNL's) Standards-Based Management System (SBMS) requirements for logging in reagents, chemicals and solutions into the associated Chemical Management System (CMS). This system provides the PNNL Laboratory with policies and

procedures regarding tracking and inventory, storage, and disposal of completed samples and analytical wastes as well as chemical use and disposal. The CMS is used to provide an up-to-date inventory to facilitate emergency response, monitor the location of various classes of materials and identify situations where acceptable limits for the building/facility determined by the assigned chemical hazard group and fire zone might be exceeded before a violation occurs. An assigned Sample Inventory Coordinator provides bar codes for each chemical item when it is received and assigns it to a location. The item then is tracked in the CMS until disposal. The system is also used to ensure that facility limits based on the chemical hazard group and the assigned fire zone determination are not exceeded.

Organisms and Feed

The procurement of organisms and feed for bioassays should include requirements for chain of custody of animals during shipping and documentation of any available feed analyses, feed storage recommendations, and expiration dates so that feed quality can be monitored. Animal shippers should be requested to document conditions of animals and environmental parameters (temperature) at the time of shipping for comparison with conditions encountered at the time of receipt. In some cases, it might be important to include QA requirements for a minimum/maximum thermometer or temperature strip in the cooler at the time of shipping. Requirements regarding common carriers, Saturday delivery acceptability and locations, and other details might also be specified in QA requirements documents.

Computer Software and Hardware

QA requirements for the procurement of hardware must ensure that hardware is compliant for periods where clock or time information settings provided by the manufacturer might affect future hardware operation. QA requirements for the procurement of software should follow some general guidelines:

- Commercial software that has been developed under the manufacturer's QA Program and fully tested before release is preferable to other types of software developed under lesser or no QA Program
- Documents necessary to demonstrate that software was developed using a Life Cycle approach such as User's Manuals shall be requested when software is ordered.
- Licenses that come with the software and original documentation should be requested, obtained and protected.
- Software that requires a signed site license agreement can only be purchased by individuals with appropriate delegations.
- Hardware/Software that exceeds $5,000 can only be purchased with appropriate management approvals.
- Software procured as a product under a subcontract must specify detailed QA requirements for software development and use, and provide plans for testing, verification and validation tests and include acceptance criteria.

Solvent Storage Policies

Solvents used in the laboratory are in containers of 4 liters or less. On receipt they are logged in, bar-coded, and tracked, as are all chemicals. No more than a working day's supply of flammable or combustible solvents is permitted out of flammable storage in a laboratory; at the end of the day, these materials must be returned to flammable storage. Large flammable storage cabinets, located in an area separate from the building, are used for storage of solvents that exceed the lab's storage capacity.

Waste Disposal

Hazardous wastes at the MSL are managed in accordance with Washington State Department of Ecology's Chapter 173-303 WAC, "Dangerous Waste Regulations." The MSL is a "less the 90-day storage" facility and a medium-quantity generator and, as such, fulfills all the requirements outlined in the

regulation regarding proper labeling, designating, inspections, and timely disposal of hazardous waste. Staff who generate/handle waste are trained annually in waste management procedures.

Section 1.3 of Volumes 2 and 3 of this QAMP addresses specific requirements for sample disposal.

4.2 SUBCONTRACTORS

MSL policy for sending to work to subcontractors is that MSL routinely does not subcontract analyses that can be performed in house. In some situations this could occur, if the capacity of the laboratories is not adequate to meet a project deadline. MSL does contract project analyses when this approach is a project-specific requirement. All staff are expected to clearly and completely specify appropriate requirements for purchased goods and services consistent with project needs. This is done by developing a statement of work that includes, number of samples, sample matrix, required procedure, applicable holding times, quality control sample requirements and project data quality objectives, and data deliverables. Materials, equipment, and services shall be delivered at reasonable costs, with delivery times consistent with the specific project and business needs of the Laboratory. Costs and commitments will be recorded in a timely and accurate manner. Battelle MSL is ultimately responsible for the quality of work performed by its subcontractors. Therefore, procedures have been established to ensure that subcontractors involved in environmental data collection programs are qualified to perform their responsibilities, that project objectives, methods, and responsibilities are clearly defined and communicated, and the work performed is monitored to assess conformance to the project specifications.

The approved supplier list maintained by the WA DOE is one source for identifying appropriate subcontractors to provide performance evaluation samples (refer to Section 9.2). PNNL also maintains a list of approved suppliers for analyses, and this list is used as a starting point to define subcontractors. If the subcontractor is not on an approved list, then the subcontractor must have demonstrated or provided proof of the necessary technical capabilities, facilities, resources, and experience to perform the specified tasks. The contract specifies the costs, technical services, QA requirements, deliverables, and schedule of performance. The contract must include a Statement of Work (SOW) in sufficient detail so that the scope of work, methods, quality assurance requirements, responsibilities, deliverables, and due date are clearly understood by the MSL and the subcontractor.

In terms of QA, each subcontractor must have a written description of its QA program, that defines the policies, procedures, and responsibilities implemented to ensure the quality of the data or other products provided to the MSL. More detail may be found in procedure, MSL-A-012, Procurement. In addition, it is expected that the following standards will be met.

- If appropriate, the subcontractor must have an internal QC program. Analysis of internal QC samples (the type and frequency to be specified in the SOW) must be performed in conjunction with analysis of MSL samples and the results reported with the sample data.

- Written descriptions of all procedures involving environmental data collection and generation must be available and implemented.

- Equipment used to generate data must be maintained, calibrated, and operated according to written procedures.
- Subcontractor personnel must be properly trained and qualified.

- Adequate procedures for record management and reviewing documents and data products must be in place and implemented.

Whenever deemed appropriate by the Project Manager and the MSL QA Officer, the MSL QA Officer shall perform audits of subcontractors. These audits may include data audits, inspection of facilities, or inspection of project activities.

5.0 PROJECT PLANNING DOCUMENTS

Project planning documents (e.g., work plans, QAPjPs, toxicity testing plans, health and safety plans, field sampling plans) are documents that describe the objectives of a project and the methods, organization, and QA and QC activities necessary to meet the goals of the project. It is Battelle MSL policy that each project conducted by the MSL must have a planning document that adequately describes the work to be performed, has been approved by the Project Manager, and is in place prior to the start of work.

5.1 RESPONSIBILITIES

It is the responsibility of the Project Manager to:

- Ensure that a project work plan, QAPjP or both is prepared prior to work initiation and that it meets the requirements of the MSL, the client, and any applicable regulations
- Approve the plan and to obtain any other necessary approvals
- Ensure that the planning documents are made available to project personnel
- Ensure that project participants are adequately trained to perform the assigned work and that the training is documented as required.

Each staff member involved in the project is responsible for performing his/her task(s) in conformance with the planning documents. MSL staff members are also responsible for notifying their supervisor or the appropriate manager of any deviations to the procedures/methods specified in the planning documents.

The QA Officer is responsible for reviewing project planning documents for conformance to relevant regulations and MSL policies.

5.2 CONTENT AND FORMAT

A significant amount of the work performed by the MSL is conducted for the U.S. EPA. EPA requires that all environmental data-collection activities conducted for the EPA must be covered by a QAPjP. Therefore, all project planning documents prepared for the EPA must adhere to specific content and format requirements, as dictated by the EPA office involved. Protocols written for studies conducted under Food and Drug Administration (FDA) or EPA GLP standards must adhere to the specifications of 21 Code of Federal Regulations (CFR) Part 58 (FDA), 40 CFR Part 160 (EPA/ Federal Insecticide, Fungicide, and Rodenticide Act [FIFRA]), or 40 CFR Part 792 (EPA/Toxic Substances Control Act (TSCA), as applicable.

In the absence of client-driven requirements, the MSL has established minimum standards for project work plans. These are, as applicable

- A descriptive title, client name, Battelle project number, and effective date
- The identities of the project manager, task leaders, and other key project personnel, including subcontractors
- A statement of the general goals and the specific DQOs of the project
- A description of the experimental design and procedures
- A description of the QA and QC procedures that will be applied to the project tasks

- The project schedule, including milestones and deliverables
- A description of the types of data to be recorded
- A statement of deliverable requirements

5.3 APPROVAL AND DISTRIBUTION

At a minimum, the planning documents must be approved by the Project Manager. Additional approvals may be required by MSL policy or by the client. All planning documents shall be approved before work is started on the project.

The project planning document is distributed, or made available to, all key personnel involved in the project and to the QA Officer. It is expected that all work will be conducted according to the planning documents. Modifications to approved planning document procedures should be made only with the concurrence of the Project Manager.

5.4 DATA QUALITY OBJECTIVES (DQO)

DQOs are defined as the criteria needed to design an environmental data collection program. DQOs are developed from a multi-step, reiterative process that involves, project management, technical staff, and the individuals who will be using the data to make decisions. The DQO process entails

- Stating the problem to be resolved, including limitations of time and resources
- Identifying the decision that will be made using the data
- Identifying inputs to the decision, including the environmental measurements needed and the criteria for taking action
- Specifying how the results will be summarized and used
- Specifying acceptable error rates (i.e., limits on uncertainty)

The objective of the DQO development process is to design a cost-effective program that will provide the necessary amount and type of sufficient-quality data.

During the development of DQOs, the parameters of accuracy, precision, completeness, comparability, representativeness, and sensitivity are commonly considered when measuring data quality. These qualitative and quantitative parameters are described below.

Accuracy is a measure of the bias of a system or measurement. It is the closeness of agreement between an observed value and an accepted value.

Precision is the degree of mutual agreement among individual measurements of the same property obtained under similar conditions.

Completeness is the amount of data collected as compared to the amount that was needed to ensure that the uncertainty or error is within acceptable limits.

Comparability is a measure of the confidence with which one data set can be compared to another.

Representativeness is the degree to which data accurately and precisely represent a characteristic of a population.

Sensitivity is the capability of methodology or instrumentation to discriminate among measurement responses for quantitative difference of a parameter of interest.

Once the acceptable error rate has been defined, the program's QA requirements are developed in response. The specific types of QC samples used to measure data quality are discussed in Section 9.0 of this QAMP.

Further definitions and applications of DQOs for chemical and biological analyses are contained in Volumes 2 and 3 of this QAMP.

6.0 STANDARD OPERATING PROCEDURES

6.1 SCOPE AND PURPOSE

Battelle MSL's policy requires that SOPs be written for all routine environmental measurement procedures that are associated with data collection and analysis and related QA/QC activities. Procedures that are not routine, or are unique to a project, are described in project planning documents or in written protocols included in the project files. Subjects that are covered in SOPs include, but are not limited to:

- Sample collection
- Sample handling, preservation, and storage
- Chain-of-custody procedures
- Sample analysis
- Bioassay toxicity testing
- Equipment use, maintenance, and calibration
- Record management
- Data reduction, processing, and validation
- QA verification activities

SOPs are documents that describe procedures that must be followed to ensure the integrity and quality of data. SOPs serve a multi-purpose function, including to

- Reduce the introduction of errors and variables by ensuring the consistent use of appropriate procedures

- Communicate to the necessary people (e.g., client, project personnel) how the work will be conducted

- Increase the effectiveness of training by clearly and consistently communicating the approved method of performing a procedure

- Provide a historical record of the work performed

- Provide a basis for data comparability

- Provide a basis for maintaining reproducible results and producing defensible data.

A list of all MSL procedures is contained in Appendix B of Volume 1 of this QAMP.

6.2 CONTENT AND FORMAT

Each SOP must be clearly written and include sufficient detail to clearly describe the operation to be carried out so that a qualified individual can perform the procedure. However, it should be flexible enough to accommodate expected variations while maintaining the integrity of the procedure and the quality of the data being generated. SOPs covering equipment must include descriptions of calibration, operation, and maintenance requirements. Procedural SOPs must contain sections on preparation, procedures, calculations, and quality control. Equipment and procedural SOPs must also include a discussion of the safety concerns associated with the equipment or procedure. All SOPs must state the objective or application of the SOP topic and must stipulate the requirements for the successful completion of training. Specific requirements for content and format are stipulated in SOP, MSL-A-003, *Guidelines for SOP Format and Control*.

6.3 RESPONSIBILITIES

Each Technical Group Leader is responsible for ensuring that the routine procedures needed within their group are written and for providing resources for their preparation. The Technical Group Leader also is responsible for approving all procedures produced within his/her group.

The individual preparing the SOP is responsible for ensuring that the SOP completely and accurately describes the procedures, is based on sound scientific principles or recognized procedures, and conforms to the MSL standards for procedure documentation as specified in MSL-A-003, *Guidelines for SOP Format and Control*).

The QA Officer is responsible for

- Assigning each SOP a unique number and entering it into the SOP controlled document log
- Reviewing and approving all SOPs
- Distributing approved SOPs
- Maintaining historical files of SOPs

6.4 REVIEWS AND APPROVALS

Draft procedures must go through a formal review and approval process. At least one technical reviewer of record is assigned. Additional technical reviewers are encouraged for new procedures. The MSL QA Officer and the Technical Group Manager are also reviewers and provide final signature approval. Users should be included in the review process to ensure that the procedure is accurate and able to be implemented. Review comments for all reviewers can be submitted to the procedure file. Review comments and any documentation of comment resolution by the technical reviewer of record (who provides signature approval) should be submitted to the QA Officer to be maintained in the procedure file. The SOP is reviewed, and when satisfactory, is signed and dated, at a minimum, by the following people:

- Author – who becomes the procedure subject matter expert
- Technical Reviewer - should be someone who will be able to assure that the procedure is technically adequate, complete and correct.
- MSL Quality Assurance Officer (not required for safety procedures)
- MSL Manager for non-technical procedures or the appropriate Technical Group Manager for technical procedures.

6.5 DISTRIBUTION AND CONTROL

Copies of all current MSL procedures are kept in the QA Office and are available to all MSL staff upon request. In addition, MSL Procedure Manuals, which contain copies of all current MSL Procedures are issued to managers and professional-level staff and are located in areas that are accessible to all staff requiring their use. All procedures used at the MSL will be controlled by the QA Officer. Original SOPs, both current and historical versions, are maintained by the QA Officer.

6.6 MODIFICATION AND REVISION

Changes to SOPs must be controlled to ensure documentation and traceability to the modification. SOP modifications fall into three categories: one-time modifications, interim changes, and major changes.

<u>One-Time Modifications</u>

A "one-time" modification is used when the change is for a one-time use of the procedure. This situation is often guided by a customer request. It is not intended to result in modifications to other copies or future uses of the procedure.
If the change is significant, then the user should document the modification and associated justification, obtain the approval of the project manager, and proceed with the modified work activity.

If the change is not significant, then the user should document the modification and associated justification, proceed with the modified work activity, and later have the change reviewed by the project manager.

The modification and justification shall be documented in an appropriate place such as the data sheet, daily log or other raw data documentation.

Interim Changes

An interim change may be made when a procedure requires a modification and formal revision is not prudent or timely. This can be used for minor changes (minor procedural changes, typographical errors, etc.) or changes of a more substantive nature. The user should document the interim change; and the line or project manager who required the procedure should approve the change. The interim change should be communicated to all users of the procedure, and the user should then initiate the review and approval process to revise the procedure if the change is substantive or permanent.

Major Changes

Major changes (e.g., new equipment specifications, maintenance procedures, and major procedural changes) require a revision of the procedure. Major revisions must go through the writing of a formal review draft and receive approval consistent with the requirements specified in Section 4.3 above., before being implemented. All revised procedures retain their original number assignments but are issued a new revision number.

7.0 LABORATORY DOCUMENTATION AND RECORDS

A critical component in the generation quality products is proper record keeping and the maintenance of the records after project completion. Documentation must be sufficiently detailed so that the data are traceable and program data could be reconstructed based on the project records. These records must be maintained in a secure location and must be identifiable and retrievable.

7.1 DOCUMENTATION

It is Battelle MSL policy that data generated during the course of a project must be capable of withstanding challenges to its validity, accuracy, legibility and traceability. To meet this objective, data are recorded in standardized formats and in accordance with prescribed procedures. All staff members whose responsibilities include recording data must be aware of, and adhere to, the procedures during the performance of their work. Briefly, data must be entered onto data sheets or in project notebooks directly, promptly, and legibly. All entries must be made in reproducible ink, and must be initialed and dated by the person making the entry. Changes or corrections to data must not obliterate the original entry, but must be indicated with a single line through the original entry. All changes or corrections must be accompanied by the initials of the person making the change, the date, and when not obvious, an explanation of the change. Specific requirements for documentation are included in procedures MSL-D-001, *Recording Data on Data Sheets and Laboratory Notebooks* and MSL-D-004, *Data Reporting, Reduction, Back Up, and Archiving*.

7.2 RECORDS

The MSL data archive system is designed to ensure that materials are stored in an orderly manner under secure conditions, and may be easily and promptly retrieved should the need arise. Specific details are found in procedures MSL-D-003, *Archiving of Records, data, and Retired SOPs* and in MSL-D-004, *Data Reporting, Reduction, Back Up, and Archiving*.

All material generated during a project conducted by the MSL must be archived upon completion of the project. All records necessary for the interpretation and evaluation of project data, including planning documents, raw data and other documentation, correspondence, and reports, should be retained. The Project Manager is responsible for ensuring the project materials are collected, organized, and forwarded to the archives at the end of the project. MSL policy is to retain electronic data files for five years, unless otherwise specified by customer request. Hard copy data are stored indefinitely as per MSL procedure MSL-D-003, Archiving of Records, Data, and Retired SOPs which primarily addresses GLP requirements. Archives are controlled access (locked) storage rooms. Data are stored and retrieved by project number.

8.0 SAMPLE CONTROL

Sample control is the formal system designed to provide sufficient information to reconstruct the history of each sample. This system involves procedural, record keeping, and organizational components and is critical for any environmental program that is generating data that may be used for regulatory decisions or in support of litigation.

8.1 PROCEDURES

The MSL sample control system encompasses the following elements

- Upon collection or preparation, each sample is assigned and labeled with a unique identification code that allows it to be tracked through analysis and reporting. Assignment of identification numbers is on a group or project-specific basis.

- Standard forms are used to document the history of each sample, including collection, storage, preservation, processing, and analysis.

- Samples are received, logged in, stored, and archived according to SOP MSL-A-001, *Sample Log-in Procedure*.

- Samples are stored in controlled or secure areas.

- Transfer of the custody of samples (both within the MSL and to outside agencies) and the removal of samples in and out of storage is documented in accordance with SOP MSL-A-002, *Sample Chain of Custody*.

Specific sample custody requirements for the Marine Chemistry and Ocean Processes Group and the Marine Ecological Processes Group are addressed in Volumes 2 and 3 respectively.

8.2 RESPONSIBILITIES

Sample custody responsibilities must be clearly defined and understood by all personnel involved for the system to be effective. Samples are considered to be in a person's custody if

- The samples are in a person's actual possession
- The samples are in a person's view after being in that person's possession
- The samples were in a person's possession and then were locked or sealed to prevent tampering
- The samples are in a secure area

The sample collector is responsible for the proper collection, preservation, and labeling of samples, and for documentation of sample history and custody in the field. The sample collector also is responsible for packaging the samples for shipment and for arranging for transportation to the laboratory.

Responsibilities of the laboratory sample custodian include receiving and inventorying the samples, placing them in storage, and completing the documentation associated with these procedures. The laboratory sample custodian also is responsible for informing the Project Manager of the samples' arrival and for promptly notifying him/her of any broken, missing, or compromised samples.

9.0 QUALITY CONTROL

QC activities are performed by technical personnel during the conduct of the project. The purpose of these functions is to measure the quality of the data and if necessary, adjust the measurement system so that the specified level of quality is attained.

9.1 INTERNAL QUALITY CONTROL CHECKS

The following are common types of QC procedures implemented by the Battelle MSL Marine Chemistry and Ocean Processes Group.

Method blanks - Method (or procedural) blanks are prepared in the laboratory using the same reagents, solvents, glassware, and equipment as the field samples and accompany the field samples through analysis. Method blanks serve as a means to measure contamination associated with laboratory processing and analysis.

Matrix spikes - Matrix spike (MS) samples are field samples that are spiked in the laboratory with target analytes and analyzed under the same condition as the field samples. Matrix spikes provide a measure of the recovery efficiency of the analytical method and are generally analyzed in duplicate (matrix spike/matrix spike duplicate [MSD]).

Blank spikes - Blank spikes are similar to matrix spikes but are prepared by spiking the target analytes into a clean matrix (e.g., deionized water). Blanks spikes also are used to measure the recovery efficiency of the analytical method, but without the interference of the matrix.

Laboratory replicates - Laboratory replicates consist of splitting a single sample or compositing and splitting two or more samples in the laboratory, and subsequently processed and analyzed as separate samples. Laboratory replicates serve as a measure of the error associated with the analytical process.

Standard reference materials (SRM) - SRMs are materials for which certain properties have been certified by a recognized authority.

Reference samples - Reference samples are samples for which selected properties are known, generally through historical analysis. Reference samples are used as a benchmark for similar analyses.

QC samples may also be collected in the field to monitor contamination and to assess sampling error. Common field-related QC samples include

Equipment blanks - Equipment blanks are prepared in the field using the freshly decontaminated sampling equipment. Deionized water is poured over and through the equipment, collected in an identical sampling container, and shipped to the laboratory for processing and analysis. Equipment blanks measure the contamination associated with the entire sampling and analytical process.

Split samples - Split samples are obtained by compositing sample material in the field and dividing the material into separate containers for processing and analysis. Split samples are used to assess the total error associated with sampling and analysis. If split samples are sent to separate laboratories for analysis, interlaboratory variation may also be obtained.

Field replicates - Field replicates are two or more separate samples that have been collected from the same sampling point. Field replicates also serve to measure the error associated with the entire sampling and analytical process, including variation inherent in the sampled media.

QC checks are associated with biological toxicity testing (independent recounting of sample, reference toxicity tests, establishment of acceptable water quality measurement ranges) and data processing (proofing or double entry/comparison programs). The specific QC procedures, frequency of performance, and criteria for acceptance for all environmental data collection procedures are defined in SOPs or in the project planning documents.

The immediate monitoring of QC results allows the data collection process to be continually compared to pre-established acceptance criteria and corrected as necessary. In addition, assessment of QC results is a critical component of the data validation process (Section 10.0) and is used to interpret the accompanying sample data and to judge its acceptability and usefulness with regard to the project DQOs. QC results are reported with the project data.

Within the Marine and Environmental Chemistry and Ecotoxicology and Risk Assessment Groups, control charts have been established for selected QC analyses (i.e., inorganic and organic analytes and reference toxicity results).

See Volumes 2 and 3 of this QAMP for specific requirements for quality control samples, quality control criteria and control charts.

9.2 APPROVALS BY OUTSIDE AGENCIES

MSL is accredited by the states listed below. As part of the state accreditation programs, MSL participates in several chemistry laboratory intercomparison and certification programs that require analysis of performance evaluation samples and also participates in inter-laboratory toxicology comparisons whenever offered. Battelle MSL also is routinely audited by its clients.

State	Accreditation Organization
Florida	Department of Environmental Protection (DEP)
New Jersey	Department of Environmental Protection (DEP)
South Carolina	Department of Health and Environmental Control (DHEC)
Washington	Department of Ecology (WA DOE)
Wisconsin	Department of Natural Resources (DNR)

Accreditation through the Navy QA Program and NELAP are in progress. In the past, MSL has participated in the following Performance Evaluation Studies:

EPA - Water Pollution (WP) Laboratory Performance Evaluation Study
EPA - Water Supply (WS) Laboratory Performance Evaluation Study
Mercury Intercomparison Program (MIP)
International Atomic Energy Agency (IAEA) - World Wide Intercomparison for Trace Elements
National Oceanic and Atmospheric Administration (NOAA) National Status and Trends

In many cases these programs have ended (e.g., WP, MIP), and have not been replaced by new programs. Currently PE samples are purchased from an approved vendor on the list maintained by the

WA DOE. MSL is currently participating in the CalFed Mercury QA Program, a study to demonstrate comparability among laboratories. Results are expected in early 2000.

9.2.1 Certifications

Certification programs are based on the demonstration of a function quality program, the existence of planning documents and procedures, the successful analysis of external performance samples at least twice per year, and in some cases, periodic on-site assessments. Specifics of MSL certification is described in MSL-A-013, *Laboratory Accreditation and PE Sample Analysis*. MSL maintains the following documentation to meet these requirements:

- Quality Assurance Management Plan
- Comprehensive QA Plan (for the State of Florida)
- Procedures in the following general areas (numbers of procedures)
  - Quality Assurance
  - Administration
  - Documentation, Records, and Reports
  - Organic Chemistry
  - Inorganic Chemistry
  - Conventional Chemistry
  - Water Quality Instrumentation
  - Toxicological Testing
  - Facilities,
  - Safety, and
  - Work Practices

MSL participates in performance studies at the required frequency as per MSL procedure, MSL-A-013*, Laboratory Accreditation and Performance Evaluation Sample Analysis*. Customers are provided with the results of recent performance studies on request.

9.2.2 Performance Evaluations

MSL analysts are degreed staff operating analytical instruments on a daily basis. The dedication of analytical staff to the specific procedures for which they are responsible, their level of training and, daily QC assessments of proficiency through the analysis of blank samples, sample replicates, SRMs, and MSs combine to make the results produced by MSL highly defensible, accurate, precise, and repeatable. MSL is a specialty laboratory, providing its customers with relatively low detection limits for environmental samples. Daily proficiency is monitored at the bench level, at the level of data assessments performed on sample sets by the analyst and the MSL Data Coordinator (data validation), and at the level of the MSL QA Officer who provides data quality verification. Internal PE samples may be provided as blind or double blind samples to the analyst by a Project Manager, the Marine and Environmental Chemistry Manager, or the MSL QA Officer. The source for internal PE samples is generally previously analyzed, archived PE samples. Internal PE samples provide an indication of analyst proficiency and instrument performance and are used to return serviced equipment to full operation, or to provide an instrument check when preventive maintenance has been performed. Blind internal PE samples are also used to test initial method/instrument proficiency when training new staff.

External PE sample results are used at MSL as an external verification of analyst proficiency and as a means of comparison with ones peers. An “Unacceptable” data evaluation through the PE sample

program is taken seriously and the entire system is reviewed for anomalies. If an “Unacceptable” data evaluation is obtained, various parts of the analytical process (e.g., digestion, dilution, instrument injection) are investigated using the archived PE sample. In addition, once the results from the previous

study are received, then that archived sample has a known certified mean and range and can be used (if used < 6 moths from sample receipt) as an internal PE sample or a QC verification sample. Most available external PE samples that can be purchased are aqueous. MSL participates in programs to analyze sediment and tissue samples (e.g., NOAA Trace Metals Intercomparison) whenever offered.

10.0 DATA REDUCTION, REPORTING, AND VALIDATION

10.1 DATA REDUCTION

Reduction of raw data shall be accomplished using established techniques. The calculations required to perform the reduction of data may be performed manually or with the aid of automated data processing systems. In either case, the procedures for the testing and analysis of samples or the QAPjPs will specify the calculations and the mode for raw data processing. If manual processing is to be used for data validation, then the procedure or QAPjP will provide the calculation method and the units for reporting derived values. In order to reduce the potential of errors in data transcription the manual transfer of data will be minimized. All calculations performed manually will be checked for accuracy by someone other than the person who performed the original calculation. Data validation checks shall be documented by the signature and date of the reviewer. Separate documentation is acceptable, provided traceable records are maintained. For automated data reduction methods, the accuracy of calculations will be verified through the use of standards or test case inputs with known resultant values.

10.2 REPORTS

Technical reports are the primary product produced by the MSL. To ensure the quality of the reports, two mechanisms are used: (1) the selection of technical staff with the appropriate mix of technical and writing skills to produce data products and reports, and (2) a formal system of review and correction.

MSL policy requires that all deliverables prepared for clients must be submitted to an internal review before being released to the client. The document is then reviewed as per MSL-Q-002, *Quality Assurance Audits of Reports*, for technical content, conformance to QA policies and procedures, and editorial correctness.

The purpose of the technical review is to evaluate the document for technical quality (including scientific validity and logic), conformance to client expectations, and for agreement with MSL policies. This review is performed by a senior technical staff member selected for familiarity with the technical discipline of the work being reported. The QA review is conducted by the QA Officer and encompasses accuracy, completeness, adequacy of QA issues, and conformance to applicable standards, including federal regulation (when applicable), project planning document requirements, and MSL policies. Editorial review addresses grammatical correctness and consistency of style and format.

The reviewer's comments are communicated in writing to the author who revises the document, if necessary. The revised document is then sent to the Technical Group Leader or designee for final approval prior to its release.

10.3 DATA VALIDATION

Prior to their use, data must be validated. Validation is defined as the process through which data are accepted or rejected and consists of proofing, verifying, editing, and technical reviewing activities. At the MSL, data validation is described in MSL-D-004*, Data Reporting, Reduction, Back Up, and Archiving*, and it is considered a technical function and must occur prior to the data being audited by the QA Officer (Section 11.2).

Data validation occurs at multiple levels as data are collected and processed:

- Individuals recording data during field or laboratory operations are responsible for reviewing their work at the end of the day to ensure that the data are complete and accurate.

- Analysts and instrument users are responsible for monitoring the instrument operation to ensure that instrument has been properly calibrated.

- Laboratory Supervisors and Project Managers are responsible for reviewing analytical results and supporting documentation to assess sample holding times and conditions, equipment calibration, and sample integrity. As an additional measure of acceptability, the results of QC samples are compared to the project DQOs.

- Technical staff are responsible for reviewing the data for scientific reasonableness.

- All manual entries into databases and spreadsheets are verified, either through proofing or by double entry/comparison programs.

- All calculations performed by hand are checked for accuracy.

Data that do not meet the pre-established criteria for acceptance may be flagged, not reported, or reported with an explanation of the limitations, at the discretion of the Project Manager.

10.4 MSL DATA AUDIT PROCESS

The MSL data audit process is primarily a data verification activity that is described in MSL-Q-005, *Quality Assurance Data Audits*. However, verification of validation activities also occurs. Complete data packages including all kit information, hard copies of instrument outputs, and summary data sheets are provided to the MSL QA Officer or designee for review. Analytical data packages are reviewed to a checklist. Project notebooks, because of their variability, are not reviewed to a checklist. However, the review process is essentially the same. Data are reviewed to ensure that the data are accurate, traceable, defensible, and complete, as compared to the planning documents and/or project requirements. The audit procedure is a randomized check that involves comparing selected reported values to the original data. This check can either be performed randomly or on a statistical basis. Results of the data audit are documented either on the checklist from MSL-Q-005 or in a summary statement. Concerns that can be corrected will be corrected before the data are released. Deviations are required to be summarized and provided to the customer.

10.5 CONFIDENTIALITY

MSL policy does not allow the release of customer data or project-related information to anyone except the customer unless expressly directed by the customer or an authorized representative.

11.0 VERIFICATION ACTIVITIES

One of the policies of the MSL is to assure that the products generated, and the services performed by the MSL meet the standards established by Battelle and its clients. The Self Assessment Program (SAP) is the MSL's performance measurement system. The SAP

- provides MSL staff and management accurate technical, business and operational performance information that promotes early identification and resolution of problems that may impact achievement of the MSL critical outcomes and objectives
- verifies conformance to established requirements
- verifies effective conduct of activities to protect the environment and the health and safety of workers and the public
- contributes to ongoing improvement in performance.

The first process of the performance measurement system is determining the MSL's critical outcomes and performance objectives and indicators. The MSL's critical outcomes and associated performance objectives and indicators are established by PNNL's Environmental Technology Division (ETD) and MSL staff on an annual basis. The key performance objectives and indicators resulting from this process drive the development of self assessment plans.

The second process of the performance measurement system is developing and implementing an assessment plan. The MSL develops an annual assessment plan as part of ETD's self assessment program. The assessment plan describes the assessment activities that the MSL performs to ensure that plans and controls are in place to achieve its objectives.

The third process of the performance measurement system is the overall evaluation of the MSL's performance and is described in section 11.1. The primary mechanism for evaluating this performance measurement system is assessment activities. Assessment activities refer to the verification of conformance to the MSL's SAP and include line management assessments, QA assessments, and data audits. During a QA assessment or data audit, the agreement between data and data quality objectives or indicators with QA policy documents (e.g., QAMP, SOPs, project planning documents) is evaluated, deficiencies are identified, and corrective action is taken.

The final step in the performance measurement system is to implement the key improvement opportunities that the evaluation processes identified (See Section 12). Improvement areas requiring action are implemented as determined by the MSL Manager, Technical Group Leader and/or QA Officer.

11.1 ASSESSMENTS

As part of the MSL SAP, assessments are performed in accordance with the SBMS subject area, *Conducting and Using Results From Operational Assessments*, by staff and line management to evaluate the performance of the MSL. Assessment methods include, but are not limited to, walkthroughs, procedure and program reviews, staff feedback, and safety, health, and environmental evaluations.

In addition, the QA Officer conducts QA assessments to assess that facilities, equipment, personnel, methods, practices, records and quality control are in conformance to approved planning documents, procedures, regulations, client requirements and Battelle policy. QA assessments are scheduled based on a request from the MSL Laboratory Manager, the definition of critical phase inspections by project managers or MSL customers, and by scheduling by the MSL QA Officer when a new procedure is

implemented or significantly revised, when a new study type is initiated, or when data quality reviews indicate technical systems problems. At least 25 assessments per annum are the target. External assessments of suppliers are conducted through the Battelle Quality Process Division in Richland, WA and are related to qualifying preferred suppliers.

QA assessments are formal or informal verification activities that are performed in accordance with procedure MSL-Q-002, *Quality Assurance Inspections of MSL System and Study Activities* and subject area, *Conducting and Using Results From Operational Assessments*. The purpose of a formal QA assessment is to determine verification with a requirement and includes formal corrective action and follow-up. If the assessment is determined to be informal, the purpose is to determine the status and to report the factual evidence and is not intended to be a verification activity with formal corrective action response, follow-up, etc. Informal assessments are generally requested by MSL management to assess the status of a particular activity.

A schedule of all QA assessments, which are not part of the MSL's self assessment plan, will be completed by the QA Officer and, as needed, issued quarterly to the MSL Manager and the Technical Group Leaders. This schedule will include verifications based on client needs, management requests and routine internal verifications (i.e., checking standards logs, sample preparation forms, QC checklists, equipment calibration and maintenance, etc.).

11.2 DATA AUDITS

MSL policy requires that all environmental measurement data produced by the Technical Groups must be audited prior to their final release. The reported data are audited, using a process that ensures that the data are complete, accurate, traceable, and defensible. Details of the data auditing process is documented in SOP MSL-Q-005, *Quality Assurance Data Audits*.

11.3 QA REPORTS TO MANAGEMENT

Upon completion of the QA assessment activity, the QA Officer prepares a written report that specifies the basis of the assessment activity, identifies the type of assessment activity and phase covered, and summarizes the results of the assessment activity. The report is signed and dated by the QA Officer and forwarded to the appropriate manager, who reviews assessment results and determines corrective action. Each deficiency must be addressed in writing. The Project Manager (when appropriate) and the Technical Group Leader then sign and date the report and return it to the QA Officer for verification of the responses.

Quarterly, the QA Officer will submit to the Technical Group Leaders and the MSL Manager a summary of the past quarters QA activities. Subjects to be covered in the quarterly QA report as addressed in MSL-Q-008, *QA Reports to MSL Management*, and shall include, but not be limited to, results of assessment activities, results of performance evaluation samples, trends of deficiencies, and other important QA-related issues.

11.4 CORRECTIVE ACTION

Typical corrective actions for exceeding the project-specified DQOs for chemistry analyses and bioassay and aquatic toxicology tests are summarized in Section 5 of volumes 2 and 3 of this document. This topic is also addressed in Section 12 below. In addition, individual analytical procedures may contain appropriate corrective actions for various routine problems. MSL procedure MSL-A-005, *Deviations from Established Requirements*, addresses an approach to differentiate between acceptable deviations that will be reported to the client and formal deviations requiring a greater level of investigation to determine the root cause, documentation and verification of corrective action implementation and the effectiveness of

actions designed to prevent recurrence. Deviations may be found during routine data validation or verification activities, during an assessment or identified by any project participant. In most cases, the MSL QA Officer and the Project Manager have the primary responsibility for evaluating the impact of the deviation on data quality, and defining required corrective actions. In some cases the client may also be involved in this assessment. Deviations and deficiencies and the assigned corrective actions are documented on a Quality Problem Report (QPR, refer to Exhibit 12.1 in the following chapter). It is the Project Manager's responsibility to ensure completion of the identified corrective action by the expected completion date, and to request independent verification (when required).

When there has been an impact on data, the Project Manager shall assure that there is a cross reference in the raw data that indicates there is an associated QPR (i.e., refer to the QPR on each of the impacted data sheets, in the laboratory record book, and in any other documents used to transcribe the information or data).

Once a quarter, the MSL QA Representative shall present to MSL Management a summary of all QPRs, any significant control limit data deficiencies, and an analysis of trends or recurring deficiencies as part of the quarterly QA Report to Management.

12.0 QUALITY IMPROVEMENT

Quality improvement is a critical aspect of the MSL Self Assessment Program and involves both corrective action to identified deviations and continuous improvement processes.

The corrective action process involves determining, implementing, approving, and verifying the appropriate remedial action. Corrective actions may be identified by technical personnel during the course of work performance, or may be in response to assessment activities.

The continuous improvement process involves determining and prioritizing improvement areas, implementing improvement action and documenting the disposition of each action.

12.1 DEVIATIONS

Each individual engaged in project activities should be alert to problems, deviations from approved procedures, out-of-control events, or other issues that may require corrective action. The appropriate response is determined by the event. SOP MSL-A-005, *Deviations from Established Requirements* provides methods for addressing deviations from MSL procedures, planning documents, and client requirements.

Briefly, deviations are identified either as observations or quality problems as follows.

Observations are defined as incidences that require action or correction but are not considered ongoing, operational problems.

Quality Problems are defined as situations where the quality and usability of data, a process, or item are indeterminate (i.e., no objective evidence is available to substantiate data quality or to indicate that established procedures/requirements were met). Quality problems can be (1) repeated incidences of an observation, (2) repeated errors due to a flaw in the data generation or validation process, or (3) assessment issues that require a change in laboratory procedures or processes.

It is MSL policy that all issues that may impact the quality of the data must be documented. The documentation must clearly state the event and the corrective action taken in response, and must be approved by the appropriate management representative. Acceptance of data that exceeds pre-established criteria also must be documented and justified.

Depending on the severity of the deviation, the MSL QA Officer and the Project Manager will determine how the deviation will be documented (i.e., through use of a Quality Problem Report form (Exhibit 12.1) per MSL-A-005, *Deviations from Established Requirements*. The MSL QA Officer and the Project Manager will determine if there is a formal deviation when one or more control limits are exceeded in a data set. In some cases, the customer may be involved in these discussions. Deviations from project control limits will be identified in the narrative accompanying the data set or package or in a letter to the customer, and the impact of the deviation addressed. The following are guidelines to resolving deviations:

- All deviations from approved procedures, project planning documents or this QAMP will be documented.

- Issues that affect cost, schedule, or performance of the project will be reported to the Project Manager. The Project Manager will then be responsible for evaluating the overall impact to the project and implementing the necessary corrective actions.

- Deficiencies identified through QA assessment activities will be brought to the attention of the Project Manager and the Technical Group Leader. Implementation of corrective action will be the responsibility of the Project Manager.

- See Section 5 of Volumes 2 and 3 of this QAMP for additional information regarding corrective action of identified deviations.

12.2 ASSESSMENT ACTIVITIES

For all assessment activities, a system of notification and verification of corrective action is in place. An assessment report is prepared and submitted to the appropriate Manager (Project Manager or Technical Group Leader). The Manager reviews the assessment results to determine overall impact and risk and then determines corrective action and prioritizes the actions. The Manager assigns the corrective actions to individuals. The Managers ensures that the corrective action is tracked to completion and as part of completion, documentation is included that describes the justification for completion of the corrective action. Issues that in the manager's judgement require significant corrective action should be scheduled for verification of that corrective action at a subsequent assessment.

Issues that in the manager's judgement require process improvement instead of, or in addition to, corrective action, are identified as such and any improvement actions are implemented and documented.

EXHIBIT 12.1 Quality Problem Report Form

Originator: _____ **Project Manager:** _____	**Date:** _____ _____	**QPR Number:** **Project Number:** _____ **Project Title:** _____

Purchase Order Number/SOP/Work Plan:

10CFR830.120 Related? Yes or No

Statement of the Deviation

Impact of the Deviation

Steps to Prevent Inadvertent Use of the Item or Process

Cause of Events Leading to the Problem

Planned Corrective Action for the Immediate Problem/Independent Verification Required? Yes or No

Planned Corrective Action to Prevent Recurrence/Independent Verification Required? Yes or No

Person Responsible for the Corrective Action	**Approval of Planned Corrective Action**
Last Name, First, MI	*Cognizant Manager or Designee*

Closing the Problem

Actions Completed as Planned	Intermediate Distribution:
Name *Date*	
Independent Verification Has Been Completed (if required)	Final Distribution: MSL QA Office
MSL QA Representative *Date*	

APPENDIX A
List of Acronyms

ANWAP	Arctic Nuclear Waste Assessment Project
APDC	Ammonium pyrrolidinedithiocarbamate
AVS	Acid Volatile Sulfide
CCV	Continuing Calibration Verification
CFR	Code of Federal Regulations
CHP	Certified Health Physicist
CMS	Chemical Management System
CoC	Chain of Custody
CREM	Coastal Resource and Ecosystem Administrative Management
CRM	Certified Reference Material
DEP	Department of Environmental Protection
DHEC	Department of Health and Environmental Control
DO	Dissolved Oxygen
DOE	Department of Energy
DNR	Department of Natural Resources
DQO	Data Quality Objective
ECD	Electron-capture Detector
EPA	Environmental Protection Agency
ES&H	Environment, Safety, and Health
EPRI	Electric Power Research Institute
FDA	Food and Drug Administration
FID	Flame-ionization Detector
FIFRA	Federal Insecticide, Fungicide, and Rodenticide Act
FPD	Flame Photometric Detector
GC	Gas Chromatography
GFAA	Graphite Furnace Atomic Absorption
GPM	Gallons Per Minute
GLP	Good Laboratory Practices
HMC	Hazardous Materials Coordinator
HPLC	High-Performance Liquid Chromatography
HVAC	Heating Ventilation and Cooling System
IAEA	International Atomic Energy Agency
ICP-AES	Inductively Coupled Plasma – Atomic Emissions Spectrometry
ICP-MS	Inductively Coupled Plasma (Emissions) – Mass Spectrometry
ICV	Initial Calibration Verification
ID	Identification
MDL	Method Detection Limit

APPENDIX A
List of Acronyms

MIP	Mercury Intercomparison Program
MS	Mass Spectroscopy
MSL	Marine Science Laboratory
NAUI	National Association of Underwater Instructors Technical Diving International
NBS	National Bureau of Standards
NELAP	National Environmental Laboratory Accreditation Program
NIST	National Institute of Standards and Technology
NOAA	National Oceanic and Atmospheric Administration
NPDES	National Pollutant Discharge Elimination System
NRCC	National Research Council of Canada
PAH	Polycyclic Aromatic Hydrocarbon
PCB	Polychlorinated Biphenyls
PNNL	Pacific Northwest National Laboratory
PSEP	Puget Sound Estuary Program
PVC	Polyvinyl Chloride
QA	Quality Assurance
QAMP	Quality Assurance Management Plan
QAMS	Quality Assurance Management Staff
QAPjP	Quality Assurance Project Plan
QC	Quality Control
QPR	Quality Problem Report
REM	Registered Environmental Manager
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
RSO	Radiation Safety Officer
SBMS	Standards Based Management System
SEM	Simultaneously Extracted Metals
SESP	Surface Environmental Surveillance Project
SIC	Sample Inventory Coordinator
SOP	Standard Operating Procedures
SOW	Statement of Work
SRM	Standard Reference Material
T	Temperature
TBT	Tributyl Tin
TDI	Technical Diving International
TSCA	Toxic Substances Control Act
UV	Ultraviolet (light)
VIS	Visible (light)

WA DOE	State of Washington, Department of Ecology
WHP	World Hydrographic Global Measurement Program
WIPP	Waste Isolation Pilot Plant
WP	Water Pollution
WS	Water Supply

APPENDIX B
BATTELLE MSL PERSONNEL

The following is a list of MSL personnel and a summary of their current position, and length of relevant experience. Educational background and work experience are documented on the Qualification and Training form which is a part of each person's training file.

Mr. Liam Antrim, has over 21 years experience in environmental science. He has worked on a variety of aquatic toxicity projects, focusing on acute and chronic toxicity testing of industrial effluents, the effects of contaminants in near-shore and urban sediments on marine and freshwater organisms, and collection and toxicity testing of the sea-surface microlayer. He has participated in numerous sample collection projects in Puget Sound, the Gulf Stream, Chesapeake Bay, and southern California for federal and industrial clients. Mr. Antrim is currently the MSL Dive Officer.

Mr. Chuck Apts has been working on trace metal research at MSL for more than 29 years, contributing to studies involving the bioavailability of trace metals in marine ecosystems and effects of trace metals in the sea-surface microlayer. He has managed numerous projects in the field of metal analysis, ranging from sedimentary samples from the Beaufort Sea prior to oil-well drilling to the determination of the effects of dredging around Oakland Harbor, CA. Through his work on these studies, Mr. Apts has gained considerable experience in field sampling and trace metal analytical techniques using ICP/MS.

Ms. Blythe Barbo is the MSL Marketing Information Specialist. She has nearly 9 years experience at the MSL. Some of her duties include proposal coordination and production, marketing materials development, capability information organization, business intelligence tracking, database research, information distribution services, media and public relations, and market and proposal communications.

Mr. Michael Blanton specializes in the areas of water quality and ecotoxicology. Other areas of expertise include strategic planning, experimental design, and ecological surveys/evaluations. Mr. Blanton participated in the Columbia River Comprehensive Impact Assessment, which modeled fate and exposure of contaminants to various trophic levels in the ecosystem and to humans; he has also participated in environmental exposure and risk analysis programs for the Surface Environmental Surveillance Project (SESP), Hanford, Washington. He recently investigated the potential injury to fall Chinook salmon from exposure to chromium releases to the Columbia River and the development of an Information Management System to aid in ESA compliance under FIFRA for new agricultural chemical product registration. He is currently the project manager for an Evaluation of the Environmental Effects of Synthetic – Based Drilling Fluids on Coastal and Marine Waters. Mr. Blanton has 7 years of experience at Battelle Richland and joined MSL in October 1999.

Ms. Susan Blanton joined MSL in October 1999. She previously supported the Pacific Northwest National Laboratory for 6 years as a Science & Engineering Associate II in the Ecology Group within Environmental Technology Division. Her research has focused on diverse salmonid issues in the Columbia and Snake River Basins. She has evaluated fish screening facilities in the Yakima River Basin, supported hydroacoustic fish passage research efforts at Snake and Columbia River hydroelectric projects, studied the effects of gas

supersaturated water on salmonids, contributed to preparation of environmental impact statements, and conducted teacher workshops on numerous aspects of aquatic ecology.

Ms. Elisabeth Smolski Barrows has over 22 years experience working environmental science encompassing project management, analytical organic and inorganic chemistry, marine and freshwater toxicity testing, and field work. She is currently involved with the environmental assessment of dredged material disposal options in New York. She is currently managing projects for the U.S. Army Corps of Engineers. Ms. Barrows has been with Battelle for 15 years, with 9 of those years at MSL.

Dr. Peter Becker, is a physical oceanographer who specialized in the description, analysis, and modeling of physical processes in freshwater distribution and transport in the Arctic Ocean. He is currently a post-doctoral research associate at the MSL, working on the Arctic Nuclear Waste Assessment Project (ANWAP) for the Office of Naval Research. During his position as oceanographic consultant at the University of Washington, from 1974 to 1989, his duties included head oceanographer on over 35 field projects.

Ms. Linda Bingler, marine chemist, has been at MSL for 9 years and serves as a project manager and provides support for sample digestion, distillation, and extraction. She has researched the effective removal of contaminants by thermal and hydrocyclone processing. As part of the World Hydrographic Global Measurement Program (WHP), she managed Battelle's portion of a 60-day cruise from Dutch Harbor to New Caledonia to collect CTD and high-accuracy measurements of anthropogenic and natural tracers. Ms. Bingler has taken shipboard measurements of TCO_2 in seawater using the SOMMA/coulometric system and participated in World Ocean Circulation Experiment cruises as part of the WHP global measurement program. Ms. Bingler has also researched rare earth element nutrient complexation in seawater, determination of formation constants for metal-nutrient complexation, and phytoplankton uptake of rare earth elements over time.

Ms. Amy Borde has been with the MSL since 1995. Her research has focused on wetland ecology, specifically marine habitat assessment and restoration. She has conducted reviews of wetland ecology and policy issues for EPA, contributed to long-term studies of habitat change in PNW estuaries, supported eelgrass restoration efforts in Puget Sound, provided GIS support for numerous studies, and has acted as a teachers assistant for wetland ecology and restoration classes.

Ms. Deborah Coffey is the MSL QA Officer and ES&H Representative. She has more than 16 years of quality assurance experience supporting U.S. EPA and NQA-1 QA Programs at the Corvallis Environmental Research Laboratory, and at Sandia National Laboratories supporting the Waste Isolation Pilot Plant (WIPP) program. She joined Battelle in September 1999. She is responsible for overseeing all QA/QC aspects of the MSL's project performance, such as developing QA planning documents, assessing and improving processes, ensuring that all protocols are followed, and that data are accurately presented. She is an NQA-certified and Lead Auditor and routinely conducts internal QA assessments to verify procedural compliance and data acceptability. Ms. Coffey reports to the Process Quality Department of the Battelle Quality Division and is therefore, independent of MSL.

Dr. Eric Crecelius, the Technical Group Manager of the Marine and Environmental Chemistry Group, has over 20 years of experience in freshwater and marine geochemistry

studies with an emphasis on concentrations, fates, and effects of trace metals. Dr. Crecelius is internationally recognized in the field of marine pollution and trace metal chemistry. He is frequently an invited participant or session chairman for workshops or scientific meetings that deal with arsenic speciation, marine monitoring, and the fate of contaminants in coastal waters.

Dr. Val Cullinan, specializes in the statistical design, analysis, and interpretation of results from multidisciplinary experimental research. Her research has addressed marine resources, as well as agricultural systems, terrestrial and aquatic ecosystems. She specializes in developing statistically efficient sampling designs to detect ecological change at landscape levels of spatial heterogeneity.

Ms, Mary Ann Deuth, is a technical specialist responsible for ongoing analyses for trace amounts of mercury in tissue, sediment, and water. She has been at the MSL for 5 years and she is currently developing proficiency with new mercury analyzers.

Ms. Ann Skillman Drum, has 20 years experience conducting research on biological mechanisms of marine invertebrates and fist infectious diseases and cancer, environmental pathology and toxicology, parasitology, and aquatic animal health management, disease diagnosis and prevention. Her areas of specialization include health management of salmon and invertebrates, relationship of animal health to resource management, development of new aquaculture techniques and aquatic animal drug registration studies conducting under GLP.

Mr. Richard Ecker returned to MSL in March 1999 to serve as the MSL Manager. He is the Associate Director of (CREM) Coastal Resource and Ecosystem Administrative Management , a joint venture of Battelle Duxbury and the MSL. He is also a Battelle Product Line Manager for the Resource and Ecosystem Management Product Line, which is dedicated to finding solutions to complex environmental issues. Prior to his current position as Product Line Manager, he was the Department Manager of the Water and Land Resources Department and Product Line in the Environmental Technology Division managing a department of over 180 staff. He also previously managed the Battelle Marine Sciences Laboratory for 6 years. Dick started with Battelle in 1978 and has been involved in all aspects of the business; business development and marketing, deployment of technology, technical research and line management. Before his career with Battelle, Dick served with the Army Corps of Engineers, San Francisco District where he directed environmental projects.

Mr. Paul Farley, is responsible for high resolution acoustic survey and precision seafloor mapping programs. He has formal training and more than 20 years of experience in side scan sonar operations, high resolution seismic data acquisition, interpretation and bathymetric survey, and satellite and microwave navigation systems. He also supervises the electronics/instrumentation shop where duties include trouble-shooting and repair of various analog and digital instrumentation and to design and construct various equipment as needed.

Mr. Tim Fortman is a technical specialist in organic chemistry who has participated in a variety of environmental pollution monitoring projects such as NOAA's status and trends, the Exxon Oil Spill Herring study, and the EPA's Great Lakes assessment program. He also has worked on all aspects of trace metal and organic contaminant analysis, and set up an automated gel permeation chromatography system used as an advanced cleanup of organic environmental analysis. In addition, he trains and supervises several technicians. Mr. Fortman is the MSL Acid Neutralization Drum Custodian and has been at MSL for 14 years.

Mr. Brian Gruendell's expertise is in biologic oceanography. His recent research has focused on bioaccumulation and toxicity testing using amphipods and bivalve larvae, dredged material evaluation, and field sampling efforts for private and Federal agencies. He also serves as the MSL Hazardous Materials Coordinator (HMC).

Mr. Tom Hausmann recently joined the staff of MSL after 2 years as a Bioremediation Research Assistant Fellow at MSL under the Associated Washington Universities program. He is completing his thesis in Environmental Engineering and he will be providing support for GC/MS analyses for the Marine and Environmental Chemistry Group. Mr. Hausmann is a QA Representative who is trained to perform data reviews.

Mr. Lyle Hibler specializes in studies of contaminant transport in rivers, estuaries, the open ocean, and groundwater. He has developed and applied numerical computer models and processing codes, and has been involved in the statistical and uncertainty analysis associated with numerical algorithms used by these types of models.

Dr. Michael Huesemann, is involved in bioremediation research and project management. His areas of specialization are hazardous waste soil and groundwater bioremediation, including field applications of composting, bioventing, and air-sparging technologies.

Ms. Lara Johnson joined MSL in December of 1999 as a Scientist Engineer Associate I. For the prior 7 months she has supported MSL toxicity tests of marine sediments as an intern through the Associated Western Universities (AWU) program. During her internship she was involved in numerous bioassay and bioaccumulation tests on sediments from various parts of the world. Her experience is shared between technical laboratory skills, data processing, and data analysis. Ms. Johnson is currently working closely with project managers on the same type of testing activities.

Ms. Rhonda Karls, a laboratory supervisor in the toxicology testing laboratory, is responsible for preparation of the laboratory for testing, maintaining water quality instruments, ordering supplies, animal care and maintenance of test organisms, and the conduction of the actual test.

Ms. Nancy Kohn, research scientist, has experience in conducting sediment evaluation studies for the U.S. Army Corps of Engineers and other clients. She has participated in laboratory bioassays and has served in a project management role with the responsibility for planning and leading field sampling efforts and for coordinating sample preparation tasks and laboratory testing schedules. Her recent research has concentrated on understanding the effects of ammonia to benthic organisms, primarily amphipods, under varying environmental conditions.

Ms. Brenda Lasorsa, senior research scientist, supervises the mercury analytical laboratory. She has helped develop methods for total and methyl mercury analysis by cold-vapor atomic fluorescence; sulfide analysis using gas chromatographic techniques, and an acid volatile sulfides analysis system. Ms. Lasosa has been at the MSL for 10 years.

Ms. Mary McGahan, a technician in the Marine and Environmental Chemistry Group, has over 15 years of experience. Her experience includes sample log in and performing preparation and analysis of tissue, water and soil samples.

Ms. Laurie Niewolny, research scientist, is experienced at identifying algae, zooplankton, aquatic insects, and fish and organizing and completing field data collection of water, sediment, and biota. She is also proficient in most laboratory procedures for standard chemical and toxicological water and sediment quality analyses and maintains laboratory records, equipment, chemical solutions, food supplies, water supply, and test organism cultures. Ms. Niewolny has been at the MSL for 4 years, and within the last year supports mercury lab analyses. She is a QA Representative who is trained to perform data reviews.

Mr. James Nimmo is the Manager for Facilities and Operations; he has over 30 years experience in facility operations, and facility and building management. Mr. Nimmo has been at MSL since 1967 and has served as the Project Manager and/or field engineer representative on most MSL construction projects. He has a background in electronics, specializing in airborne radar, navigation, and weapons systems. Mr. Nimmo was previously an electronic and pneumatic instrument specialist for nuclear facilities. He has numerous course completion certificates in environmental engineering, air and hydronic balancing, property conservation (relative to fire and flood construction practices and natural disasters), pneumatic control systems for building heating, ventilation and cooling (HVAC), steam systems, crane-hoist rigging techniques, and safety including national SCUBA certification and boat operation.

Ms. Peg O'Neill, technical specialist, is experienced in the operation of inorganic analytical equipment including atomic absorption and ICP/MS. She is also proficient in distillation and acid digestion (hot plate, water bath, and microwave) sample preparation techniques.

Ms. Meg Pinza, research scientist, has a background in aquatic toxicology. She is currently involved in field sampling and sediment evaluation studies conducted for several district offices of the U.S. Army Corps of Engineers. She has also conducted bioassays on pulp mill effluents to determine effluent quality and compliance with discharge permit standards, and is currently part of the team developing biotechnology for remediation of contaminants in various matrices.

Ms. Jeni Franklin Ross is the point of contact for shipping and receiving and in this role, assigns bar codes for chemical solutions to implement the Chemical Management System (CMS). She has 7 years of experience at MSL and supports travel, accounts payable, and security through the badging process.

Mr. Jan Slater is the Manager for Business Administration. He has been at MSL for 3 years. Prior to that he supported the Pacific Northwest National Laboratory for 7 years as Manager and Sr. Technical Team Lead in various US Department of Energy Programs including the privatization of the Tank Waste Remediation Systems, the Tritium Target Qualification Program, ADPE Procurement, the Global Studies Program, and the Environmental Management Operations. Prior to coming to PNNL, he worked as Bonneville Power Administration as a Sr. Contract and Financial Specialist.

Mr. John Southard, joined MSL in December 1999. He is part of the MSL Dive Team and has been certified since 1993. He is an active SCUBA instructor with National Association of Underwater Instructors (NAUI) and Technical Diving International (TDI). He is familiar with underwater survey activities and has used lines, quadrats, and linear count methods, and has

experience with underwater species identification. Mr. Southard is a collection diver for the State of WA and the Arthur Feiro Marine Laboratory in Port Angeles, WA.

Ms. Karen Steinmaus has 15 years experience in remote sensing and image processing for both environmental and national security applications. From 1983-1987, Karen worked for the Defense Mapping Agency (currently NIMA) in NTM Image Exploitation. At the Pacific Northwest National Laboratory, Karen has focused on the development and application of remote sensing and geographic information system (GIS) technologies for a very wide variety of government and commercial clients. Throughout her career, Karen has emphasized multidisciplinary problem solving, and multisensor data fusion and integration. Karen has contributed to, managed, and developed business for basic, applied, and technology transfer R&D projects, resulting in a very unique opportunity to understand client needs, technology gaps, and future trends in the areas of hyperspectral/multi-spectral image exploitation and multisensor data fusion. Karen holds DOE, DOD and SCI clearances.

Mr. Monte Sula is a Registered Environmental Manager (REM) and a certified Health Physicist (CHP). As the MSL environmental engineer he is in charge of Environmental Waste Operations at MSL. He is also the MSL Radiation Safety Officer (RSO). Mr. Sula has been at MSL for 7 years, and at the Pacific Northwest National Laboratory for the past 20 years. He is responsible for all activities associated with liquid and airborne discharges at the MSL and for work conducted under the laboratory's radioactive materials license.

Ms. Carolyn Suslick is the Data Coordinator for the Marine Chemistry and Ocean Processes Group and the Sample Inventory Coordinator (SIC). She is the custodian of and manages the chemistry data central filing system. She creates and formats data tables and control charts from raw data, tracks data for projects, and assists Program Managers in preparing and editing reports.

Dr. Ronald Thom, a senior research scientist, has 21 years of professional experience as an algologist, wetlands ecologist, toxicologist and fisheries biologist. Dr. Thom specializes in environmental impacts of navigation and marina dredging and dredged material disposal; habitat construction and restoration of marine and estuarine systems; and ecology of fisheries resources in nearshore systems. He also serves as the Technical Group Manager for the Marine Ecological Processes Group.

Dr. Susan Thomas, senior research scientist, is part of the biotechnology team in remediation research that is using fungal organisms for the degradation and removal of contaminants from natural matrices. She has worked in environment assessment and reporting and as environmental impact statement coordinator. She also has laboratory experience in radiation biology, olfactory/taste chemistry, and human DNA synthesis and repair.

Mr. Jeffrey Ward, senior research scientist, has over 15 years experience in environmental studies. He provides management and technical expertise for projects involving toxicity testing, dredged sediment evaluations, analyses of benthic community structure, and environmental impact assessments. He has coordinated and participated in numerous field sampling efforts, and is experienced in a variety of field sampling procedures. Mr. Ward is currently the Technical Group manager for the Toxicology and Risk Assessment Group.

Dr. Dana Woodruff currently conducts research in benthic habitat mapping using side-scan sonar and underwater video. Her background is in remote sensing of coastal and estuarine waters, specializing in optical water quality modeling, in-situ spectral characterization, and remote estimation of water clarity using satellite imagery. She received her Ph.D. in 1996 from the University of North Carolina, where she developed algorithms to predict turbidity in Pamlico Sound, NC, using satellite imagery. Dr. Woodruff recently completed a National Research Council Research Associateship with the National Marine Fisheries Service and has also served as the Southeast Regional Manager for NOAA's CoastWatch Program. Her previous marine research experience has included primary productivity studies from coastal North Carolina to the Sargasso Sea, sewage pollution assessments of coastal sediments using bacterial indicators, king and tanner crab feeding ecology studies in the Bering Sea, and behavioral research on fish and crustaceans relative to oil contamination. Dr. Woodruff was at MSL from 1976 to 1988, when she left to resume her studies. She returned in 1998, and has a total of 13 years at MSL.

Ms. Jordana Wood has been at MSL for 1 year. She supports analyses using the ICP-AES; GFAA; and FIAS for selenium, mercury, and arsenic. Prior to her position at MSL, she was a Supervisor at Battelle Duxbury for ICP-AES and GFAA analyses. She has 4 years of experience as a Supervisor at the EPA's laboratory facilities in Las Vegas, NV and 3 years experience at ICF Kaiser for the same set of analyses.

APPENDIX C

BATTELLE MSL STANDARD OPERATING PROCEDURES

QUALITY ASSURANCE

MSL-Q-001 Maintaining the Master Schedule Sheet
MSL-Q-002 Quality Assurance Inspections of MSL System and Study Activities
MSL-Q-003 Quality Assurance Audits of Reports
MSL-Q-004 Quality of Testing Water and Feed
MSL-Q-005 Quality Assurance Data Audits
MSL-Q-006 Procedures for Control Charting
MSL-Q-007 Procedure for Determining Method Detection Limits
MSL-Q-008 QA Reports to Management
MSL-Q-009 Method Development, Validation, and Implementation

ADMINISTRATION

MSL-A-001 Sample Log-In Procedure
MSL-A-002 Sample Chain of Custody
MSL-A-003 Guidelines for SOP Format and Control
MSL-A-004 Guidelines for Protocol Preparation and Assignment of Study Numbers
MSL-A-005 Deviations From Established Requirements
MSL-A-006 Marine Sciences Laboratory Training
MSL-A-008 Control of Reagents/Solutions, Test/Control Articles and Specimens
MSL-A-009 GLP Study Initiation Requirements
MSL-A-010 Document Control
MSL-A-011 MSL Access Control
MSL-A-012 Procurement
MSL-A-013 Laboratory Accreditation and PE Sample Analysis
MSL-A-014 Sample Container Requests

DOCUMENTATION, RECORDS, REPORTS

MSL-D-001 Recording Data on Data Sheets and Laboratory Notebooks
MSL-D-002 GLP Records Management
MSL-D-003 Archiving of Records, Data and Retired SOPs
MSL-D-004 Data Reporting, Reduction, Back Up, and Archiving

ORGANIC CHEMISTRY

MSL-O-001 Butyltin in Sediments and Tissues
MSL-O-002 Butyltin in Water
MSL-O-003 Identification and Quantification of Polynuclear Aromatic Hydrocarbons by Gas Chromatography/Mass Spectrometry
MSL-O-004 Analysis of Polychlorinated Biphenyls and Chlorinated Pesticides by Gas Chromatography with Electron Capture Detection
MSL-O-005 Stock and Standard Solution Preparation
MSL-O-006 HPLC Cleanup of Sediment and Tissue Extracts for Semivolatile Pollutants
MSL-O-007 Determination of Lipid Content in Tissues
MSL-O-008 Operation and Maintenance of Gas Chromatographs (GC) and Gas Chromatograph/Mass Spectrometer (GC/MS) Systems
MSL-O-009 Extraction and Clean-up of Sediments and Tissues for Semivolatile Organics following the Surrogate Internal Standard Method

MSL-O-010 Extraction and Clean-up of Water for Semivolatile Organics following the Surrogate Internal Standard Method
MSL-O-011 HOC Sampling Media Preparation and Handling; XAD-2 Resin and GF/F Filters
MSL-O-012 Extraction and Cleanup of Resin Cartridges for Polychlorinated Biphenyls and Trans-Nonachlor
MSL-O-013 Extraction and Cleanup of Glass Fiber Filters for Polychlorinated Biphenyls and Trans-Nonachlor
MSL-O-014 PCB Congener Analysis of XAD Resins and GFF Filters Using GC/ECD
MSL-O-015 Identification and Quantification of Polynuclear Aromatic Hydrocarbons by Gas Chromatography/Mass Spectrometry Following EPA Method 8270B Quality Control Criteria
MSL-O-016 Analysis of Polychlorinated Biphenyls and Chlorinated Pesticides by Gas Chromatography with Electron Capture Detection Following EPA Method 8080A Quality Control Criteria

INORGANIC CHEMISTRY

MSL-I-001 APDC Extraction for Trace Metals in Water
MSL-I-003 TAMU Sediment and Tissue Digestion
MSL-I-004 Sediment Evaporation Digestion
MSL-I-005 Hot Nitric Acid Digestion of Sediments and Tissues
MSL-I-006 Mixed Acid Sediment Digestion
MSL-I-007 Nitric Acid and Hydrogen Peroxide Tissue and Sediment Digestion
MSL-I-011 Total Mercury in Solids by CVAF
MSL-I-012 Easily Reducible Mercury in Water by CVAF
MSL-I-013 Total Mercury in Aqueous Samples by CVAF
MSL-I-014 Methylmercury in Aqueous Samples by CVAF
MSL-I-015 Methylmercury in Tissues and Sediments by CVAF
MSL-I-016 Total Mercury in Tissues and Sediment by CVAA
MSL-I-019 Determination of Trace Elements in Water by Stabilized Temperature GFAA Spectrometry
MSL-I-020 Trace Elements in Sediment and Tissues by GFAA
MSL-I-021 Arsenic Speciation in Aqueous Samples
MSL-I-022 Determination of Elements in Aqueous and Digestate Samples by ICP/MS
MSL-I-023 Selenium Speciation in Aqueous Samples
MSL-I-024 Mixed Acid Tissue Digestion
MSL-I-025 Methods of Sample Preconcentration: Cobalt/APDC Co-precipitation and Borohydride Reductive Precipitation for Trace Metals Analysis in Water
MSL-I-026 Use of Laboratory Refrigerators and Freezers
MSL-I-027 Determination of Metals in Aqueous and Digestate Samples by ICP/AES
MSL-I-028 Navy Sample Analysis Plan
MSL-I-029 Determination of Metals in Aqueous and Digestate Samples by GFAA

CONVENTIONAL/GENERAL CHEMISTRY

MSL-C-001	Acid Volatile Sulfide (AVS) and Simultaneously Extracted Metals (SEM) in Sediments
MSL-C-002	Total Volatile Solids
MSL-C-003	Percent Dry Weight and Homogenizing Dry Sediment, Soil, and Tissue
MSL-C-004	pH in Water
MSL-C-005	Total Dissolved Solids
MSL-C-006	Grain Size
MSL-C-007	Total Suspended Solids
MSL-C-008	Total Solids
MSL-C-009	Use and Performance Checks of Balances
MSL-C-010	Calibration and Use of Pipettes
MSL-C-011	Glassware and Equipment Cleaning Procedures
MSL-C-012	Pb^{210} Dating Digestion and Analysis
MSL-C-013	^{137}Cs Analyses by Gamma Counting
MSL-C-015	Preparation of Sediment Porewater for Analysis of Organic Compounds and Metals

WATER QUALITY/INSTRUMENTATION

MSL-W-001	Calibration and Use of pH Meters
MSL-W-002	Calibration and Use of Dissolved Oxygen Meters
MSL-W-003	Calibration and Use of Thermometers
MSL-W-004	Calibration and Use of Refractometers
MSL-W-005	Calibration and Use of LI-COR Light Meter, Model LI-185A
MSL-W-006	Operation of Atlas CFA-3232 Incinerator
MSL-W-007	Determination of Ammonia
MSL-W-008	Routine Water Quality Measures for Toxicity Tests

TOXICITY/BIOLOGICAL TESTING

MSL-T-001 Water and Tissue Sample Collection
MSL-T-002 Animal Receipt, Acclimation, and Holding
MSL-T-003 Test Organism Observations
MSL-T-004 Sediment Bioaccumulation Testing
MSL-T-005 Acute Sediment Toxicity Testing Using Amphipods
MSL-T-006 Solid Phase Flow-Through Bivalve and Worm Test
MSL-T-007 Suspended Particulate Phase Preparation
MSL-T-008 Suspended Particulate Phase Bivalve Larvae Test
MSL-T-009 Suspended Particulate Phase Fish Test
MSL-T-010 Suspended Particulate Phase Mysid Test
MSL-T-012 Sediment Preparation for Chemical and/or Biological Evaluation
MSL-T-013 Suspended Particulate Phase Echinoderm Larvae Test
MSL-T-020 Preparation of Sediment Porewater
MSL-T-021 Preparation of Sediment Porewater for Sulfide Analysis
MSL-T-022 Collection and Handling of Aquatic Surface Microlayer Samples
MSL-T-023 Collection and Handling of Fish Samples Using a Backpack Electroshocker
MSL-T-024 Sediment Bioassay Testing Using *Mysidopsis bahia*
MSL-T-025 Bivalve Larvae Test
MSL-T-026 45-Day Sediment Bioaccumulation Testing
MSL-T-027 Supplemental Feeding for Oysters and Clams
MSL-T-028 Sediment and Water Dosing for GLP Study Number SS-00-0001
MSL-T-029 Use of Hemacytometer
MSL-T-030 Collection of Sediment, Tissue, and Water Samples for Good Laboratory Practices Study SS-00-0001
MSL-T-031 Calibration and Use of Extech Heavy Duty Light Meter
MSL-T-032 Receipt, Holding, and/or Testing of Fish

FACILITIES

MSL-F-001 Seawater and Freshwater System Maintenance
MSL-F-002 Wastewater Discharge Permit Monitoring Procedures
MSL-F-003 Beach Facility Wastewater Control Procedure

SAFETY

MSL-S-001 Safe Diving Practices

WORK PRACTICES

Biological Hazards
Handling, Storing, and Disposing of Samples
Neutralization of Waste, Acid Solutions
Exposure Control Plan

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Marine Sciences Laboratory

QUALITY ASSURANCE MANAGEMENT PLAN

VOLUME 2

Marine and Environmental Chemistry

May 2000

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Battelle Marine Sciences Laboratory
MARINE AND ENVIRONMENTAL CHEMISTRY
QUALITY ASSURANCE MANAGEMENT PLAN
VOLUME 2

Concurrences and Approvals

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Battelle Marine Sciences Laboratory
MARINE AND ENVIRONMENTAL CHEMISTRY
QUALITY ASSURANCE MANAGEMENT PLAN
VOLUME 2

Contents

1.0 SAMPLE CONTROL

Sample handling and tracking with the Marine and Environmental Chemistry Group is covered by two procedures: MSL-A-001, *Sample Log-in Procedure* and MSL-A-002, *Sample Chain of Custody*. The processing of data collected from these activities discussed in procedure MSL-D-004*, Data Reporting, Reduction, Back Up, and Archiving*. The following is a description of the procedure used for receipt and tracking of samples, as well as chain of custody procedures.

1.1 SAMPLE RECEIPT AND LOG-IN

Samples or test organisms are logged in when received in the shipping area. If a Chain of Custody (CoC) form accompanies the samples or test organisms, this form is used to document the date and time of sample receipt and condition. If a CoC form is not shipped with samples, an MSL form will be initiated. For test organisms, a shipping form can be signed and dated and the condition of organisms noted. Cooler temperatures are taken and recorded on the CoC. The sample labels are compared to the CoC and assigned an identification code plus sequential numbering of samples upon arrival. If sample preservation is indicated by the type of analysis or customer specification, samples may be pH adjusted or the pH of a set of subsamples measured to ensure that samples needing to be at a pH of $\leq$ 2 pH units are acidified. This is recorded on the CoC. If samples require filtration, they will be filtered and this information recorded on the CoC form. Samples are counted and assigned an MSL project number (i.e., a central file number and sequential numbering of the set of samples that were received). In some cases samples in a set may arrive on different days depending on the customer's needs and direction.
For analytical samples, an electronic spreadsheet is generated (Login Sheet) listing the customer or sampling identification (ID) number, (sponsor code), the MSL sample ID Number (Battelle code), the sample matrix, the parameters requested, the date of sample collection, and the initials of the person logging in the samples (the Sample Custodian or Designee). The location of sample storage until preparation is also noted.

The "kit" is initiated at this time and generally includes:

- kit initiation date
- assigned central file number
- client name
- project title
- data due date
- work package number (charge code)
- any holding time specifications[1]
- expected sample concentration level, if known (high, moderate, low)
- expected number of samples
- blank correction instructions
- matrix and summary of requested preparation activities, digestions, and analyses
- hazardous material designation
- sample disposal instructions
- project manager signature and date
- Project Workplan Sheet – page 2; specifies sample preparation instructions
- Metals – page 3; specifies matrix and analysis method(s)
- QA/Quality Control (QC) Requirement Sheet – page 4; specifies precision (number of replicates, number of spiked replicates), and accuracy (standard reference material [SRM] type and frequency, number of matrix spikes and concentrations levels] method detection limit [blank and blank spike frequency] and initial calibration verification (ICV) and continuing calibration verification [CCV]) sample frequencies. Project control limits may also be specified or attached. When the customer does not specify project control limits, MSL default limits will be used.

[1] In the absence of customer-specified holding times, holding times defined in standard methods (e.g., EPA 1600 series methods; U.S. Army Corps of Engineers, 1994. Inland Testing Manual. EPA-823-B-94-002. U.S. EPA, Office of Water. Washington, D.C.) may be assigned by the MSL Project Manager.

- The CoC forms are appended to the last sheet of the kit.

An addendum is prepared for anything that is added to the kit. Additions might include the receipt of another sample set to be included in the project sample set, changes to the analysis request, or deletion of some samples to be analyzed.

The kit and any addedums are copied and distributed by the Project Manager to all analysts involved. The original pages of the kit become part of the Chemistry Central File System.

1.2 SAMPLE TRACKING

Sample tracking while samples are in the laboratory is the responsibility of the individual Laboratory Supervisors and the Project Manager. It is the responsibility of the Project Manager to ensure that the samples are given the appropriate priority in the laboratory and that the proper analyses and methods are being performed.

1.3 SAMPLE ARCHIVING AND DISPOSITION

The Project Manager is responsible for proper disposal of leftover sample material. Sample disposition takes three forms: 1) dispose of by appropriate means depending on sample content; 2) return to client; or 3) archive indefinitely. Unless arrangements have been made previously, the samples are generally disposed of by Battelle MSL.

When samples are disposed of by a subcontractor laboratory:

If the subcontractor laboratory or testing facility is responsible for disposing of the samples, the subcontractor is asked to notify the MSL Project Manager before final disposition. The MSL Contact will notify the originator that the samples are scheduled to be destroyed, or will define customer requirements for an extended period of storage.

After destruction of samples, the subcontractor laboratory or testing facility is asked to return a copy of the Chain-of-Custody Form to the MSL Contact for placement in project files. The originator may be forwarded a copy of the final Chain-of Custody documentation if requested.

The MSL Contact records the date of receipt on the Chain-of-Custody Form in the "Received by" section of the form space and indicates the samples were destroyed ending the chain of possession.

When samples are disposed of by the Marine Sciences Laboratory (MSL):

If the laboratory or testing facility is not responsible for disposal of the samples, MSL personnel will obtain custody of the samples from the subcontractor laboratory or testing facility along with the Chain-of-Custody Form.

For returned samples or samples that have never left MSL custody, the MSL Contact will notify the originator that the samples are scheduled to be destroyed, or will define customer requirements for an extended period of storage. If extended storage is not requested, then MSL will dispose of the samples following the guidelines specified in the Pacific Northwest National Laboratory's (PNNL's) Standards-Based Management System (SBMS). This system provides a framework for logging in reagents, chemicals and solutions into the associated Chemical Management System (CMS). This system provides the PNNL Laboratory with the policies and procedures regarding tracking and inventory, storage and disposal of completed samples and analytical wastes, as well as chemical use and disposal. The CMS is used to provide an up-to-date inventory to facilitate emergency response, monitor the location of various classes of materials and identify situations where

acceptable limits for the building/facility determined by the assigned chemical hazard group and fire zone might be exceeded before a violation occurs.

After destruction of samples, MSL personnel responsible for sample destruction returns a copy of the Chain-of-Custody Form to the MSL Contact and the Sample Disposal Log Book entry is updated. The MSL Contact records the date of receipt on the Chain-of-Custody Form in the "Received by" space next to the Sample Custodian's signature and indicates the samples were destroyed ending the chain of possession.

When samples are returned to the customer for disposal:

Samples may be returned to the customer (or the sampling site) by customer request. Samples are shipped to meet Department of Transportation regulations. Generally, the samples are shipped in the same way that they were initially shipped to MSL. Sample disposition should be documented in the central file of each project. The MSL Contact shall ensure that completed Chain-of-Custody Forms are filed in the appropriate project files. The originator may be forwarded a copy of the final Chain-of Custody documentation if requested.

2.0 ANALYTICAL PROCEDURES

All routine analytical laboratory activities are directed and controlled by internal MSL procedures. Where possible, U.S. Environmental Protection Agency (EPA) and consensus methods (e.g., NOAA Status and Trends) are used where the technique is applicable to the sample matrix and the overall objective of the analysis. Table 2.1 lists the analytes and applicable SOPs associated with metals and ancillary measurement analysis. Table 2.2 lists the analytes and applicable SOPs associated with organic analysis. Table 2.3 is a list of the MSL Chemistry procedures and the corresponding EPA or other reference methods upon which the SOPs are based.

TABLE 2.1 List of Analytes and SOPs for Metals and Ancillary Measurements

Analyte	Sed/Tiss Preparation Method(s)	Sed/Tiss Analysis Method(s)	Water Preparation Method(s)	Water Analysis Method(s)
METALS (1)				
Aluminum	MSL-I-003/004/006/024/C-003/015	MSL-I-022/020/027	I-025	MSL-I-022/I-019/027
Antimony	MSL-I-003/004/006/024/C-003/015	MSL-I-022/020/027	I-025	MSL-I-022/I-019/029
Arsenic	MSL-I-003/004/006/024/C-003/015	MSL-I-022/020/027	I-025	MSL-I-022/I-019/021/027/029
Barium	MSL-I-003/004/006/024/C-003/015	MSL-I-022/020/027	I-025	MSL-I-022/I-019/027
Beryllium	MSL-I-003/004/006/024/C-003/015	MSL-I-022/020/027	I-025	MSL-I-022/I-019/027
Cadmium	MSL-I-003/004/006/024/C-003/015	MSL-I-022/020/027	MSL-I-001/-025	MSL-I-022/I-019/027
Chromium	MSL-I-003/004/006/024/C-003/015	MSL-I-022/020/027	I-025	MSL-I-022/I-019/027
Copper	MSL-I-003/004/006/024/C-003/015	MSL-I-022/020/027	MSL-I-001/025	MSL-I-022/I-019/027
Lead	MSL-I-003/004/006/024/C-003/015	MSL-I-022/020/027	MSL-I-001/025	MSL-I-022/I-019/027/029
Manganese	MSL-I-003/004/006/024/C-003/015	MSL-I-022/020/027	I-025	MSL-I-022/I-019/023/027
Mercury	MSL-I-003/004/006/024/ C-003/015	MSL-I-016	MSL-I-012/013	MSL-I-012/013
Methyl Mercury	MSL-I-015	MSL-I-015	MSL-I-014	MSL-I-014
Nickel	MSL-I-003/004/006/024/C-003/015	MSL-I-022/020/027	MSL-I-001/025	MSL-I-022/I-019/027
Selenium	MSL-I-003/004/006/024/025/C-003/015	MSL-I-022/020/027	I-025	MSL-I-022/I-019/023//029
Silver	MSL-I-003/004/006/024/C-003/015	MSL-I-022/020/027	MSL-I-001	MSL-I-022/I-019/027/029
Tin	MSL-I-003/004/006/024/C-003/015	MSL-I-022/020/027	I-025	MSL-I-022/I-019
Thallium	MSL-I-003/004/006/024/C-003/015	MSL-I-022/020/027	I-025	MSL-I-022/I-019/029
Vanadium	MSL-I-003/004/006/024/C-003/015	MSL-I-022/020/027	MSL-I-001	MSL-I-022/I-019/027
Zinc	MSL-I-003/004/006/024/C-003/015	MSL-I-022/020/027	I-025	MSL-I-022/I-019/027
ANCILLARY MEASUREMENTS				
Total Lipids		MSL-O-007		
Grain Size		MSL-C-006		
Percent Moisture		MSL-C-003		
AVS		MSL-C-001		
TVS		MSL-C-002		

(1) List is a partial listing - additional metals can be analyzed

TABLE 2.2 List of Analytes and SOPs for Organics

Analyte	Sediment/Tissue Preparation Method(s)	Sediment/Tissue Analysis Method(s)	Water Preparation Method(s)	Water Analysis Method(s)
ORGANICS				
PAHs				
Acenaphthene	MSL-O-006/009	MSL-O-003/015	MSL-O-010	MSL-O-003/015
Acenaphthylene	MSL-O-006/009	MSL-O-003/015	MSL-O-010	MSL-O-003/015
Anthracene	MSL-O-006/009	MSL-O-003/015	MSL-O-010	MSL-O-003/015
Fluorene	MSL-O-006/009	MSL-O-003/015	MSL-O-010	MSL-O-003/015
Naphthalene	MSL-O-006/009	MSL-O-003/015	MSL-O-010	MSL-O-003/015
Phenanthrene	MSL-O-006/009	MSL-O-003/015	MSL-O-010	MSL-O-003/015
Benzo(a)anthracene	MSL-O-006/009	MSL-O-003/015	MSL-O-010	MSL-O-003/015
Benzo(a)pyrene	MSL-O-006/009	MSL-O-003/015	MSL-O-010	MSL-O-003/015
Benzo(b)fluorene	MSL-O-006/009	MSL-O-003/015	MSL-O-010	MSL-O-003/015
Benzo(g,h,i)perylene	MSL-O-006/009	MSL-O-003/015	MSL-O-010	MSL-O-003/015
Benzo(k)fluoranthene	MSL-O-006/009	MSL-O-003/015	MSL-O-010	MSL-O-003/015
Chrysene	MSL-O-006/009	MSL-O-003/015	MSL-O-010	MSL-O-003/015
Dibenzo(a,h)anthracene	MSL-O-006/009	MSL-O-003/015	MSL-O-010	MSL-O-003/015
Fluoranthene	MSL-O-006/009	MSL-O-003/015	MSL-O-010	MSL-O-003/015
Indeno(1,2,3-cd)pyrene	MSL-O-006/009	MSL-O-003/015	MSL-O-010	MSL-O-003/015
Acenaphthylene	MSL-O-006/009	MSL-O-003/015	MSL-O-010	MSL-O-003/015
Phthalates				
Dimethyl Phthalate	MSL-O-006/009	MSL-O-003	MSL-O-010	MSL-O-003
Diethyl Phthalate	MSL-O-006/009	MSL-O-003	MSL-O-010	MSL-O-003
Di-n-butyl Phthalate	MSL-O-006/009	MSL-O-003	MSL-O-010	MSL-O-003
Butyl benzyl Phthalate	MSL-O-006/009	MSL-O-003	MSL-O-010	MSL-O-003
Bis(2-ethylhexyl)Phthalate	MSL-O-006/009	MSL-O-003	MSL-O-010	MSL-O-003
Di-n-butyl Phthalate	MSL-O-006/009	MSL-O-003	MSL-O-010	MSL-O-003
PCB Congeners (1)				
8 (2,4')	MSL-O-006/009	MSL-O-004	MSL-O-010	MSL-O-004
18 (2,2',5)	MSL-O-006/009	MSL-O-004	MSL-O-010	MSL-O-004
28 (2,4,4')	MSL-O-006/009	MSL-O-004	MSL-O-010	MSL-O-004
44 (2,2',3,5')	MSL-O-006/009	MSL-O-004	MSL-O-010	MSL-O-004
49 (2,2',4,5')	MSL-O-006/009	MSL-O-004	MSL-O-010	MSL-O-004
52 (2,2',5,5')	MSL-O-006/009	MSL-O-004	MSL-O-010	MSL-O-004
66 (2,3',4,4')	MSL-O-006/009	MSL-O-004	MSL-O-010	MSL-O-004
87 (2,2',3,4,5')	MSL-O-006/009	MSL-O-004	MSL-O-010	MSL-O-004
101 (2,2',3,5,5')	MSL-O-006/009	MSL-O-004	MSL-O-010	MSL-O-004
105 (2,3,3',4,4')	MSL-O-006/009	MSL-O-004	MSL-O-010	MSL-O-004
118 (2,3',4,4',5)	MSL-O-006/009	MSL-O-004	MSL-O-010	MSL-O-004
128 (2,2',3,3',4,4')	MSL-O-006/009	MSL-O-004	MSL-O-010	MSL-O-004
138 (2,2',4,4',5,5')	MSL-O-006/009	MSL-O-004	MSL-O-010	MSL-O-004
153 (2,2',4,4',5,5')	MSL-O-006/009	MSL-O-004	MSL-O-010	MSL-O-004
170 (2,2',3,3',4,4',5)	MSL-O-006/009	MSL-O-004	MSL-O-010	MSL-O-004
180 (2,2',3,4',5,5',6)	MSL-O-006/009	MSL-O-004	MSL-O-010	MSL-O-004
183 (2,2',3,4,4',5',6)	MSL-O-006/009	MSL-O-004	MSL-O-010	MSL-O-004

TABLE 2.2 Continued

Analyte	Sediment/Tissue Preparation Method(s)	Sediment/Tissue Analysis Method(s)	Water Preparation Method(s)	Water Analysis Method(s)
PCB/Aroclors(2)				
1242	MSL-O-006/009	MSL-O-004	MSL-O-010/012	MSL-O-004
1248	MSL-O-006/009	MSL-O-004	MSL-O-010/012	MSL-O-004
1254	MSL-O-006/009	MSL-O-004	MSL-O-010/012	MSL-O-004
1260	MSL-O-006/009	MSL-O-004	MSL-O-010/012	MSL-O-004
Organotins				
Tributyltin	MSL-O-001	MSL-O-001/016	MSL-O-002	MSL-O-002/016
Dibutyltin	MSL-O-001	MSL-O-001/016	MSL-O-002	MSL-O-002/016
Monobutyltin	MSL-O-001	MSL-O-001/016	MSL-O-002	MSL-O-002/016
Pesticides				
Aldrin	MSL-O-006/009	MSL-O-004/016	MSL-O-010	MSL-O-004/016
-Chlordane	MSL-O-006/009	MSL-O-004/016	MSL-O-010	MSL-O-004/016
Trans nonachlor	MSL-O-006/009	MSL-O-004/016	MSL-O-010/012	MSL-O-004/016
Dieldrin	MSL-O-006/009	MSL-O-004/016	MSL-O-010	MSL-O-004/016
p,p'-DDT	MSL-O-006/009	MSL-O-004/016	MSL-O-010	MSL-O-004/016
o,p'-DDT	MSL-O-006/009	MSL-O-004/016	MSL-O-010	MSL-O-004/016
p,p'-DDD	MSL-O-006/009	MSL-O-004/016	MSL-O-010	MSL-O-004/016
o,p'-DDD	MSL-O-006/009	MSL-O-004/016	MSL-O-010	MSL-O-004/016
p,p'-DDE	MSL-O-006/009	MSL-O-004/016	MSL-O-010	MSL-O-004/016
o,p'-DDE	MSL-O-006/009	MSL-O-004/016	MSL-O-010	MSL-O-004/016
Endosulfan I	MSL-O-006/009	MSL-O-004/016	MSL-O-010	MSL-O-004/016
Endosulfan II	MSL-O-006/009	MSL-O-004/016	MSL-O-010	MSL-O-004/016
Endosulfan sulfate	MSL-O-006/009	MSL-O-004/016	MSL-O-010	MSL-O-004/016
Heptachlor	MSL-O-006/009	MSL-O-004/016	MSL-O-010	MSL-O-004/016
Heptachlor epoxide	MSL-O-006/009	MSL-O-004/016	MSL-O-010	MSL-O-004/016
Lindane	MSL-O-006/009	MSL-O-004/016	MSL-O-010	MSL-O-004/016

(1) List is a partial listing - up to 100 congeners can by analyzed
(2) List is a partial listing - additional aroclors can be analyzed

TABLE 2.3 List of Chemical Analytical Methods Proposed and Their Similar or Equivalent Methods

MSL Procedure	Method Used as Basis for Procedure
MSL-C-001, Acid Volatile Sulfide (AVS) and Simultaneously Extracted Metals (SEM) in Sediments and Aqueous Samples	Allen *et. al.,* 1990
MSL-C-002, Total Volatile Solids	Standard Methods 1995 (Method 2540 E)
MSL-C-003, Percent Dry Weight and Homogenizing Dry Sediment, Soil, and Tissue	EPA 1979 (Method 160.3)
MSL-C-004, pH in Water	EPA 1979 (Method 150.1)
MSL-C-005, Total Dissolved Solids	Standard Methods 1995 (Method 2540 C)
MSL-C-006, Grain Size	Plumb, 1981
MSL-C-007, Total Suspended Solids	Standard Methods 1995 (Method 2540 D)
MSL-C-008, Total Solids	Standard Methods 1995 (Method 2540 B)
MSL-C-015, Preparation of Sediment Porewater for Analysis of Organic Compounds and Metals	NA
MSL-I-001, APDC Extraction for Trace Metals in Water	EPA 1996c (Method 1640)
MSL-I-003, TAMU Sediment and Tissue Digestion	NOAA 1993
MSL-I-004, Sediment Evaporation Digestion	NA
MSL-I-005, Hot Nitric Acid Digestion of Sediment and Tissue	NA
MSL-I-006, Mixed Acid Sediment Digestion	Kostas, O'Conner, and Crecelius 1997
MSL-I-007, Nitric Acid and Hydrogen Peroxide Tissue and Sediment Digestion	EPA 1991 (Method 200.3)

TABLE 2.3 Continued

MSL Procedure	Method Used as Basis for Procedure
MSL-I-011, Total Mercury in Solids by CVAF	Bloom and Crecelius, 1983
MSL-I-012, Easily Reduceable Mercury in Water by CVAF	Bloom and Crecelius, 1983
MSL-I-013, Total Mercury in Aqueous Samples by CVAF	Bloom and Crecelius, 1983; EPA 1996b (Method 1631)
MSL-I-014, Methylmercury in Aqueous Samples by CVAF	Bloom, 1989; EPA 1998a (Method 1630)
MSL-I-015, Methylmercury in Tissues and Sediments by CVAF	Bloom, 1989
MSL-I-016, Total Mercury in Tissues and Sediments by CVAA	EPA 1991 (Methods 245.5 and 245.6)
MSL-I-020, Trace Elements in Sediments and Tissues by GFAA	EPA 1991 (Method 200.9)
MSL-I-021, Arsenic Speciation in Aqueous Samples	EPA 1998b (Method 1632)
MSL-I-022, Determination of Elements in Aqueous and Digestate Samples by ICP/MS	EPA 1991 (Method 200.8), EPA 1996d (Method 1638)
MSL-I-023, Selenium Speciation in Aqueous Samples	EPRI 1986
MSL-I-025, Methods of Sample Preconcentration: Cobalt/APDC Coprecipitation and Borohydride Reductive Precipitation for Trace Metals Analysis in Water	Brugmann *et. al.*, 1983
MSL-I-027, Determination of Metals in Aqueous and Digestate Samples by ICP/AES	EPA 1994 (EPA 200.7); EPA 1997 (SW-846 Method 6010B)
MSL-I-029, Determination of Trace Elements in Water by Stabilized Temperature GFAA	EPA 1991 (Method 200.9)
MSL-O-001, Butyltins in Sediment and Tissue	Unger *et. al.,* 1986
MSL-O-002, Butyltins in Water	Unger *et. al.,* 1986

TABLE 2.3 Continued

MSL SOP	Method Used as Basis for SOP
MSL-O-003, Identification and Quantification of Polynuclear Aromatic Hydrocarbons by GC/MS	EPA 1996a (Method 8270C)
MSL-O-004, Analysis of PCBs and Chlorinated Pesticides by GC/ECD	EPA 1996a (Method 8081A)
MSL-O-006, HPLC Cleanup of Organic Extracts	Krahn *et. al.,* 1988
MSL-O-007, Determination of Lipid Content in Tissues	Bligh & Dyer, 1959
MSL-O-009, Extraction and Cleanup of Sediment and Tissue for Semivolatile Organics following the Surrogate Internal Standard Method	NOAA 1993
MSL-O-010, Extraction and Cleanup of Water for Semivolatile Organics following the Surrogate Internal Standard Method	NOAA 1993
MSL-O-012 Extraction and Cleanup of Resin Cartridges for Polychlorinated Biphenyls and Trans-Nonachlor	EPA 1986
MSL-O-016, Analysis of PCBs and Chlorinated Pesticides by GC/ECD Following EPA Method 8080A Quality Control Criteria	EPA 8080A

3.0 EQUIPMENT MAINTENANCE AND CALIBRATION

3.1 PREVENTATIVE MAINTENANCE

Equipment is serviced regularly by qualified individuals, either trained in-house personnel or through service contracts with the manufacturer or an authorized representative. Written records of all instrument maintenance, calibration, testing, and inspection are maintained. Maintenance records contain a description of the operation or problem, the remedial action taken (if necessary), date, the person responsible, and where applicable, documentation of the instrument's return to analytical control. Each major instrument listed in Appendix A has it's own logbook used to document the preventative maintenance.

3.2 EQUIPMENT CALIBRATION

Calibration procedures are performed on each piece of analytical equipment prior to use. Requirements for specific levels and frequency of calibration are described in the procedures specific for the equipment and methods that are being used. These criteria are summarized in Table 3.1. Note that these are the minimum requirements and project specific requirements may be different. All raw calibration data are kept in the data files and are traceable to sample runs. Corrective actions when calibration criteria are not met are described in section 5.0 of this document and in the specific procedures.

Initial Calibration Verification

After instrument calibration, an initial calibration verification (ICV) sample should be run to verify instrument control. Normally, this check will consist of running a standard reference material (SRM) or one of the same standards that were used for the initial calibration. For samples that are to be analyzed for the Navy, or when requested by a client, a secondary source ICV shall be run prior to running any samples. This ICV will be a standard from a different source than those used in the initial calibration.

Continuing Calibration Verification

Continuing calibration verification (CCV) samples shall be run at the frequency described in the SOP for each method. Analysts will attempt to run CCVs such that they bracket the analytical range of the samples run in the analytical batch. If CCVs do not bracket the samples, the data will be flagged.

TABLE 3.1 Marine and Environmental Chemistry Calibration Procedures

Equipment	SOP No./Section	Parameters	Description [a]	Criteria
GC/ECD	MSL-O-004/ Sec. 4.1.1	PCBs, chlorinated pests	4 pt calibration	The RSD[b] of the RRF[c] $\leq$ 30% for each analyte
GC/MS	MSL-O-003/ Sec. 5.1.1	PAH, phthalates	3 pt calibration	The RSD of the RRF $\leq$ 30% for each analyte
GC/FPD	MSL-O-001/002/ Sec. 5.1 and 6.1	Butyltins	4 pt calibration	The RSD of the RRF $\leq$ 30% for each analyte
HPLC	MSL-O-006	Semivolatile clean-up	only set collection windows	Not used for quantitation
GFAA	MSL-I-029/Sec 5.4.2	Metals in water, sediment and tissue	3 pt calibration	$r^2 \geq 0.995$
ICP/MS	MSL-I-022/ Sec 5.0	Metals in water, sediment	3 pt calibration	$r^2 \geq 0.995$
ICP/AES	MSL-I-027/Sec. 5.4.2	Metals in water, sediment value and tissue	1 pt. calibration	ICV within 10% of concentration
CVAA	MSL-I-016/ Sec. 4.4.1 and 5.1	Total Hg in sediment and tissue	4 pt calibration	$r^2 \geq 0.995$
CVAF	MSL-I-012/013/014/015	Total Hg in water and MeHg in water, sediment and tissue	4 pt calibration	$r^2 \geq 0.995$
PID	MSL-C-001	Acid Volatile Sulfides	3 pt calibration	$r^2 \geq 0.99$

(a) Minimum number of calibration points
(b) RSD = relative standard deviation
(c) RRF = Relative Response Factor

4.0 QUALITY CONTROL

The characteristics used to define data quality are accuracy, precision, completeness, comparability, representativeness and sensitivity (limits of detection). The definition and application of these parameters are discussed below.

4.1 LIMITS OF DETECTION

Method detection limits (MDLs) are determined for all parameters for a number of different matrices. The matrices generally used for MDL studies are freshwater collected from the in-house deionized water system, filtered seawater from Sequim Bay, Sequim Bay or other clean sediment, and *Macoma* tissue. The method used to determine MDLs is covered in procedure MSL-Q-007, *Procedure for Determining Method Detection Limits*. Briefly, MDLs are determined by spiking a minimum of 7 replicate matrices with low levels of the analytes of interest. MDLs are calculated by multiplying the standard deviation of the replicate results by the student t value (99th percentile) for the number of replicates analyzed. Limits of quantitation may also be reported on request as more conservative estimates of detection limits and are defined as 10 times the standard deviation of the replicate analyses. MDL studies should be performed annually.

Tables 4.1 and 4.2 show representative MDLs for the majority of parameters analyzed at Battelle and for a variety of matrices. Since MDLs change yearly and sometimes are performed specifically for individual projects, these exact MDLs are not used to report all data, however; they give a good approximation of the level of detection capable for the various parameters using the methods specified. Because the types of matrices actually analyzed at Battelle vary quite significantly, the MDLs determined on representative matrices may only be estimates of actual detection limits achievable. In addition, MDLs will change if insufficient sample is received by the MSL, if sample matrix interference dictates higher detection limits, or if modifications to existing methods are requested by the client.

TABLE 4.1 Typical Inorganic MDLs

	SEDIMENT (g/g dry wt.)			TISSUE (g/g dry wt.)			WATER (g/L)			
METALS	**ICP/MS**	**GFAA**	**CVAA/F**	**ICP/MS**	**GFAA**	**APDC/ CVAA/F**	**ICP/MS**	**ICP/MS**	**GFAA**	**CVAA/F**
Aluminum	1.0	1.0	NA	1.8	2.0	NA	0.06	NA	2.0	NA
Antimony	0.05	0.50	NA	0.04	0.2	NA	0.009	NA	2.9	NA
Arsenic	0.60	1.0	NA	0.04	0.03	NA	0.04	0.003	1.0	NA
Barium	0.10	10	NA	0.04	2.0	NA	0.006	NA	5.0	NA
Beryllium	0.10	0.30	NA	0.2	0.10	NA	0.05	NA	0.20	NA
Cadmium	0.024	0.03	NA	0.03	0.10	NA	0.02	0.003	0.05	NA
Chromium	0.50	0.50	NA	0.25	0.10	NA	0.04	NA	0.10	NA
Copper	0.70	0.10	NA	0.014	0.10	NA	0.01	0.03	0.50	NA
Lead	0.75	0.50	NA	0.02	0.10	NA	0.005	0.03	1.0	NA
Manganese	0.65	0.50	NA	0.63	0.10	NA	0.001	NA	0.50	NA
Mercury	NA	NA	0.009	NA	NA	0.02	NA	NA	NA	0.0002
Methylmercury	NA	NA	0.00006	NA	NA	0.005	NA	NA	NA	0.00004
Nickel	1.2	0.50	NA	0.02	0.10	NA	0.03	0.06	1.5	NA
Selenium	0.25	0.27	NA	0.12	0.40	NA	0.39	NA	0.78	NA
Silver	0.02	0.02	NA	0.03	0.05	NA	0.004	0.005	0.50	NA
Tin	0.05	1.0	NA	0.0	0.50	NA	0.02	NA	2.0	NA
Thallium	0.02	0.50	NA	0.02	0.20	NA	0.004	NA	1.0	NA
Vanadium	1.0	NA	NA	0.10	NA	NA	0.02	NA	NA	NA
Zinc	3.3	1.0	NA	0.10	0.50	NA	0.04	NA	0.55	NA

APDC Ammonium pyrrolidinedithiocarbamate
ICP/MS Inductively Coupled Plasma Mass Spectrometry
GFAA Graphite Furnace Atomic Absorption
CVAA/F: Cold Vapor Atomic Absorption/Fluorescence

TABLE 4.2 Typical Organic MDLs

PARAMETER	SEDIMENT (μg/kg dry wt)	TISSUE (μg/kg wet wt)	WATER (ng/L)
PAHS			
Naphthalene	0.23	1.85	2.8
Dimethyl Phthalate	10.8	2.91	NA
Acenaphthylene	0.43	0.55	30.7
Acenaphthene	0.39	1.39	3.6
Diethyl Phthalate	24.5	76.6	NA
Fluorene	0.53	1.28	8.8
Phenanthrene	3.51	2.67	15.7
Anthracene	0.72	2.25	17.0
Di-n-butyl Phthalate	21.4	7.68	NA
Fluoranthene	0.45	3.10	8.7
Pyrene	0.52	2.79	7.9
Butyl benzyl Phthalate	15.3	6.02	NA
Benzo(a)anthracene	0.19	0.90	3.2
Chrysene	0.52	1.74	3.4
Bis(2-ethylhexyl) Phthalate	45.0	17.0	NA
Di-n-octyl Phthalate	24.1	5.64	NA
Benzo(b)fluoranthene	0.63	1.14	14.5
Benzo(k)fluoranthene	0.44	1.50	14.8
Benzo(e)pyrene	1.17	1.30	19.5
Benzo(a)pyrene	0.85	1.28	7.5
Perylene	1.30	1.35	2.9
Indeno(1,2,3-cd)pyrene	0.84	1.53	11.8
Dibenzo(a.h)anthracene	0.51	1.22	13.1
Benzo(g,h,i)perylene	0.55	1.07	11.4

TABLE 4.2 Typical Organic MDLs (continued)

PARAMETER	SEDIMENT (μg/kg dry wt)	TISSUE (μg/kg wet wt)	WATER (ng/L)
PESTICIDES			
Hexachlorobenzene	0.64	0.13	1.18
a-BHC	0.45	0.18	2.00
G-BHC	0.28	0.13	1.23
Heptachlor	0.08	0.19	1.02
Aldrin	0.27	0.13	0.76
b-BHC	0.45	0.18	2.00
d-BHC	0.45	0.18	2.00
Heptachlor Epoxide	0.39	0.13	2.14
2,4'-DDE	0.85	0.26	0.74
Endosulfan I	0.45	0.18	2.00
g-Chlordane	0.45	0.18	2.00
a-Chlordane	0.64	0.10	1.93
Trans Nonachlor	0.29	0.15	0.57
4,4'-DDE	0.18	0.19	0.84
Dieldrin	0.26	0.52	0.36
2,4'-DDD	0.26	0.25	0.98
Endrin	0.45	0.18	2.00
4,4'-DDD	0.33	0.26	0.28
Endosulfan II	0.45	0.18	2.00
4,4'-DDT	0.94	0.15	0.50
Endrin Aldehyde	0.45	0.18	2.00
Endosulfan Sulfate	0.45	0.18	2.00
Methoxychlor	0.45	0.18	2.00
Mirex	0.28	0.20	6.29
Endrin Ketone	0.45	0.18	2.00
T-Chlordane	5.0	5.0	5.0
Toxaphene	20	20	20

TABLE 4.2 Typical Organic MDLs (continued)

PARAMETER	SEDIMENT (μg/kg dry wt)	TISSUE (μg/kg wet wt)	WATER (ng/L)
PCB/CONGENERS			
Range	0.06 - 3.05	0.06 - 0.28	0.17 - 1.15
AROCLORS			
1242	3.16	6.95	NA
1248	3.16	6.95	NA
1254	3.16	6.95	NA
1260	3.16	6.95	NA
BUTYLTINS			
Tributyltin	0.48	0.37	3.07
Dibutyltin	0.56	1.39	12.0
Monobutyltin	1.82	1.97	10.8

NA = Not Applicable

4.2 DATA QUALITY OBJECTIVES

The quality control (QC) measurements that are performed during the chemical analysis of the sediments, waters and tissues are outlined in each applicable analytical SOP. The precision and accuracy objectives specified in the SOPs are based on standard method performance information (when available) and historical laboratory performance but may change based on project specific criteria. When required by the client or MSL project manager, other QC checks for accuracy, precision, comparability and completeness shall be applied to each batch of samples. Corrective actions when data quality objectives (DQOs) are addressed in Section 5.0.

4.2.1 Precision

Precision measures the similarity of individual measurements of the same property, usually under prescribed similar conditions.

Within the Marine Chemistry and Ocean Processes Group, measures of analytical precision will be determined by the analysis of laboratory replicates or matrix spike/matrix spike duplicate recoveries. Duplicates are normally performed unless more are requested by the client. Laboratory replicates will be prepared by homogenizing and splitting a sample in the laboratory, and carrying the subsamples through the entire analytical process. Precision can be expressed in terms of relative percent difference (RPD) or relative standard deviation (RSD).

For replicates where duplicates are performed, RPD will be used:

$$RPD = \frac{C_1 - C_2}{[(C_1 + C_2)/2]} \times 100$$

where RPD = relative percent difference
C_1 = larger of the two observed values
C_2 = smaller of the two observed values

For replicates where triplicates or more are performed, RSD (coefficient of variation) will be used:

$$RSD = \frac{s}{m} \times 100$$

where RSD = relative standard deviation
s = standard deviation of replicates
m = mean of replicates

4.2.2 Accuracy

Accuracy is a measure of the bias of a system or measurement. It is the closeness of agreement between an observed value and an accepted value.

Within the Marine and Environmental Chemistry Group, accuracy of chemical analysis will be determined [for each matrix of interest (sediment, tissue and seawater)] through the analysis of matrix spikes, surrogate internal standards, method blanks and, when available, SRMs. SRMs are materials that have been certified by a recognized authority [e.g., National Institute of Standards and Technology (NIST)] and

which are treated and analyzed as an actual sample. Matrix spikes will be performed by adding a known quantity of target analytes into a sample and preparing and analyzing the sample the same as a regular sample. Surrogate internal standards will be spiked into each sample for organics analyses just prior to extraction and will be used to monitor the method performance. Method blanks will be used to measure contamination associated with laboratory processing and analyses.

For measurements where matrix spikes are used, percent recovery will be used to assess accuracy:

$$\%R = \frac{S - U}{C_{sa}} \times 100$$

where %R = percent recovery
S = measured concentration in spiked aliquot
U = measured concentration in unspiked aliquot
C_{sa} = actual concentration of spike added

For situations where a SRM is used, percent difference or percent recovery will be used:

$$PD = \frac{C_1 - C_2}{C_2} \times 100$$

where PD = percent difference
C_1 = measured value
C_2 = certified or consensus value

$$\%R = \frac{C_1}{C_2} \times 100$$

where %R = percent recovery
C_1 = measured value
C_2 = certified or consensus value

4.2.3 Representativeness

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

Representativeness will be addressed primarily by the proper handling and storage of samples and analysis within the specified holding times so that the material analyzed reflects the material collected as accurately as possible. Representativeness of data will be discussed, when appropriate, in deliverable reports.

4.2.4 Comparability

Comparability expresses the confidence with which one data set can be compared to another. Comparability will not be quantified, but will be addressed through the use of laboratory methods that are based on EPA or other recognized methods. The use of standard reporting units also will facilitate comparability with other data sets. Comparability of other data will be discussed, when appropriate, in deliverable reports.

4.2.5 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions.

Target completeness values are 100% for chemical sample analysis.

4.3 HOLDING TIMES AND PRESERVATION

Holding times for analytical chemistry typically begin with the day of sample collection. These holding times and requirements are listed in Table 4.3. Because MSL can not control the fate of samples prior to receiving them, MSL calculates holding times from the time of sample receipt. However, sample collection data are recorded and holding times can be assessed from both the date of sample collection and the date of sample receipt, depending on customer preference.

TABLE 4.3 Sample Holding Times and Preservation

Analysis	Preservation	Holding Time
Sediment*		
Metals (except Hg)	freeze dried at room temp. or frozen at -20 °C	6 months
Mercury	freeze dried at room temp. or frozen	28 days(a)
Organic Compounds	4 °C / -20 °C	30 days(b) to extraction; 40 days (to analysis after extraction)
Grain Size	4 °C	6 months
Tissue*		
Metals (except Hg)	freeze dried at room temp. or frozen at -20 °C	6 months
Mercury	freeze dried at room temp. or frozen	28 days(a)
Organic Compounds	4 °C / -20 °C	30 days(b) to extraction; 40 days (to analysis after extraction)
Water		
Metals (except Hg)	<2 pH with HNO_3/room temp.	6 months (Hg 28 days)
Organic Compounds	4 °C	7 days to extraction; 40 days (to analysis after extraction)

(a) If samples are freeze dried, then samples can be held for up to 6 months.

(b) Two references state that if sediments and tissues are held frozen (-20 °C), then holding times for chemical analysis may be extended up to 6 months: (1) Puget Sound Estuary Program, *Recommended Guidelines for Measuring Organic Compounds in Puget Sound Sediment and Tissue Samples*, EPA, December 1989; and (2) EPA, *Analytical Methods for U.S. EPA Priority Pollutants and 301(h) Pesticides in Estuarine and Marine Sediments*, EPA, May 1986.

* After receipt at the laboratory, sediment and tissue samples for analysis of metals will be held refrigerated (4 °C ± 2 °C) until freeze dried.

4.4 CONTROL CHARTS

Control charts are used to assess quality control (QC) efforts in the laboratory by graphically presenting the variability over time of the various analyses performed. The control charts produced are theoretically based on normally-distributed measurements and short-term variation. The data that are presented in a control chart may vary with the analysis, information sought, the amount of data available, and customer specifications. Details of the control charting process used at the MSL are covered in procedure, MSL-Q-006, *Procedures for Control Charting*. A brief description of the methods used, the criteria used for assessing out of control events, and the administration of the control charts is presented here.

4.4.1 Control Chart Methodology

The control charts produced are based on normally distributed measurements and short-term variation. Precision is charted over time by calculating a mean recovery for the control sample parameters and then establishing upper and lower warning and control limits. The warning limit is defined as $\pm 2\sigma$ and the control limits are defined as $\pm 3\sigma$. The control samples used for organic parameters are blank spikes and for inorganic parameters results from the analyses of a standard reference material are plotted. A minimum of 20 points are used to set the initial control limits for each parameter.

SRMs used as inorganic control samples are generally obtained from either NIST or the National Research Council of Canada (NRCC). All certificates of accepted values for the SRMs are kept in a central SRM log book by inorganic laboratory supervisor. Inorganic SRMs are available for water, sediment and tissue and separate control charts for each SRM analyzed will be maintained.

4.4.2 Criteria for Assessing Out of Control Events

The laboratory process for a particular analyte will be considered out of control whenever, as a minimum, any one of the following conditions is demonstrated:

1. Any one point is outside of the control limits;
2. Any three consecutive points are outside of the warning limits;
3. Any eight consecutive points are on the same side of the centerline;
4. Any six consecutive points are such that each point is higher or lower than its immediate predecessor;
5. Any obvious cyclic pattern is seen in the points.

When any one of the situations listed above occurs, it is the responsibility of the appropriate laboratory supervisor to notify the MSL QA Officer and Project Manager so that appropriate corrective actions can be determined and the situation documented by filling out a Quality Problem Report form and attaching a copy to the control chart. Details regarding the procedure and information required on a Quality Problem Report form are described in procedure MSL-A-005, *Deviations from Established Requirements.*

4.4.3 Administration of Control Charts

One set of control samples (e.g. one set of blank spikes for organic parameters and one SRM for inorganic parameters) is analyzed with each batch of samples, with a batch consisting of no more than 20 samples. Control charts are produced quarterly by the data manager and distributed to the laboratory supervisors and appropriate project managers. Project-specific requirements may have a greater frequency, or may require that control be prepared only for control samples run with project-specific samples. Table 4.4 lists the minimum numbers of parameters to be charted.

Note that control charts are only used for monitoring blank spikes and SRMs. Control limits for matrix spikes, replicate analyses, blank analyses etc. are generally defined by the project guidelines. If available, standard EPA control limits are used. Additional or alternate compounds may be charted if necessary.

TABLE 4.4 Control Chart Parameters

Matrix	QC Sample	Analyte and/or Method
	ORGANICS	
Matrix	Parameter	Compounds
Water or Sediment/Tissue	PAHs	Anthracene, Benzo(a)pyrene
Blank Spikes	PCBs	Aroclor 1254
	PCBs	2 Congeners or Total PCB
	Pesticides	Dieldrin
	Butyltins	Tributyltin
	INORGANICS	
Matrix	Metals/ Method	
Water (fresh)	Cd, Cu, Zn, Pb/ ICP-MS,	
Sediment (estuarine)	Cd, Cu, Zn, Pb/ ICP-MS or GFAA, Hg/CVAA	
Tissue (shellfish)	Cd, Cu, Zn, Pb/ ICP-MS or GFAA, Hg/CVAA	

5.0 CORRECTIVE ACTION

The need for corrective action may be identified by the technical staff during the course of their work or through assessments or data audits. Each individual performing laboratory or data processing activities will be responsible for notifying the appropriate supervisory personnel of any circumstance that could affect the quality or integrity of the data.

5.1 DEVIATIONS

All deviations from approved procedures, project planning documents or this QAMP will be documented. Depending on the severity of the deviation, the MSL QA Officer and the Project Manager will determine how the deviation will be documented through

- use of a Quality Problem Report (QPR) form (Exhibit 12.1 of Volume 1) per MSL-A-005, *Deviations from Established Requirements*;
- documented as part of the narrative summary provided to the customer, and
- documented directly on the raw data.

The MSL QA Officer and the Project Manager will determine if there is a formal deviation when one or more control limits are exceeded in a data set. In some cases, the customer may be involved in these discussions. Deviations from project control limits will be identified in the narrative accompanying the data set or package or in a letter to the customer, and the impact of the deviation addressed. The following are guidelines to resolving deviations identified within the Marine and Environmental Chemistry Group:

- When sample integrity is compromised or questionable (e.g., mislabeling, broken or leaking sample containers, improperly preserved samples, expiration of sample holding times), it is the responsibility of the staff who identify the problem to bring it immediately to the attention of the Project Manager or Technical Group Leader for resolution.
- In the event of an instrument problem, it is the responsibility of the operator to attempt to correct the problem (e.g., recalibrate the instrument). If the problem persists or cannot be identified, the issue should be brought to the attention of the Technical Group Leader for resolution.
- Corrective actions for results outside established DQOs are addressed in section 5.2.

5.2 CORRECTIVE ACTION FOR DATA OUTSIDE OF CONTROL LIMITS

It is the responsibility of the analyst to monitor QC sample results. Results outside the established criteria in method procedures or project specific criteria will be brought to the attention of the Laboratory Supervisor and Project Manager who will determine and document the appropriate corrective action. The corrective actions may include, but are not limited to, review of data and calculations, flagging of suspect data (flagging requirements are addressed in Section 6.0) or re-extraction and/or re-analysis of individual or entire batches of samples. Documentation may take the form of flagging the QC data and/or sample data in the report. The form of documentation is project specific, but at a minimum, the QC data that is outside the established criteria shall be flagged. In addition, during the process of data review performed by the MSL QA Officer or representative as per procedure, MSL-Q-005, *Quality Assurance Data Audits*, the QC data of concern my be required to be addressed in the narrative to the customer accompanying the sample data.

6.0 DATA REPORTING

All reported data will be validated and verified in accordance with Section 10 of Volume 1. Chemistry data and all accompanying QC data will be reported as tables of validated data points for analysis. Reporting limits are defined as MDLs or, when required by a client, target detection limits. When reporting data, the following example data flags will be used where appropriate:

U Analyte not detected at or above the detection limit shown

J Analyte detected below the detection limit; concentration reported may be an estimate

B Analyte detected in sample is less than 5 times the blank value

E Analyte concentration estimated because of matrix interference in sample

X Analyte quantified outside of the calibration range of the instrument

D Analyte determined from diluted sample

In addition, all QC data that falls outside established control limits will be flagged.

7.0 REFERENCES

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APPENDIX A

MARINE CHEMISTRY EQUIPMENT LIST

The following is a list of the major pieces of equipment in both the organic and inorganic chemistry laboratories. This list is intended to demonstrate the types of equipment available and will be revised when the QAMP is revised, but not each time equipment or instruments are added or deleted.

Balances			
Make	**Model**	**Serial Number**	**Location MSL 5**
Sartorius	B3100P	40019183	118
Mettler	AE50	M21198	118
Fisher/Denver	XL300	09630	126
Denver	XP-300	990131	126
Ohaus	CT200	CDO3172	126
Mettler	PE3600	D56329	215
Sartorius	BP3100S	50806575	223
Mettler	AC100	A89515	231
Sartorius	LC1200S	10606711	231

ORGANIC CHEMISTRY EQUIPMENT			
Measures	**Description**	**Serial or ID Number**	**Location MSL 5**
Determination of TBT, alkanes, phosphorus and sulfur compounds	Hewlett Packard 5890 Gas Chromatograph/ Flame Photometric Detector/ Flame Ionization Detector	WB73809	223
PAHs and phthalates	Hewlett Packard 5890 Gas Chromatograph/Model 5970 Quadropole Mass Selective Detector	N821982	215
Chlorinated compounds, PCBs and pesticides	Varian Star 3600 CX Gas Chromatograph/Electron Capture Detector/Flame Ionization Detector	N830047	223
Presently idle	VG Fison Model TRIO 1000 Gas Chromatograph/Liquid Chromatograph/Mass Spectrometer with Thermospray/Plasmaspray Interface	N828035	215
Sample clean up, Gel permeation chromatography, explosives, and PAHs	High Performance Liquid Chromatograph (HPLC) system with autosampler, UV/Florescence Detector	WD28663	223
Sample clean up, Gel permeation chromatography, explosives, and PAHs	High Performance Liquid Chromatograph (HPLC) system with autosampler, UV/Florescence Detector	N828182	114

INORGANIC CHEMISTRY EQUIPMENT			
Measures	**Description**	**Serial or ID Number**	**Location MSL 5**
Metals	Perkin-Elmer Elan 5000 inductively coupled plasma mass spectrometer (ICP-MS) Perkin-Elmer Elan 6100 inductively coupled plasma mass spectrometer (ICP-MS)	WDO8519 PT06550	227
Mercury and Methylmercury	Cold Vapor Atomic Fluorescence Unit - In-house design - #1	PT08031	126
Mercury and Methylmercury	Cold Vapor Atomic Fluorescence Unit - In-house design - #2	N830368	126
Mercury and Methylmercury	Cold Vapor Atomic Fluorescence Unit - In-house design - #3	R101823	126
Metals	Perkin-Elmer Optima 3000 ICP-AES	N830377	114
Metals	GFAA Perkin-Elmer 5100 ZL graphite furnace	N830371	114
Metals	Dionex 4500i, Ion Chromatograph, autosampler, conductivity detector and UV/VIS Detector	WB67819	114
Atomic Absorption Spectrometers			
Metals	Perkin-Elmer Model 5100 Zeeman-effect graphite furnace	N830372	222
Metals	Perkin-Elmer Model 5000 with Zeeman-effect graphite furnace	WB73815	222
Mercury (back up only)	Cold Vapor Atomic Absorption Units - Lab Data Control - #1	WA71764	126
Mercury (back up only)	Cold Vapor Atomic Absorption Units - Lab Data Control - #2	WA26316	126
Mercury (back up only)	Cold Vapor Atomic Absorption Units - Lab Data Control - #3	N822042	126
Mercury	Thermo Separation Products (TSP) 3200 Automated Mercury Analyzer	R101553	126

GFAA Graphite Furnace Atomic Absorption
HPLC High Performance Liquid Chromatograph
ICP-MS Inductively Coupled Plasma (Emissions) – Mass Spectrometry
ICP-AES Inductively Coupled Plasma – Atomic Emissions Spectrometry
PAH Polycyclic Aromatic Hydrocarbons
PCB Polychlorinated Biphenyls
TBT Tributyl Tin
UV Ultraviolet (light)
VIS Visible (light)

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QUALITY ASSURANCE MANAGEMENT PLAN

MARINE ECOLOGICAL PROCESSES GROUP

VOLUME 3

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Contents

1.0 SAMPLE CONTROL

Sample handling and tracking with the Marine and Environmental Chemistry Group is covered by two procedures: MSL-A-001, *Sample Log-in Procedure* and MSL-A-002, *Sample Chain of Custody*. The processing of data collected from these activities discussed in procedure MSL-D-004*, Data Reporting, Reduction, Back Up, and Archiving*. The following is a description of the procedure used for receipt and tracking of samples, as well as chain of custody procedures.

1.1 SAMPLE RECEIPT AND LOG-IN

Samples or test organisms are logged in when received in the shipping area. If a Chain of Custody (CoC) form accompanies the samples or test organisms, this form is used to document the date and time of sample receipt and condition. If a CoC form is not shipped with samples, an MSL form will be initiated. For test organisms, a shipping form can be signed and dated and the condition of organisms noted. Cooler temperatures are taken and recorded on the CoC. The sample labels are compared to the CoC and assigned an identification code plus sequential numbering of samples upon arrival. Chain of Custody forms (if present) are compared to sample container labels and sample containers are inspected for sample integrity (e.g., broken seals, broken or cracked containers, spilled samples, sample temperature). Any discrepancies are brought to the attention of the Project Manager who is responsible for contacting the client as well as returning a signed copy of the custody form. If the samples are not immediately prepared for testing, they are stored at approximately 4 ^{o}C until used.

See Section 4.1 for holding times for suspended sediment, sediment, effluent, and elutriate samples.

1.2 SAMPLE TRACKING

Sample tracking, while samples are in the laboratory, is the responsibility of the individual Laboratory Supervisors and the Project Manager. It is the responsibility of the Project Manager to ensure that the samples are given the appropriate priority in scheduling and that the proper tests and methods are being performed.

1.3 SAMPLE ARCHIVING AND DISPOSITION

The Project Manager is responsible for proper disposal of leftover samples material. Sample disposition takes three forms: 1) dispose of by appropriate means depending on sample content; 2) return to client; or 3) archive indefinitely. Unless arrangements have been made previously, the samples are generally disposed of by Battelle. If samples are to be disposed of by Battelle, the Project Manager notifies the Health and Safety Officer who then completes a Chemical Disposal Recycle Request form in accordance with Subject Area, *Managing Nonradioactive Chemical Waste*. The Health and Safety Officer then must determine the appropriate disposition and approve the form prior to sample disposition. A copy of this form is maintained in the appropriate project central file.

2.0 BIOLOGICAL PROCEDURES

All routine, repetitive biological laboratory activities are directed and controlled by internal Standard Operating Procedures (SOP). Where possible, U.S. Environmental Protection Agency (EPA) and consensus methods are used where the technique is applicable to the testing matrix and the overall objective of the analysis. Table 2.1 lists the more routine toxicity tests performed at the MSL along with the corresponding methods and SOPs associated with each of those tests. Table 2.2 lists the test organisms and type of tests that are performed at the MSL.

All toxicity tests are controlled by some type of planning document, generally in the form of a Test Plan. Other project planning documents such as work plans are occasionally used. Table 2.3 is an example of a test conditions table that would normally be included in a Test Plan.

TABLE 2.1 List of Biological Tests and Associated Methods and SOPs

Test	Method	SOP
Amphipod	EPA 600/4-90/027F; ASTM 1367-92	MSL-T-005
Echinoderm, Embryo - Larval	EPA 600/R-95/136; ASTM E-1563-95	MSL-T-013
Echinoderm, Fertilization	EPA 600/R-95/136 (Method 1008.0)	MSL-T-011
Echinoderm, Sediment	PSEP 1995	MSL-T-018
Fish, Acute	EPA 600/4-90/027F	NA[2]
Bivalve, Sediment	PSEP 1995	MSL-T-006
Bivalve, Embryo - Larval	EPA 600/R-95/136 (Method 1005.0); ASTM E-724-94	MSL-T-008
Inland Silverside, Acute	EPA 600/4-90/027F	MSL-T-017
Inland Silverside, Chronic	EPA 600/4-87/028; 600/4-91/003	MSL-T-016
Microtox	Microbics Corporation	NA
Microtox, Sediment	PSEP 1995	NA
Mysid, Acute	EPA 600/4-90/027F	MSL-T-014
Mysid, Chronic	EPA 600/4-87/028; 600/4-91/003	MSL-T-015
Polychaetes, Sediment	PSEP 1995	MSL-T-006
Holmesimysis	EPA 600/R-95/136; EPA/503/8-91/001	MSL-T-010
Bioaccumulation	EPA 503/8-91/001	MSL-T-004

[2] NA Not available

TABLE 2.2 Test Organisms Commonly Used by the MSL

			Appropriate Use of Test Organism		
Scientific Name	**Common Name**	**Test Type**	**Aquatic Phase**	**Solid Phase**	**Bioaccu-mulation**
Acropora elseyi	Coral	Acute	✓		
Ammodytes hexapterus	Sandlance	Acute	✓		
Amphiprion clarkii	Clownfish	Acute	✓		
Arbacia punctulata	Sea Urchin	Acute/Chronic	✓		
Capitella capitata	Polychaete	Acute/Chronic		✓	✓
Champia parva	Algae	Acute/Chronic	✓		
Citharichthys stigmaeus	Sanddab	Acute/Chronic	✓	✓	✓
Clupea pallasi (eggs, larvae, adults)	Pacific Herring	Acute/Chronic	✓		
Crassotrea gigas	Oyster	Acute/Chronic	✓		
Cyprinidon vulgaris	Sheepshead Minnow	Acute/Chronic	✓		
Daphnia spp.	Water Flea	Acute/Chronic	✓		
Dendraster excentricus	Sand Dollar	Acute/Chronic	✓		
Dinophilus spp.	Polychaete	Acute/Chronic		✓	✓
Holmesimysis spp.	Mysid	Acute/Chronic	✓		
Isochrisis spp.	Algae	Acute	✓		
Selenastrum spp.	Algae	Acute	✓		
Menidia beryllina	Inland Silverside	Acute/Chronic	✓		
Mysidopsis bahia	Mysid	Acute/Chronic	✓	✓	
Mytilus spp.	Mussel	Acute/Chronic	✓		
Onchorhynchus spp.	Salmon	Acute/Chronic	✓		
Oryzias latipes	Medaka	Acute/Chronic	✓		
Penaeus spp.	Shrimp	Acute	✓		
Photobacterium phosphoreum	Microtox	Acute/Chronic	✓	✓	
Strongylocentrotus purpuratus	Sea Urchin	Acute/Chronic	✓		
Ampelisca abdita	Amphipod	Acute		✓	
Corophium spinicorne	Amphipod	Acute		✓	
Eohaustorius estuarius	Amphipod	Acute		✓	
Grandidierella japonica	Amphipod	Acute		✓	
Hyallella azteca	Amphipod	Acute		✓	
Leptocheirus plumulosus	Amphipod	Acute/Chronic		✓	✓
Neanthes arenoceodentata	Polychaete	Acute/Chronic		✓	✓
Panopea generosa	Clam, Geoduck	Acute		✓	✓
Rhepoxynius abronius	Amphipod	Acute	✓	✓	
Abarenicola pacifica	Polychaete	Acute		✓	✓
Macoma nasuta	Clam, Bent Nose	Chronic			✓
Nephtys caecoides	Polychaete	Acute/ Chronic		✓	✓
Nereis virens	Polychaete	Acute/ Chronic		✓	✓

TABLE 2.3 Example of a Test Conditions Table Established for a Test Plan

Parameter	Conditions		
	M. beryllina	*H. costata*	*M. galloprovincialis*
Test Type	Water-column, static	Water-column, static	Water-column, static
Water Quality	Temperature, pH, salinity, and DO will be monitored on all replicates on Day 0 and Termination Day and in one replicate on remaining days		
Temperature	20 °C ± 2 °C	20 °C ± 2 °C	15 °C ± 2 °C
Salinity	30‰ ± 2‰	30‰ ± 2‰	30‰ ± 2‰
pH	7.30 - 8.30 pH units	7.30 - 8.30 pH units	7.30 - 8.30 pH units
Dissolved Oxygen (DO)	>40% saturation; aeration provided to all chambers only if DO is <40%		>60% saturation
Photoperiod	16L:8D	16L:8D	16L:8D
Test Chamber	500 mL glass jar	400 mL glass jar	500 mL glass jar
Test Solution Volume	300 mL	200 mL	300 mL
Life Stage of Organisms	≤ 5 days	5 days	4 hours
# of Organisms per Chamber	10	10	4500 to 9000 embryos per chamber
# of Replicate Chambers per Treatment	5	5	5
Feeding	Concentrated *Artemia nauplii*, fed daily	Concentrated *Artemia nauplii*, fed daily	None
Reference Toxicant Concentration Series	Cu at 0, 150, 200, 300, 400 µg/L NH_3 at 0, 10, 30, 60, 90 mg/L	Cu at 0, 50, 100, 150, 200 µg/L NH_3 at 0, 20, 40, 60, 80 mg/L	Cu at 0, 1, 4, 16, 64 µg/L NH_3 at 0, 1, 4, 8, 16, 32 mg/L
Dilution Water	0.45 µm-filtered Sequim Bay seawater	0.45 µm-filtered Sequim Bay seawater	0.45 µm-filtered Sequim Bay seawater
SPP Prep Water	dredging site water	dredging site water	dredging site water
Dilution Series and Concentrations[a]	0%, 10%, 50%, and 100% SPP	0%, 10%, 50%, and 100% SPP	0%, 10%, 50%, and 100% SPP
Test Duration	96 h	96 h	48 to 72 h
Endpoint	Survival (LC_{50})	Survival (LC_{50})	Survival (LC_{50}) and normal development (EC_{50})

[a] A 1% SPP dilution may be added if low-level affects are anticipated.

Test Acceptability	90% Survival in control	90% Survival in control	90% Survival and 70% normal development in the controls

3.0 EQUIPMENT MAINTENANCE AND CALIBRATION

Instruments used for routine measurements of chemical and physical parameters, such as pH, dissolved oxygen (DO), temperature, salinity, and ammonia, are calibrated and standardized prior to use. All calibration and preventative maintenance data are documented.

3.1 EQUIPMENT CALIBRATION

Calibration procedures are performed on each water quality instrument prior to use. Requirements for levels and frequency of calibration are described in procedures specific to each water quality instrument:

- MSL-W-001 *Calibration and Use of pH Meters*
- MSL-W-002 *Calibration and Use of Dissolved Oxygen Meters*
- MSL-W-003 *Calibration and Use of Thermometers*
- MSL-W-004 *Calibration and Use of Refractometers*
- MSL-W-007 *Routine Water Quality Measures for Toxicity Tests*, and
- MSL-W-008 *Determination of Ammonia*

All calibration records are kept in the data files and must be traceable to date and standards. Corrective actions to be taken when calibration criteria are not met, are described in section 5.0 of this document and in the specific procedures.

3.2 PREVENTATIVE MAINTENANCE

Instruments are serviced regularly by trained in-house personnel. Written records of all instrument maintenance, calibration, testing, and inspection are maintained. Maintenance records contain a description of the operation or problem, the remedial action taken (if necessary), date, the person responsible, and where applicable, documentation of the instrument's return to acceptable use.

4.0 QUALITY CONTROL

Quality control in toxicity tests consists of establishment of criteria for water quality, test acceptability, reference toxicant tests, replication, control treatments, etc. Each toxicity test has its own quality control criteria that are included as part of the test design established in project planning documents. See Table 2.3 for an example of an established test conditions table.

4.1 HOLDING TIMES AND PRESERVATION

Holding times for toxicity tests typically begin the day of sample collection. Holding times and preservation requirements are listed in Table 4.1.

TABLE 4.1 Sample Holding Times and Preservation

Matrix	Preservation	Holding Time
Sediment	4 ^{o}C ± 2 ^{o}C/dark/airtight	2 weeks is recommended; up to 6 weeks is acceptable
Effluent	4 ^{o}C ± 2 ^{o}C/dark/airtight	36 hours from sample collection (1)
SPP/Elutriate	4 ^{o}C ± 2 ^{o}C/dark/airtight	24 hours from preparation

(1) Every effort must be made to initiate the test with an effluent sample on the day of arrival in the laboratory. The holding time should not exceed 36 hours unless a variance is approved by the client.

4.2 WATER QUALITY MONITORING

Acceptable criteria for water quality (pH, DO, salinity, temperature, ammonia) measurements are established for each test and are identified in project planning documents, such as a test conditions table.

4.3 REFERENCE TOXICANT TEST

Reference toxicant tests (positive controls), are performed to demonstrate that test organisms used are appropriately sensitive and that the laboratory procedures and techniques are appropriate and repeatable. A reference toxicant test is normally performed with each test, or at a minimum, once with each batch of test organisms.

4.4 ACCEPTABILITY OF TOXICITY TESTS

Each test method contains specific test acceptability criteria for controls, reference toxicant results, test conditions, etc. See Section 5.2 for corrective action when criteria are not met.

An individual test may be conditionally acceptable if temperature, DO, or other specified conditions fall outside specifications, depending on the degree of the departure from the specified conditions and the overall impact on the test. The acceptability of the test will depend on the professional judgment of the laboratory supervisor and project manager. Any deviation from test specifications must be noted when reporting data.

4.5 CONTROL CHARTS

Control charts are used to asses QC efforts in the laboratory by graphically presenting the variability over time of the various analyses performed. Details of the control charting process used at the MSL are covered in procedure MSL-Q-006, *Procedures for Control Charting*. A brief description of the methods used, the criteria used for assessing out of control events, and the administration of the control charts is presented here.

4.5.1 Control Chart Methodology

The control charts are based on normally distributed measurements and short-term variation. Precision is charted over time by calculating an LC_{50} and EC_{50} for the reference toxicity tests and then establishing upper and lower warning and control limits. The warning limit is defined as $\pm 2\sigma$ and the control limits are defined as $\pm 3\sigma$. A minimum of 10 points, but preferably 20, are used to set the initial control limits for each parameter.

Reference toxicity tests are run concurrently with the majority of toxicity tests. Separate control charts are maintained for each reference toxicant for each species.

4.5.2 Criteria for Assessing Out of Control Events

The laboratory process for a particular analyte will be considered out of control whenever, as a minimum, any one of the following conditions is demonstrated:

1. Any one point is outside of the control limits;
2. Any three consecutive points are outside of the warning limits;
3. Any eight consecutive points are on the same side of the centerline;
4. Any six consecutive points are such that each point is higher or lower than its immediate predecessor;
5. Any obvious cyclic pattern is seen in the points.

When any one of the situations listed above occurs, it is the responsibility of the appropriate laboratory supervisor to notify the MSL QA Officer and Project Manager so that appropriate corrective actions can be determined and the situation documented by filling out a Quality Problem Report form and attaching a copy to the control chart. Details regarding the procedure and information required on a Quality Problem Report form are described in procedure MSL-A-005, *Deviations from Established Requirements.*

4.5.3 Administration of Control Charts

A minimum of one reference toxicant test is run with each toxicity test or at a minimum, once with each batch of test organisms. Therefore, control results will be tracked after no more than 20 sequential sample analyses. It is the responsibility of Project Managers to provide data after each test to the control chart administrator. Control charts are produced on at least a quarterly basis by the control chart administrator, and this information is passed on to the Laboratory Supervisor and Project Managers, as appropriate. If specific projects require it, more frequent updates and reviews of control charting will be performed.

5.0 CORRECTIVE ACTION

The need for corrective action may be identified by the technical staff during the course of their work, and through assessments or data audits. Each individual performing laboratory or data processing activities will be responsible for notifying the appropriate supervisory personnel of any circumstance that could affect the quality or integrity of the data.

5.1 DEVIATIONS

All deviations from approved procedures, project planning documents or this QAMP will be documented. Depending on the severity of the deviation, the MSL QA Officer and the Project Manager will determine how the deviation will be documented through

- use of a Quality Problem Report (QPR) form (Exhibit 12.1 of Volume 1) per MSL-A-005, *Deviations from Established Requirements*;
- documented as part of the narrative summary provided to the customer, and
- documented directly on the raw data.

The following are guidelines for resolving deviations identified within the Marine Ecological Processes Group:

- The response to technical problems in the field, such as broken equipment, weather delays, or inability to sample specific locations, is the responsibility of Field Task Leader. This individual determines the appropriate action, in conjunction with the Project Manager and/or client representative.
- The need for corrective action at the laboratory level, for events such as broken samples or improper instrument calibration, will be addressed by the Laboratory Supervisor or Project Manager.
- Corrective actions for results outside established DQOs are addressed in section 5.2.

5.2 CORRECTIVE ACTION FOR DQO EXCEEDENCES

DQO deviations are defined as deviations that are outside of test specific criteria addressed in Section 2. Out-of-compliance data may be due to deviations from test protocols or deficiencies associated with toxicological tests. Examples of DQO deviations in toxicological tests are shown in Table 5.1. Poor control survival, out-of-range water quality measurements, out-of-range reference toxicant results, or mishandling of test organisms or test sediment/water may result in a decision to retest; minor episodes of out-of-range water quality conditions, incomplete test monitoring information, or broken or misplaced test containers may only require that data be flagged and qualified. A summary of typical test deviations and suggested corrective actions is presented in Table 5.1.

Corrective actions relative to toxicological tests may include, but are not limited to, review of data and calculations, flagging and/or qualification of suspect data, or possible retesting. A review that provides a preliminary check of all “out of limit” events should be performed as soon as the data for a given parameter or test is tabulated and verified for accuracy.

TABLE 5.1 Summary of Test Deviations and Suggested Responses

	Suggested Responses	
Deviation	Retesting Required	Retesting May Be Required (1)
Lack of test array randomization	✓	
Testing was not blind	✓	
Required references or controls were not tested	✓	
Test chambers not identical	✓	
Test container(s) broken or misplaced		✓
Test organism mortality in controls exceeds acceptable limits	✓	
Excessive test organism mortality in a single replicate of a control	✓	
Test organisms were not randomly assigned to test chambers	✓	
Test organisms were not from the same population		✓
Test organisms were not all from the same species (or species complex)	✓	
Test organism holding times were exceeded		✓
Test organism sensitivity out of acceptable control chart range		✓
Water quality parameters consistently out of range	✓	
Brief episodes of out-of-range water quality problems		✓
Test monitoring was not documented	✓	
Test monitoring was incomplete		✓
Sediment/testing water holding times were exceeded	✓ (2)	
Sediment/testing water storage conditions deviated from acceptable ranges		✓(2)

(1) If retest not completed, data may have to be qualified.

(2) Unless evidence is provided to show that sediment quality (geochemistry and contaminant levels) has not been affected

6.0 DATA REPORTING

All reported data will be validated in accordance with Volume 1, Section 10 of this QAMP. Reduced or summarized data from the toxicity tests will be reported to the client. The following is a list of data that is typically reported.

- description of test sediment or water; it's handling, manipulation, storage, and disposal
- description of test organisms; scientific name, age, size (when applicable), life stage, source, and their handling, culturing, and acclimation
    - toxicity test method used
- date and time test started and terminated
- percent survival for each test treatment
- control treatment survival
- results of water quality measurements (may be reported as mean, range of measurements, number of times criteria limits were exceeded)
- number of organisms used per test chamber
- number of replicate test chambers per treatment
- summary of statistical endpoints (mortality, growth, LC_{50}, no observed effect concentration [NOEC],)
- gender determinations (when appropriate)
- growth (when appropriate)
- reproduction (when appropriate)
- summaries of biological observations
- summaries of reference toxicant evaluations
- summary of any problems encountered and corrective actions
- description of any deviations from prescribed laboratory protocols

7.0 REFERENCES

ASTM 1367-92	1992 – Standard Guide for Conducting 10-Day Static Sediment Toxicity Tests with Marine and Estuarine Amphipods. American Society for Testing and Materials, Philadelphia, Pennsylvania.
ASTM E-724-94	1994 – Conducting Static Acute Toxicity Tests Starting with Embryos of Four Species of Saltwater Bivalve Mollusks. American Society for Testing and Materials, Philadelphia, Pennsylvania.
ASTM E-1563-95	1995 - Conducting Static Acute Toxicity Tests with Echinoid Embryos. American Society for Testing and Materials, Philadelphia, Pennsylvania.
EPA 503/8-91/001	Evaluation for Dredged Material Proposed for Ocean Disposal - Testing Manual. U.S. Environmental Protection Agency (EPA) and U.S. Army Corps of Engineers. U.S. EPA, Office of Water, Washington D.C.
EPA 600/4-87/028	Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms. May 1988. U.S. EPA. Office of Research and Development, Cincinnati, Ohio.
EPA 600/4-90/027F	Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms. Fourth Edition. September 1991. U.S. EPA. Office of Research and Development, Washington, D.C.
EPA 600/4-91/003	Short-Tem Methods for Estimating The Chronic Toxicity of Effluents and Receiving Water to Marine and Estuarine Organisms. Second Edition. July 1994. U.S. EPA. Office of Research and Development, Washington, D.C.
EPA 600/R-95/136	Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to West Coast Marine and Estuarine Organisms. August 1995. U.S. EPA. Office of Research and Development, Washington DC.
PSEP 1995	Puget Sound Estuary Program (PSEP). 1995. Recommended Guidelines for Conducting Laboratory Bioassays on Puget Sound Sediment. Prepared for the U.S. EPA, Region 10, Seattle, WA..

APPENDIX A

MARINE ECOLOGICAL PROCESSES EQUIPMENT LIST

The following is a list of the major pieces of equipment in the MSL bioassay wet laboratory. This list is intended to demonstrate the types of equipment available and will be revised when the QAMP is revised, but not each time equipment or instruments are added or deleted.

Water Quality Measures		
Ammonia	Orion 900A ph Meter	SN 039388
	Orion 900A ph Meter	SN 039548
	Various ammonia probes	
Combined Measures		
DO, pH, T, salinity	YSI Meter Environmental Monitoring System 610-DM/ Data Logger	312188R
	Sonde 600 probe	SN 99110867
Individual Measures		
T	NIST-traceable thermometer	A-09618
	Fluke 52 K/J thermometer	4986664
	Fluke 52 K/J thermometer	4655092
	Fluke 52 K/J thermometer	480056
	Fluke 52 K/J thermometer	5000160
	Fluke 52 K/J thermometer	5425282
	Fluke 52 K/J thermometer	5305158
	Fluke 52 K/J thermometer	5025363
Salinitiy	Reichert Refractometer	10212-8
		10392-8
DO Meter	YSI Model 57	37222
	YSI Model 57	15679 (TBT)
pH Meter	Orion SA250	7335
	Orion SA250	7428
	Orion SA250	6478
Other Equipment		
Light Intensity	Licor 185-A	
	Extech Instruments Light Meter 407026	E002938
Microscopes	Leica Wild M3Z	Z-050-262
	Wild Heerbrugg 28003	28003
	Nikon Labophot	100476-89
Centrifuge	Bock Extractor	9183-P

Weight/Balances		
AND FX3000	5213125	0-3000 g
AND FX3200	5314791	0-3000 g
AND ER120A	3502726	0-100 g
Mettler PE3600	D07416	0-3000 g
Mettler P2010	633034	0-2000 g
Mettler AE163	QC03126	0-150 g
Ohaus DS4	60919	0-20 g
Ohaus DS4	77927	0-20 g

Attachment 6

SERP Comprehensive QA Plan (Analytical and Mercury Laboratories)

COMPREHENSIVE QUALITY ASSURANCE PLAN

Prepared by and for:

Southeast Environmental Research Program
Florida International University
Miami, Florida

COMPREHENSIVE QUALITY ASSURANCE PLAN

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_____ _____

Ronald D. Jones, Ph.D. Date
SERP Director and Professor

_____ _____

Doraida Diaz Date
SERP Quality Assurance Officer

_____ _____

Laboratory Certification Program Date
Department of Health

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APPENDICES

3.0 Statement of Policy

The Southeast Environmental Research Program (SERP) is made up of university research professors and their staff from Florida International University (FIU). FIU is one of the nine State University System (SUS) universities and all SERP personnel are employees of the State of Florida. The goals of SERP are to advance scientific research, the understanding of biogeochemical processes, and to publish results in high quality refereed scientific publications. Pertinent to these goals, is the need to collect accurate, high quality, and reproducible data, which can only be obtained through strict internal and external quality assurance practices. SERP is committed to follow sound quality assurance/quality control (QA/QC) practices for the purposes of producing verifiable quality data.

The professors associated with SERP have been involved in monitoring surface water quality in Florida Bay, Biscayne Bay, the Everglades, other areas of South Florida, and the world's oceans for over 15 years. The data collected by SERP to date is considered to be of excellent quality, and has been used by FDEP, South Florida Water Management District (SFWMD), the National Park Service, Department of Interior, Department of Justice, and the EPA.

Research conducted by SERP is mainly focused on water quality nutrients (nitrogen and phosphorus), which are important influences to South Florida's ecosystem. In support of interpreting the nutrient data, SERP also measures other water quality and physiochemical parameters such as salinity, temperature, turbidity and chlorophyll. Nutrients commonly measured by SERP typically occur in surface waters of South Florida at relatively low concentrations (parts per billion). Often the nutrients occur at concentrations below typical contract laboratory method detection limits; however, small changes in these surface water nutrients can have a significant effect on the ecology of South Florida. As a university research facility, SERP is committed to obtaining the most accurate measurements as well as obtaining the lowest possible method detection limits for these nutrients. To obtain low level detection and calibration, SERP has had to modify and optimize analytical methods and equipment for detection of nutrients in freshwater, brackish waters, and seawater. SERP has also had to modify equipment decontamination procedures to ensure contamination-free sampling for low concentrations of nutrients. Many of the analytical and sampling methods employed by SERP have been included in scientific publications.

This Comprehensive Quality Assurance Plan (CompQAP) describes the sampling and analytical methods used by SERP personnel to ensure the integrity and accuracy of field and laboratory data collection and analysis. The CompQAP has been prepared in accordance with the Florida Department of Environmental Protection (FDEP) guidelines. Project-specific objectives and sampling protocols will be described in more detail in Quality Assurance Project Plans (QAPPs).

4.0 Organization and Responsibility

4.1 Capabilities

The research group at SERP conducts both field sampling and laboratory analysis. SERP performs field sampling of surface water, pore water (water in soils and sediments), soils, sediments, and plant tissue. Analyses performed in the laboratory include inorganic nutrients, organic nutrients, and physical parameters of surface waters, ground waters, pore waters, soils, sediments, and plant tissue. SERP is fully capable of analyzing nutrients in fresh water, brackish water and sea water.

4.2 Key Personnel

Dr. Ronald D. Jones is the director of the Southeast Environmental Research Program (SERP) at Florida International University (Figure 4-1). As director, Dr. Jones supervises all laboratory and field operations and personnel. He provides a final review of all data and documents produced.

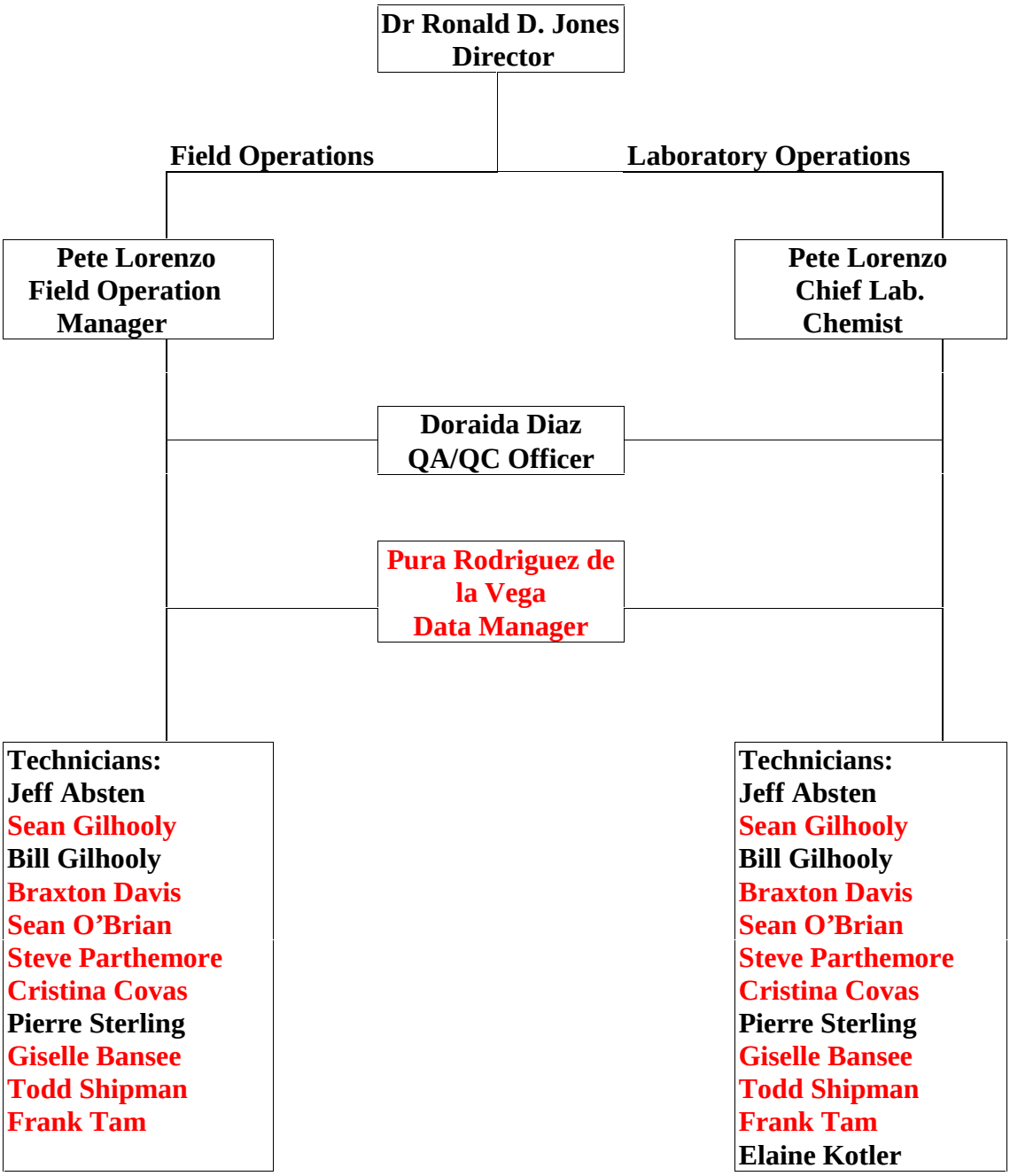
Mr. Pete Lorenzo is the chief laboratory chemist and the field operation manager. In this role, he is responsible for the proper execution of the daily field and laboratory operations. He provides scheduling of field and laboratory personnel, and is responsible for the collection, custody, storage, and analysis of all samples.

Ms. Pura Rodriguez de la Vega is the SERP Data manager. She is responsible for checking all the data produced in the lab according with QC criteria and for the preparation of the final data reports.

Ms. Doraida Diaz is the SERP QA officer. She is responsible for preparing all QAPs, and overseeing that the field and laboratory operations are performed according to the QAPs. She is also responsible for a final check of all data produced with respect to QC criteria, initiating and conducting audits, and preparing QA reports.

Sample collection, analysis, and data entry is performed by technicians and graduate students at SERP under the direction of Dr. Jones and Mr. Lorenzo. They are trained in the proper procedures for sample collection, preservation, transportation, and analysis.

Figure 4.1 SERP Organization Chart



5.0 Quality Assurance Objectives (Precision, Accuracy, and Method Detection Limits)

All sampling and analytical work is performed to obtain accurate, reproducible data using consistent standard curves and extremely low method detection limits. The SERP laboratory is equipped with state-of-the art analytical equipment. All students and staff are trained on proper use of the equipment and supervised during all phases of sample collection and analysis by Dr. Jones. In general, the people responsible for sample collection are also performing the laboratory analysis of the samples, thereby, maintaining control of all aspects of sample collection and analysis.

Parameters routinely measured in the field are listed on Table 5.1 and include temperature, salinity/conductivity, dissolved oxygen, pH, and turbidity. Matrices analyzed include surface waters, pore waters, ground waters, soils, sediments and plant tissue. Laboratory precision, accuracy, and method detection limits (MDLs) for specific parameters in each matrix are summarized in Table 5.2. The listed precision, accuracy, and MDLs are determined using in-house, historically generated data.

Analytical procedures performed by SERP are listed in Table 5.2. In general, SERP follows analytical procedures described in Methods for Chemical Analyses of Water and Wastes, EPA-600/4-79-020, Revised March 1983 and in Standard Methods for the Examination of Water and Wastewater, 18th Edition, 1989. For solid samples SERP follows the methods described in the Annual Book of ASTM Standards, Volume 4.08, Procedures for Handling and Chemical Analysis of Sediments and Water Samples, May 1981, EPA/CE-81-1 and Methods of Soil Analysis, Part 2-Chemical and Microbiological Properties, Second Edition, American Society of Agronomy, Inc. Soil Science Society of America, Inc., 1982.

TABLE 5.1
Quality Assurance Objectives
Field Measurements

Method No.	Matrix	Parameter
EPA 170.1	Surface Water, Pore Water	Temperature
SM 2520 (B)	Surface Water, Pore Water	Salinity
EPA 120.1	Surface Water, Pore Water	Conductivity
EPA 360.1	Surface Water, Pore Water	Dissolved Oxygen
EPA 150.1	Surface Water, Pore Water	pH
EPA 180.1	Surface Water, Pore Water	Turbidity
Photosynthetically Active Radiation (a)	Surface Water	Light Attenuation Coefficient
Pressure Transducer, Depth Sounder (b)	Surface Water	Depth

EPA = U.S. Environmental Protection Agency. Methods for Chemical Analysis Water and Wastes, Revised March 1983.
SM = Standard Methods for Examination of Water and Wastewater, 1989, 18th Edition.
ASTM= Annual Book of ASTM Standards, Vol 11.01.

EPA/Corps of Engineers, Procedures for Handling and Chemical Analysis of Sediments and Water Samples. May 1981. EPA/CE-81-1 Page 3-52.

(a) See Section 6 for method details.

(b) Pressure transducer method is used on the SEA-BIRD CTD; depth sounder method is used when the CTD is not used.

TABLE 5.2
Sample Preparation Methods

Sample Prep. Method Number	Description	Matrix	Sample Prep. for these Methods
ASTM D 4638(7.4)	Evaporation	Water	EPA 365.1
ASTM D 4638(9.2)	Dry Ashing	Water, Soil, Sediment, Tissue	EPA 365.1

Method References:

EPA Methods for Chemical Analysis of Water and Wastes, Revised March 1983.

Annual Book of ASTM Standards, Vol 11.01.

Solórzano L. and J.H. Sharp. 1980. Determination of Total dissolved Phosphorus and Particulate Phosphorus in Natural Waters. Limnol. Oceanogr. 25(4), pp. 754-758. See Appendix B.

TABLE 5.3
Quality Assurance Objectives
Laboratory Measurements

Analyte	Matrix	Analytical Method	Precision (RPD) (a)	Conc. Range (b)	Accuracy (%R) (a)	MDL (c) (µmol/l, unless noted)	MDL (c) (mg/l, unless noted)
Ammonium-N	SW, GW, PW	EPA 350.1 (d)	<20 %	L, M, H	78-128	0.06 0.05	0.0008 0.0007
Nitrite - N	SW, GW, PW	EPA 353.2 (d)	<20 %	L, M, H	84-107	0.02 0.02	0.0003 0.0003
Nitrate - N	SW, GW, PW	EPA 353.2 (d)	<20 %	L, M, H	84-107	0.05 0.05	0.0007 0.0007
Soluble Reactive Phosphate	SW, GW, PW	EPA 365.1 (d)	<20 %	L, M, H	78-110	0.02 0.04	0.0006 0.0012
Dissolved Silica	SW, GW, PW	EPA 370.1 (d)	<20 %	L, M, H	85-123	0.07 (f)	0.002 (f)
Total Nitrogen	SW, GW, PW	(g)	<20 %	L, M, H	75-108	2.1	0.03
Total Phosphorus	SW, GW, PW	EPA 365.1 (e)	<20 %	L, M, H	79-125	0.01 0.01	0.0003 0.0003
Total Organic Carbon, Dissolved Organic Carbon	SW, GW, PW	EPA 415.1 (h)	<20 %	L, M, H	85-118	10.00	0.12
Chlorophyll a	SW	SM 10200H (i)	<20 %	L, M, H	80-120	NA	0.0001
Alkaline Phosphatase Activity	SW	(j)	<20 %	L, M, H	80-120	0.01 µmol/l.hr $^{-1}$	NA
Bulk Density	S, SED	ASTM D4531-86	<20 %	L, M, H	80-120	NA	0.001 g/cc (k)
Total Nitrogen Total Carbon	S, SED, T	MSA 29-2.2.5 (l) EPA/CE-81-1 SID, S3, 2	<20 %	L, M, H	80-120	NA	10 mg/kg (l)
Moisture Cont./ % Solids	S, SED	ASTM D2216-80	<20 %	L, M, H	80-120	NA	3 % (k)
Ash Free Dry Weight	S, SED	ASTM D2974-87	<20 %	L, M, H	80-120	1.7 µmol/kg (k)	0.02 mg/kg (k)
Total Phosphorus (m)	S, SED, T	EPA 365.1 (e)	<20 %	L, M, H	96-118	0.97 µmol/kg (k)	0.03 mg/kg (k)

TABLE 5.3 Continued
Quality Assurance Objectives
Laboratory Measurements

(a) QA targets for precision and accuracy determined from in-house, historical data.

(b) Concentration Range: L = lower 20% of linear calibration or range.
M = from 20% to 80% of linear calibration range.
H = The upper 80% of linear calibration range.

(c) Method Detection Limits (MDLs) determined by EPA procedure described in 40 CFR Part 136, Appendix B, revision 1.11.

(d) Ammonium, nitrite, nitrate, soluble reactive phosphate, and silica of water samples are determined on an ALPKEM 305 Rapid Flow Analyzer and ALPKEM 501 autosampler.

(e) Total phosphorus of water and solid samples is determined on an ALPKEM 305 Rapid Flow Analyzer and ALPKEM 501 autosampler using the automated method of EPA 365.1, with the samples prepared according to a modification of Solorzano and Sharp (1980; see Section 8.1.4 and Appendix B), instead of persulfate digestion.

(f) Theoretical MDL for the method. The actual MDL for silica for our laboratory has not yet been determined.

(g) Total Nitrogen of water samples is determined using an ANTEK Instruments Model 7000N Total Nitrogen Analyzer. Method validation package included as Appendix A.

(h) Total Organic Carbon of water samples is determined by high temperature catalytic combustion with a Shimadzu 5000 Total Organic Carbon Analyzer with autosampler.

(i) Chlorophyll a is determined using a modification of SM 10200H as outlined in Section 8.1.6.

(j) The analytical method for Alkaline Phosphatase Activity is currently under experimental research. This method is described in more detail in Section 8.

(k) Values represent minimum reportable quantities.

(l) Total nitrogen and total carbon of solid samples determined using a Carlo Erba Model 1500 N/C analyzer. Minimum reportable quantity of 0.01 % based upon a sample size between 0.5-100 mg.

(m) The results of quality control check samples are summarized in Appendix B.

SW-Surface Water; PW-Pore Water; GW-Ground Water; S-Soils; SED-Sediments; T-Tissue

Method References:

EPA Methods for Chemical Analysis of Water and Wastes, Revised March 1983.
EPA/Corps of Engineers, Procedures for Handling and Chemical Analysis of Sediments and Water Samples. May 1981. EPA/CE-81-1.
Standard Methods for the examination of Water and Wastewater, 17th edition. 1989.
Annual Book of ASTM Standards, Method D4531-86, Volume 04.08, 1989.
Methods of Soil Analysis, Part 2-Chemical and Microbiological Properties, Second Edition, 1982.

6.0 Sampling Procedures

6.1 Sampling Capabilities

SERP performs sampling of surface water, pore water, soil and sediments, and plant tissue for determination of the field and laboratory analytical parameters listed in Table 6.1.

6.2 Sampling Equipment and Cleaning Procedures

6.2.1 Sampling Equipment

Preceding a trip to the field, the personnel responsible for collection of the samples are required to ensure that everything is prepared for the expedition. This entails making sure that all sample containers are clean and properly labeled, and that all sampling and field measurement equipment are properly cleaned, charged and functioning within acceptable limits. Table 6.2 lists the field sampling equipment used for sampling each matrix, while Table 6.3 is an equipment checklist prepared for the sampling team.

In general, sampling equipment used is dictated by a specific project. Surface water sampling equipment may include plastic sample containers, syringes, filter holders, and buckets. Pore water samples are collected from lysimeters using a peristaltic pump or a syringe equipped with tygon tubing. Soil and sediment samples are collected using plastic core tubes or by hand. Tissue samples are generally collected by hand and stored in plastic, sealable bags.

6.2.2 Sampling Equipment Laboratory Cleaning Procedures

All reusable field sampling and measurement equipment is subjected to precleaning in the laboratory prior to transportation to a field site according to the following procedures:

a. Wash all surfaces thoroughly with hot, tap water. Use a brush to remove large or stubborn particles.
b. Rinse thoroughly with analyte free water (deionized water).
c. Let air dry completely, or dry with Kimwipes.
d. Wrap equipment in plastic bags for storage and transportation.

Note, the above cleaning procedure does not include the use of soaps or acids as recommended by DEP. The concentrations of the nutrients of interest (phosphate, ammonium and nitrate) can be significantly affected by the use of these cleaning solutions. The cleaning procedures used by SERP in the last ten years have produced non-detectable concentrations of the analytes listed in Table 6.1 in equipment blanks, and historical data supporting this is available upon request.

TABLE 6.1
SERP Sampling Capabilities

Parameter Group	Sample Source
Inorganic Anions and Nutrients Ammonium Nitrite Nitrate Phosphate Silica	Surface Water, Pore Water
Total Nitrogen	Surface Water, Pore Water, Soils, Sediments, Tissue
Total Phosphorus	Surface Water, Pore Water, Soils, Sediments, Tissue
Organics	
Total Carbon	Surface Water, Pore Water, Soils, Sediments, Tissue
Dissolved Organic Carbon	Surface Water, Pore Water
Chlorophyll-a	Surface Water
Alkaline Phosphatase Activity	Surface Water, Pore Water, Soils, Sediments, Tissue
Field Parameters Temperature pH Salinity/Conductivity Dissolved Oxygen Turbidity Light Attenuation Coefficient	Surface Water, Pore Water Surface Water
Other Bulk Density Percent Mineral Content Ash Free Dry Weight	Soils, Sediments

TABLE 6.2
Field Sampling Equipment

Equipment	Construction	Use	Parameter Groups	Restrictions, Precautions, Notes.
Surface Water				
Sampling Equipment				
Syringes	140 ml plastic (HDPP)	Collection	Soluble nutrients, Suspended matter Chlorophyll	None
Bottles	60 ml plastic (HDPE)	Collection	Soluble nutrients	Filtered samples only, 2 per location
Bottles	125 ml plastic (HDPE)	Collection	Total nutrients, organics	2 per location
Microcentrifuge Tubes	1.8 ml plastic (HDPE)	Filter storage	Suspended matter, Chlorophyll	2 per location, preserve with acetone
Bucket	2-5 gallon plastic (LDPE)	Collection	All surface parameters	None
Niskin Sampler	2.5 L PVC	Collection	All surface parameters	None
Field Filtration				
In-line Filter Holders	2.5 cm plastic (Gelman)	Filtration	Soluble nutrients, Suspended matter, Chlorophyll	Attach to end of syringe
Filters	2.5 cm Whatman GF/F glass fiber	Filtration	Soluble nutrients, Suspended matter, Chlorophyll	Use filter forceps to place into and remove from filter holder

TABLE 6.2 Continued.
Field Sampling Equipment

Equipment	Construction	Use	Parameter Groups	Restrictions, Precautions, Notes.
Pore Water Sampling Equipment				
Peristaltic Pump	Tygon Tubing	Purging, Sampling	All parameters	Use new tubing prior to each sampling event.
In-line Filter	Plastic, 2.5 cm Whatman GF/F glass filter	Filtration	Soluble nutrients	None
Syringe	120 ml plastic (HDPP) with Tygon Tubing	Purging, Sampling	All parameters	Use new tubing prior to each sampling event.
Soils, Sediments				
Spade, Spatula	Stainless Steel	Sample collection, cutting	All parameters	None
Core Tubes	Polycarbonate or PVC	Sample collection	All parameters	None
Wildco Eggshell Core	Stainless Steel	Corer	All parameters	None
Eckman Dredge	Stainless Steel	Penetration	All parameters	None
Plant Tissue				
Bags	Plastic	Sample collection	All parameters	None

TABLE 6.2 Continued.
Field Sampling Equipment

Equipment	Construction	Use	Parameter Groups	Restrictions, Precautions, Notes.
Miscellaneous Equipment				
Acetone	90%, ACS grade	Preservation	Chlorophyll	Store in a 250 ml HDPE bottle in a ziploc bag
Disposable pipettes	Plastic (polyethylene)	Dispensing	Chlorophyll	None
Ice (wet)		Preservation	Soluble nutrients Chlorophyll Tissue samples	Need sufficient quantity to ensure even temperature distribution
Coolers	Plastic	Transportation	All parameters	None
DI water	1 L plastic (LDPE) 250 ml plastic (LDPE) squeeze bottle	Equipment blanks Rinsing	All parameters	None
Field notebook	Field data sheets Instrument calibration sheets Field equipment checklist	Documentation	All parameters	None
Pens	Waterproof	Documentation	All parameters	None
Labeling tape	Waterproof	Documentation	All parameters	None
Site charts	Waterproof	Reference	All parameters	None

TABLE 6.2 Continued.
Field Sampling Equipment

Equipment	Construction	Use	Parameter Groups	Restrictions, Precautions, Notes.
Miscellaneous Equipment				
Field instruments	DO meter SCT meter pH meter CTD	Field measurements	All parameters	None
pH buffers	7.00 and 10.00 pH solutions	Meter calibration	All parameters	None
S/C check standard	Gulfstream seawater	Calibration check	All parameters	None

TABLE 6.3
Field Equipment Checklist

Surface Water Sampling Equipment

1. Labeled and cleaned sample bottles (narrow-mouth plastic)
   60 ml (2 per site)
   125 ml (2 per site)
2. 140 ml clean plastic syringes
3. Microcentrifuge tubes
4. 2.5 cm in-line filter holders
5. 2.5 cm Whatman GF/F glass fiber filters
6. Filter forceps
7. 2-5 gallon plastic bucket
8. Niskin sampler

Pore Water Sampling Equipment

1. Peristaltic pump or 120 ml plastic syringes
2. Tygon tubing
3. Water level indicator

Field Measurement Equipment

1. pH meter
2. S/C/T meter
3. Dissolved oxygen meter
4. CTD
5. Light meter
6. pH Buffers (7.00 and 10.00)
7. Salinity/Conductivity check standard
8. Plastic beaker

Sample Preservation

1. 100% Acetone
2. Disposable polyethylene pipettes
3. Ice
4. Coolers
5. DI water (1L) for equipment blanks

Soil, Sediment & Sampling Equipment

1. Labeled sampling bottles
2. Spatula
3. Spade
4. Measuring rule
5. Core tubes

TABLE 6.3 Continued
Field Equipment Checklist

Tissue Sampling Equipment

1. Labeled sample bags

Boat Supplies

1. Depth finder
2. GPS (Magellan 5000 D)
3. VHF Radio
4. PFD's (adequate for number for passengers)
5. Boat hook
6. Emergency flares
7. Charts
8. Tool box
9. Fire extinguisher

Miscellaneous Equipment

1. Clipboard with waterproof field data and calibration sheets
2. Pencils
3. Waterproof label tape and waterproof pens
4. Deionized water squeeze bottle (filled)
5. Watch
6. CompQAP (available in the field for reference)

Two types of analyte-free water are produced in the laboratory: deionized-distilled water and double-deionized water. In general, the deionized-distilled water is used for washing equipment and glassware, while the double-deionized water is used as reagent water. Tap water is first deionized using a Culligan system containing activated carbon and 2-mixed bed ion exchange beds followed by filtering through a 0.45 μm polypropylene filter cartridge. The water is then either distilled through a Corning Mega-Pure 11 Liter Automatic Water Still to produce the deionized-distilled water or further deionized with a Barnsted model D8911 HN Ultrapure mixed bed deionization cartridge to produce the double-deionized water. Both types of water have proven to be analyte-free for the nutrients analyzed. The quality of this water is frequently checked with laboratory method and field equipment blanks. Containers used to store analyte-free water are kept dedicated to this use, therefore, cleaning of these containers is not necessary.

6.2.3 Sampling Equipment Field Cleaning Procedures

In the field, all field equipment used for collection of surface and pore water sampling is triple-rinsed with sample water prior to sample collection or field measurement. New tygon tubing is replaced on the peristaltic pump or syringe at the beginning of each pore water sampling event. Between sampling locations, the tygon tubing is rinsed with analyte-free water. Reusable field equipment used to collect soil and sediment equipment is cleaned between sampling locations by rinsing with analyte-free water. If the sampling equipment is used only once in the field, and not cleaned in the field, the equipment is tagged with the sample location and cleaned according to the laboratory cleaning procedures described in Section 6.2.1. The probes of field instruments are wiped if necessary to remove large particles, rinsed with DIW, and allowed to air dry for as long as possible before using at the next station. The cleaning procedures for all field equipment used during a sampling event are documented in the field notebook and include which equipment was cleaned, the procedure used, and the date and initials of the person performing the procedure.

If samples containing high concentrations are suspected of being collected during an event (such as surface waters downgradient of a landfill), then the sampling program will be performed to collect samples from lowest suspected concentration to highest suspected concentration. Any equipment suspected of contamination from these sampling events, will by thoroughly cleaned; any equipment that can not be cleaned is discarded.

TABLE 6.4
Sample Containers, Sizes, Preservations and Holding Times

Sample Type/Parameter	Container/Size	Preservative	SERP Holding Time	Maximum Allowable Holding Time (a)
Water Samples				
Ammonia	Plastic, 60 ml	Cool, 4^0C	24 hours	28 days (b)
Nitrite	Plastic, 60 ml	Cool, 4^0C	24 hours	28 days (b)
Nitrate	Plastic, 60 ml	Cool, 4^0C	24 hours	28 days (b)
Soluble Reactive Phosphate	Plastic, 60 ml	Cool, 4^0C	24 hours	28 days (b)
Silica	Plastic, 125 ml	Cool, 4^0C	28 days	28 days
Total Nitrogen	Plastic, 125 ml	Cool, 4^0C	28 days	28 days
Total Phosphorus	Plastic, 125 ml	Cool, 4^0C	28 days	28 days
Total Organic Carbon, Dissolved Organic Carbon	Plastic, 125 ml Plastic, 60 ml	Cool, 4^0C	7 days	28 days
Chlorophyll-a	GF/F Glass Fiber filter, 1.8 ml HDPE Microcentrifuge Tube	Acetone, Freeze, -15^0C, Store in the Dark	7 days	21 days
Alkaline Phosphatase Activity	Plastic, 125 ml	Store in the Dark	12 hours	N/A (c)

TABLE 6.4 Continued
Sample Containers, Sizes, Preservations and Holding Times

Sample Type/Parameter	Container Size	Preservative	SERP Holding Time	Maximum Allowable Holding Time (a)
Turbidity	Plastic, 125 ml	Cool, 4^0C	12 Hours	48 hours
pH	None	None	Analyze Immediately	Analyze Immediately
Temperature	None	None	Analyze Immediately	Analyze Immediately
Salinity/ Conductivity	None	None	Analyze Immediately	28 days
Dissolved Oxygen	None	None	Analyze Immediately	Analyze Immediately
Light Attenuation Coefficient	None	None	Analyze Immediately	Analyze Immediately
Soils Sediments All Parameters	Plastic core tubes, plastic wide-mouth specimen cups	Cool, 4^0C, dark Dry, grind, and store in desiccator Freeze, -15^0C, dark	48 hours 48 days 48 days	48 hours Indefinitely Indefinitely
Tissue All Parameters	Plastic Bags	Freeze, -15^0C, dark	1 Year	N/A (c)

(a) According to U.S. EPA, 1983, methods for chemical analyses of Water and Wastes.
(b) If SERP maximum holding times are exceeded for these parameters, then samples are frozen and analyzed within the maximum allowable holding time.
(c) Not Applicable.

6.3 Sample Containers and Cleaning Procedures

6.3.1 Sample Containers

Sample containers, preservation methods, and appropriate holding times are listed in Table 6.4. Three types of sample containers are used for surface water nutrient sampling: 60 ml HDPE screw-cap bottles for filtered water samples; 125 ml HDPE screw-cap bottles for unfiltered water samples; and Whatman GF/F glass fiber filters, stored in 1.8 ml microcentrifuge polypropylene tubes with caps for suspended matter samples. Sample containers used for pore water samples are the same as those used for surface water samples except that chlorophyll-a is not collected. Soil and sediment samples are collected in plastic core tubes or wide-mouth plastic specimen cups. Plant tissue samples are stored in plastic, sealable bags.

6.3.2 Sample Container Cleaning

Similar to the field equipment cleaning protocols, no soaps or acids are used in cleaning of sample containers, since we have found these cleaning solutions have the potential to contaminate the sample containers for the nutrients listed in Table 6.1. All surface water and pore water sample bottles are further cleaned in the field by triple rinsing with sample water. Each surface water sample is collected in duplicate in the field, providing for a quality assurance check of possible container contamination. Any sample container suspected of being contaminated is discarded. The equipment blank results are documented with the corresponding sample set runs so it is possible to track potential bottle contamination. The water sample bottles can be re-used after the proper cleaning procedure during 2 years (after they were used for the first time) for sampling purposes and after this period of time they are discarded independently from how many times they were used.

6.3.2.1 Surface Water and Pore Water Sample Containers for Filtered Nutrient Analyses

HDPE sample bottles used for collection of filtered nutrient analyses are cleaned by the following methods:

a. Remove all labels, and wash all surfaces thoroughly with hot, tap water. Use a brush to remove large or stubborn particles.
b. Rinse thoroughly (at least three times) with analyte-free water.
c. Rinse once with acetone to aid in drying, and to remove organics.
d. Shake dry then cap.
e. Store sample containers in plastic bags for transportation to the field.
f. In the field, triple rinse with sample water prior to sample collection.

6.3.2.2 Surface Water and Pore Water Sample Containers for Unfiltered Nutrient Analyses

Sample bottles used for collection of non-filtered samples are cleaned following the procedures described in Section 6.3.2.1, except they are not rinsed with acetone, and they are allowed to air dry.

6.3.2.3 Surface Water Sample Containers for Suspended Matter (Chlorophyll Analyses)

Both the Whatman GF/F glass fiber filters and the 1.8 ml tubes used for storage of suspended matter sediments are obtained clean directly from the manufacturer, used once, then discarded.

6.3.2.4 Soil and Sediment Sample Containers

The polycarbonate or PVC core tubes and specimen cups used for soil and sediment collection are washed according to the following procedures:

a. Remove all labels, and wash all surfaces thoroughly with hot, tap water. Use a brush to remove large or stubborn particles.
b. Rinse thoroughly (at least three times) with analyte-free water.
c. Allow to air dry.

6.3.2.5 Tissue Sample Containers

The plastic bags used to store tissue samples are used once, then discarded.

6.4 Sampling Protocols

Specific sampling locations are chosen based on criteria described in the appropriate Quality Assurance Project Plans. In general, surface water, sediment, and plant tissue samples are collected from a boat, helicopter, airboat or by a SCUBA diver. To ensure collection of undisturbed samples, the boat is advanced toward a sampling station from the downstream direction. Surface water samples are collected as grab samples from the bow of the boat, away from the outboard engine. Sediment and tissue samples are collected by SCUBA diver or by wading upgradient of the anchor, if one is used, or upgradient of the bow if an anchor is not used. If surface water samples and sediment and/or tissue samples are to be collected at one location, then the surface water samples will be collected prior to the collection of sediment or tissue samples. In areas of suspected high concentrations, such as downgradient of a landfill, samples are collected in order of suspected low concentration to higher concentration.

6.4.1 Surface Water Sample Collection

SERP generally collects three types of surface water samples are collected: suspended matter samples, filtered, and unfiltered, samples in that order. Each of these types of samples are collected in duplicate by collecting from successively collected volumes. The quantity of each subsample collected is recorded in the field notebook. Surface water samples are collected according to the following procedures:

I. Suspended Matter Samples for Chlorophyll Analysis
   a. Use clean 140 ml polypropylene syringes to collect suspended matter samples.

b. Place the syringes to draw water 10 cm below the surface of the water into the direction of water flow (if applicable).
c. Partially fill the syringe and rinse with sample water three times.
d. Completely fill the syringe.
e. Using filter forceps, put a 25 mm Whatman GF/F glass fiber filter into a 25 mm in-line filter holder.
f. Attach the filter holder to the end of the syringe.
g. Force a known amount of sample water (50 - 200 ml) through the filter (Do not rinse the filter first). Discard Filtrate.
h. Record the amount of water filtered in the field notebook, along with date and time of sample collection.
i. Transfer the filter, with the filter forceps, to a 1.8 ml microcentrifuge tube.
j. Add 1.5 ml of acetone to the tube with a disposable polyethylene pipet. Acetone extracts the chlorophyll from the cells collected on the filter.
k. Check that tube is properly labeled.
l. Place the tube immediately in the cooler on ice and in the dark.

II. Filtered surface water samples for inorganic nutrient determinations
a. Use clean 140 ml polypropylene syringes to collect filtered surface water samples.
b. Place the syringes to draw water 10 cm below the surface of the water into the direction of water flow (if applicable).
c. Partially fill the syringe and rinse with sample water three times.
d. Completely fill the syringe.
e. Using filter forceps, put a 25 mm Whatman GF/F glass fiber filter into a 25 mm in-line filter holder.
f. Attach the filter holder to the end of the syringe and force about 10 ml of sample through the filter to rinse.
g. Use the remaining filtrate from syringe to rinse a 60 ml HDPE sample bottle three times.
h. Fill syringe with sample water again (if necessary), and re-attach filter holder with filter.
i. Fill sample bottle to neck and cap. Multiple syringe volumes may contribute to a single sample bottle.
j. Repeat steps *a* through *h* to collect a duplicate sample.
k. Check that the sample bottles are properly labeled.
l. Record date and time of sample collection in the field notebook.
m. Place the samples in a cooler with ice.

III. Unfiltered surface water samples for total nitrogen, phosphorus, and carbon and alkaline phosphatase activity determinations:
a. Unfiltered surface water samples are collected directly into clean 125 ml HDPE bottles.
b. Submerge the bottles neck first to about 10 cm below the surface of the water.
c. Invert the bottle with neck upright and pointing into the direction of water flow (if applicable).

d. Partially fill the bottle (at least 25 percent filled) cap, and shake, and pour the rinse water downstream of the sampling location.
e. Repeat this procedure two more times for a total of three rinses.
f. Fill the bottle to the neck and cap.
g. Repeat procedures *a* through *f* to collect a duplicate sample.
h. Check that the sample bottles are properly labeled.
i. Record date and time of sample collection in the field notebook.
j. Place the samples in a cooler in the dark. Alkaline phosphatase activity is a microbiological parameter, therefore, these samples can not be stored on ice. Once alkaline phosphatase activity has been determined on the samples, the remaining sample for the inorganic parameters are stored in a refrigerator.

When access to the surface water can not be made by boat or wading, such as from a bridge or side of canal, then a clean plastic bucket attached to a line is used to collect the surface water sample in bulk. This bucket is rinsed with sample water three times, with the rinse water poured downstream of the sampling location, prior to collection of the sample. Sample bottles, syringes, and filters are then rinsed and filled from the water collected in the bucket following the procedures described above. If necessary, split samples are collected from consecutive sample volumes from the same sample device.

When water samples are to be collected from depths below the water surface, a Niskin sampler is used. The sampler is cocked open, then lowered from the boat to the appropriate depth, closed at depth, and returned to the surface. Sample bottles, syringes, and filters are then rinsed and filled from the water collected in the bucket following the procedures described above. For good comparability between duplicate samples and all parameters, it is important to fill all sample bottles for a location from one cast of the Niskin sampler. If for some reason all of the bottles cannot be filled from one cast, as may happen if the Niskin sampler does not fill completely or that some of the sample water is lost due to spillage, then any sample already put into bottles needs to be rinsed and filled again with water collected from a new cast of the Niskin sampler.

6.4.2 Field Measurements

Temperature, salinity/conductivity, pH, dissolved oxygen, and light attenuation coefficient are measured directly in the field at each sampling location using properly calibrated (Section 9) portable electronic meters and/or a SEA-BIRD Model 19-03 CTD. These measurements are taken contemporaneously with the sample collection to ensure direct correlation of laboratory results with field measurements. Temperature, salinity/conductivity and dissolved oxygen are measured both at the surface and at the bottom of the water column; pH is measured at the surface.

The water depth is determined from a depth finder on the boat and/or the pressure transducer on the SEA-BIRD CTD. Meter probes are attached 10 cm from the bottom of the weighted line to obtain bottom water measurements.

6.4.2.1. Temperature

Surface temperature is measured (in ^{0}C) by submersing the probe of the salinity/conductivity/temperature (SCT) meter 10 cm under water. After the digital readout stabilizes (less than 5 minutes), the temperature is recorded in the field notebook. The probe is then lowered to 10 cm from the bottom of the water column. After the digital readout stabilizes, the bottom temperature is recorded in the field notebook. Temperature can also be measured by the thermistor on the SEA-BIRD CTD.

6.4.2.2. Salinity/Conductivity

Surface salinity and conductivity are measured in units of parts per thousand (ppt). Surface and bottom salinity are measured contemporaneously with temperature. The probe of the SCT meter is submersed 10 cm under water. After the digital readout stabilizes (less than 5 minutes), the surface salinity is recorded in the field notebook. The probe is then lowered to 10 cm from the bottom of the water column. After readout stabilization, the bottom salinity is recorded in the field notebook. Salinity and conductivity can also be measured by the SEA-BIRD CTD.

6.4.2.3. pH

An automatic temperature compensation (ATC) probe on the pH meter adjusts the pH reading for temperature differences between standards and samples. A sample of surface water is collected in a clean, 400 ml polyethylene beaker after it is rinsed three times with sample water. The pH probe and ATC probe are submersed in the beaker, and the pH is recorded in the field notebook. Successive aliquots of surface water are collected until the pH of three successive aliquot agrees within 0.02 pH units.

6.4.2.4. Dissolved Oxygen

Automatic temperature, atmospheric pressure and salinity corrections are made by the Orion model 840 Dissolved Oxygen meter. Switch the salinity compensation on, and use the Mode Key Pad to select the Cal mode. The last salinity entered in the system is displayed. Adjust the salinity display with the up and down arrow keys to match the previously-measured station salinity. Dissolved oxygen (DO) concentration, in mg/l, is determined from the surface water by submersing the probe 10 cm. The probe is gently agitated to approximate a velocity of 15 cm/sec past the membrane. After a brief equilibration time, the meter displays a stable DO reading. Once the surface DO is recorded in the field notebook, the probe is then lowered to 10 cm from the bottom and gently agitated to approximate a flow of 15 cm/sec past the membrane. After a stable reading is reached, the DO of the bottom water is recorded in the field notebook. Dissolved oxygen can also be measured by the dissolved oxygen sensor (SB23B) on the SEA-BIRD CTD.

6.4.2.5 Light Attenuation Coefficient

Light attenuation coefficient is a measurement of the attenuation of photosynthetically active radiation (PAR) as measured by two spherical quantum light sensors (LI-COR model LI-193SA) at different depths in the water. The two light sensors are mounted on two extensions, each 90^{o} apart, from a PVC pole, such that one probe is held just below the water surface, while the other is 0.5 or 1 m below (depending on water depth). The measurements are made and the ratio of light at depth

(l_z)/ light near surface (l_o) is calculated by a LI-COR model LI-1000 DataLogger. After a stable reading is reached, the ratio value and distance (z, 0.5 or 1 m) are recorded in the field notebook. The light attenuation coefficient is then calculated as $\ln(l_z/l_o)/z*-1$. Light attenuation coefficient can also be calculated by PAR measured by quantum light sensor on the SEA-BIRD CTD.

6.4.3 Pore Water Sample Collection

SERP collects pore water from either temporary or permanently placed lysimeters. Prior to sampling, the water level and bottom of the lysimeter are measured to determine the volume of water in the lysimeter. Using either a peristaltic pump or a syringe, the lysimeter is purged of three volumes of standing water or pumped dry. The volume of water removed is recorded in the field notebook. Specific conductance, temperature, and pH are monitored while purging if the lysimeter produces sufficient volume of water. If the lysimeter does not produce enough water then it is pumped dry and sampled immediately following recovery. Since SERP does not sample hazardous water, the water purged from the lysimeter is allowed to drain on the ground but away from the lysimeter. Pore water is collected in sample bottles according to the procedures for surface water outlined in Section 6.4.1.

6.4.4 Soil and Sediment Sample Collection

SERP collects surface and subsurface soils and sediment samples according to the following protocols.

6.4.4.1 Surface Soil Samples

Surface soil samples are collected from the upper 10 cm of an undisturbed location. Surface detritus is removed prior to sample collection. The surface soil samples are collected with a stainless steel trowel, spade, PVC core, polycarbonate core or by hand and placed into plastic, wide-mouth specimen cups. The physical parameters of the soil, including color, moisture content, presence of biota, and texture are described in the field notebook if required to satisfy the project objectives. The sample depth, date and time of sample collection, and the amount of sample (or subsamples) collected are also recorded in the field notebook. Roots may or may not be removed from the soil samples depending upon the project objectives.

Soil samples are homogenized either in the field or in the laboratory, depending upon the project objectives. If homogenized in the field, the soil sample is placed into a polypropylene mixing tray and homogenized by slicing, mixing, and remixing of the sample. The homogenized soil sample is then placed into a wide-mouth specimen cup and stored in a cooler in the dark for transport to the laboratory. In the laboratory, soil samples are homogenized by mixing the entire sample in a blender.

6.4.4.2 Subsurface Soil Samples

Subsurface soil samples are collected using either polycarbonate or PVC core tubes, pushed into the soil or sediment by hand by twisting the tube in a circular clock-wise and then in a counterclock-wise movement. The depth of the soil surface on the outside and on the inside of the core tube is

measured and recorded to determine compaction.

Once the core is extracted, plastic caps or neoprene rubber stoppers are inserted and taped to the end of the tube to prevent slippage and spillage. The top direction of the core tube is marked on the tube along with the sample number and the tube is stored in an upright position during transport to the laboratory. In the laboratory, the soil or sediment is extracted from the core tube and using a stainless steel knife, a sample for analysis is collected from the center of the tube, away from the sides. The physical characteristics of the soil are described in the field notebook, along with the approximate amount of sample (or subsample collected).

In general, soil sample compositing or splitting in the field is not preferred due to potential contamination concerns; the collection of duplicate samples in the field by collecting soil from the same sample source and homogenization of the samples in the laboratory with a blender, is preferred. If samples are to be homogenized in the field, then the samples will be extracted from the core tube onto a polypropylene tray and mixed with a stainless steel or Teflon spatula. The homogenized samples are then placed into plastic, wide-mouth specimen cups and stored in a cooler in the dark for transport to the laboratory.

6.4.4.3 Sediment Sample Collection

Sediment is collected using either polycarbonate or PVC core tubes or with an Ekman Dredge. The sediment sample is removed from the tubes or dredge and placed in a polypropylene tray. A stainless steel knife is used to collect a section of soil from near the center of the sample container. These samples may be homogenized in the field by mixing with a spatula or homogenized in the laboratory using a blender. Samples are stored in plastic wide-mouth specimen cups and stored in a cooler in the dark for transport to the laboratory. The amount of sediment collected, all equipment used, the method of homogenization, and the amount of sample stored are documented in the field notebook.

6.4.5 Tissue Sample Collection

Plant tissue samples are collected by gathering the plants by hand and placed into plastic bags. The plant samples are kept in a cooler on ice until transported to the laboratory.

6.5 Sample Documentation and Identification

All sample bottles are pre-labeled in the laboratory prior to transport to the field site. Labels of colored tape are attached to the side of the bottle. Water-proof ink pens are used to mark the labels with a unique sample number (Section 7). Sample containers used for the suspended matter samples, sediment samples and tissue samples are used only once, and are marked with water-proof ink pens directly on the outside of the sample container. The collection of all samples is recorded in the field notebook.

6.6 Documentation

The following is a list of the field records that are maintained:

1. Field Equipment Checklist
2. Field Notebook with the field data sheets.
3. Field Instrument calibration Sheet.
4. Chain of Custody Form.

6.7 Sample Preservation, Holding Times, and Sample Volume

Sample containers, sizes, preservatives, and maximum holding times, by parameter are included in Table 6.4. Note, there are two columns listing sample holding times prior to analysis. SERP recognizes that samples should be analyzed as soon as possible after sample collection and the holding times are defined from date/time of sample collection. SERP has instituted its own maximum holding times as goals. In almost all cases, the SERP holding times are more stringent than those established by EPA (Table 6.4). The SERP holding times are almost always met. In the case of filtered soluble nutrients, however, if the SERP holding times are exceeded, then the samples are frozen and analyzed within the maximum holding time. Every effort is made to ensure that the EPA holding times are not exceeded, but, should a sample be analyzed after the maximum holding time, the data for that sample will be marked with a qualifier code in the data report.

In the event that filtered samples need to be frozen, then each sample bottle is examined to determine that there is adequate space for expansion (i.e. the sample bottle can only be 3/4 full). If needed, sample is removed from the bottle so that it is no more than 3/4 full. Prior to analysis the sample bottles are allowed to thaw slowly (2 - 3 hours) to room temperature then shaken well to ensure that all constituents are re-distributed evenly throughout the sample as they were at the time of sample collection. Clementson and Wayte (1992) have demonstrated that freezing of water samples results in no change in dissolved nutrient concentrations for at least 4 months (See Appendix C). SERP has also demonstrated that freezing water samples has no effect on soluble nutrient concentrations, including ammonia, for at least 35 days (See Appendix C).

Samples are preserved in the field, immediately following sample collection. For the most part, sample preservation requires placing the sample in a cooler with ice, and in the dark. Suspended matter samples collected for chlorophyll-a determination are preserved in the field by adding 1.5 ml of acetone to the HDPE microcentrifuge tube with a disposable pipet. The chlorophyll-a samples are then stored in a cooler in the dark on ice. Equipment blanks for chlorophyll-a determination are preserved with the same amount of acetone and stored in the dark on ice. Any additional chemical used to augment preservation in the field will be from the same source as the chemical used to the preserve the sample and any additional preservative added to the samples is documented on the field data sheet. The acetone preservative used is of ACS reagent-grade or better; obtained fresh from the laboratory stocks on a daily basis; and transported to the field in an HDPE bottle.

SERP does not use acid preservatives for ammonia, total nitrogen, total phosphorus, and organic carbon samples. The addition of even small amounts of acid to a sample bottle collected for ammonia determination enhances the uptake of ammonia into the sample bottle from the atmosphere. The concentrations of ammonia most commonly determined in the waters sampled and analyzed by SERP are less than 0.05 ppm (3.6 μM). At these low concentrations, even the smallest uptake of ammonia into a sample from the atmosphere will be detected and produce an

anomalous high result.

Samples collected for total phosphorus and total nitrogen are processed immediately upon receipt in the laboratory (within 12 hours of sample collection) according to the sample handling procedures for these methods (Appendix A and B), therefore, preservation with acid is not required. Samples collected for total organic carbon are refrigerated, without acidification, until analysis. SERP has demonstrated that there is no difference in TOC concentrations between samples preserved with or without acid for at least 33 days (See Appendix D).

6.8 Sample Dispatch

For most projects, samples are stored on wet ice in coolers and delivered to the laboratory by the field personnel on the same day of sample collection. SERP performs its own laboratory analyses; however, if samples must be sent to an out-side laboratory they will be shipped on the same day of sample collection to the laboratory using a common carrier and overnight delivery. These samples will be carefully packaged with bubble wrap or styrofoam to prevent breakage. Individual or duplicate samples will be placed in individual plastic, sealable, bags to prevent cross-contamination if the sample bottles break. Insulated coolers will be used for sample shipment. The lids and drain ports of the coolers will be securely sealed with shipping tape to avoid opening. The samples will be preserved in the coolers with wet ice, if appropriate.

6.9 Reagent Storage and Waste Disposal

6.9.1 Reagent Storage

The type of reagents typically transported to the field by SERP are limited to pH buffers, salinity/conductivity standard and acetone. The storage and transport procedures for these reagents are listed in Table 6.5.

6.9.2 Waste Disposal

Field generated wastes are kept to a minimum since soaps, acids, and solvent compounds are not used during equipment decontamination procedures. In addition, SERP does not perform sampling of hazardous waste sites. The only wastes generated during sampling include calibration standards for pH and acetone. The field calibration standards are taken back to the laboratory, neutralized and/or diluted then flushed down the sanitary sewer. Any acetone that may be remaining on the pipet tips is allowed to evaporate, then the pipet tips are disposed in trash receptacles.

TABLE 6.5.
Field Reagent Storage

Chemical	**Method of Storage**
pH Buffers	Stored in original containers in the laboratory and transferred to 60 ml HDPE bottles in ziplock bags for transport to the field.
Salinity\conductivity standard	Stored in their original containers in the laboratory and transferred to 250 ml HDPE bottles for transport to the field.
Acetone	Stored in original steel container in a vented cabinet designed for flammable storage. Cabinet in laboratory is locked and labeled as containing flammable substances. Transferred to a 250 ml HDPE squeeze bottles in ziplock bags for transport to the field.

7.0 Sample Custody

Sample custody is the responsibility of the sampling team and of the Chief Chemist. The sampling team is responsible for labeling, collecting, documenting in the field notebook and transporting samples to the laboratory. The Chief Chemist is responsible for proper storage of the samples within the laboratory, and that the samples are analyzed within their appropriate holding times. Figure 7.1 shows the centralized receipt log form used for any incoming sample set at SERP. This centralized receipt log form is maintained in a loose leaf notebook at the Chief Chemist office. Samples are not discarded until the analytical results are checked and approved by the Quality Assurance Officer. All documentation/logs are signed/initialed by appropriate personnel.

Currently, all analyses of all samples collected by our laboratory are performed by our laboratory. However, should the need arise to send samples via courier to another laboratory for analysis, then the SERP sample chain-of-custody form (Figure 7.2) and/or one supplied by the contract laboratory, including all necessary information, will be used. This form will be included within the sample cooler and protected within a plastic, sealable bag. A copy of the form will be retained by SERP in project specific files. Upon receipt of the samples, the receiving laboratory will be requested to sign the sample chain-of-custody form and send a copy of the signed form via mail or facsimile to SERP. The QA officer will be in charge of ensuring that a copy of the signed chain-of-custody form is obtained from the receiving laboratory.

SERP also often receives samples (surface water, ground water, soils, sediments, and tissue) collected by other researchers for analysis. A completed SERP chain-of-custody form will be required to accompany the samples and will be signed by the QA officer or SERP technician receiving the samples. These samples will be inspected by the SERP QA officer or SERP technician for integrity and completeness according to the chain-of-custody form. Any discrepancies between the samples received and the chain-of-custody form and/or missing information will be reported immediately to the researcher that collected the samples. Any samples with visible contamination, leaks, damage, or odors will be noted on the form. A copy of the chain-of-custody form will be kept in the project specific files. Records of shipping receipts for outgoing and incoming samples are maintained indefinitely at SERP office. Sample personnel responsible for sample delivery are identified in the chain of custody form as well as common carriers that might have been used in the process.

Each sample container is labeled with a unique sample identification number as indicated below:

AAA###-###XX.

The first letters in the sample identification number (AAA) refer to the client or program name. For example, the three letters FBY would be used to designate SERP samples collected from Florida Bay. The first set of numbers (###) can vary from 1 to 999 and refers to the survey or batch number. The second set of numbers (###) is the site number or bottle number. The last letters (XX) refer to the type of sample (U=unfiltered, F=filtered, S=soil/sediments, T=tissue, C=chlorophyll a) and the duplicate letter (A or B, not applicable for some clients' samples).

Sample numbers are recorded on sample containers, the field data sheet, and sample chain-of-

custody/log-in form. When we receive samples collected by other researchers, a SERP technician will assign the unique code to each bottle at the time of receipt, and record it on the bottle and chain-of-custody/log-in form. An example of a sample label is included below:

FBY77-10UA

7.1 Field Custody

Loose-leaf field notebooks are used for all field documentation. Once QA checked, the sheets are removed from the notebooks and kept in project-specific files. All field notebook entries are made in waterproof ink and include the following: name and number of sampling trip; date of sampling trip; general weather and water conditions (waves and tides); name of individuals in sampling team; location and number of sample; and time of sample collection. For the collection of surface water samples, additional recorded information includes the water temperature (both surface and bottom), salinity/conductivity (both surface and bottom), pH, dissolved oxygen, the volume of water filtered, the depth of sample collection, the amount of preservative added, and a description of the water clarity (Figure 7.3). For pore water sample collection, additional information recorded in the field notebook includes the water level in the lysimeter, the bottom depth of the lysimeter, the volume of water removed during purging, and the specific conductance, temperature, and pH during purging (Figure 7.4). For soil and sediment samples, additional information recorded in the field notebook include the depth of sample collection, physical characteristics of the soil, and method of homogenization (Figure 7.5). For tissue samples, identifying characteristics of the plant are included in the field notebook (Figure 7.6). Each notebook page is signed by the sampling team. If an error is made in the field notebook, corrections are made by drawing a line through the error and entering the correct information next to the error.

In addition to the field notebook, the field sampling team keeps a field instrument sheet (Figure 7.7). On this sheet is recorded the number of each field instrument and probe, as well as instrument calibration check information. All sampling equipment and decontamination procedures are recorded, along with the use of any fuel powered units (boats, generators, or pumps).

7.2 Laboratory Custody

Upon transport of the samples to the SERP laboratory at FIU, the field sampling personnel log-in the samples on the Sample Checklist (Figure 7.8). All of the sample bottles are inspected for integrity, proper documentation (labelling), and preservation (cooler with wet ice). Any samples bottles found broken, leaking, not properly marked or not properly preserved are rejected from analysis, and noted on the bottom of the Sample Checklist. Samples are stored in the appropriate conditions, refrigerator or freezer, in a locked room, with access to the room limited to SERP employees. Standards are stored separately from samples.

The Sample Checklist serves to track the samples collected from a sampling event through sample analysis, data validation, and sample disposition. The Sample Checklist is stored in the project files. These files are checked on a daily basis by the chief Chemist and the QA Officer to ensure that samples are analyzed within their appropriate holding times. The checklist includes the date of sample collection, initials of field sampler(s), date of receipt in the laboratory, and requested analyses. Special

Figure 7.1
Centralized Sample Receipt Log-in Form

ANALYSES REQUIRED AND MAXIMUM ALLOWABLE HOLDING TIMES

DATE REC'D	PROJECT	SAMPLE #'S	# OF REPLICATES	MATRIX	APA 12 hrs	TURB 48 hrs	CHLA 21 days	TOC 28 days	TN 28 days	TP 28 days	NUTR 28 days	SI 28 days	TSI 28 days	DOC 28 days

Figure 7.2
Chain-of-Custody/Sample Log-in Form

SOUTHEAST ENVIRONMENTAL RESEARCH PROGRAM

OE 148 (office)/VH 321 (lab), University Park, Miami, FL 33199, 305-348-3095 Page _____ of _____

Chain of Custody Record/Sample Log

CLIENT/PROJECT NAME:	ACCOUNT NO.:	AUTHORIZATION:
DELIVERED BY:	RECEIVED BY:	DATE AND TIME:
RECEIPT ASSESSMENT/COMMENTS:		ICE IN COOLERS?:

BOTTLE ID	SAMPLE ID	MATRIX FW/SW	H2S OR ODORS ?	COLLECTION DATE	COLLECTION TIME	# OF REPLICATES	ANALYSES REQUIRED* APA	CHLA	TOC	TN	TP	NUTR	SI	TSI	DOC	SAMPLE COMMENTS

*: Analyses and methods APA (Alkaline Phosphatase Activity); CHLA (chlorophyll a, SM 10200H); TOC, DOC (Total and Dissolved Organic Carbon, EPA 415.1); TN (Total Nitrogen, Antek);

TP (Total Phosphorus, EPA 365.1); nutr (Soluble NO3, NO2 [EPA 353.2], NH4 [EPA 350.1], PO4 [EPA 365.1]); SI, TSI (Soluble and Total Silica, [EPA 370.1])

Figure 7.3
Surface Water Field Data Sheet

Sampling Event		Date		Names				Weather Conditions		
Station No.	**Station Name**	**Time**	**Depth**	**Temp**	**Salinity**	**D.O**	**Z (1;0.5)**	**Iz/Io**	**Volume Filtered**	**Comment**

Figure 7.4
Lysimeter Field Data Sheet

Sampling Event		Date		Names				Weather Conditions		
Station No.	**Station Name**	**Time**	**Water Level**	**Total Depth**	**Specific Cond.**	**Temp**	**pH**	**Sample Volume**	**Preservativ.**	**Comment**

Figure 7.5
Soil/Sediment Field Data Sheet

Sampling Event		Date		Names		Weather Conditions
Station No.	**Station Name**	**Time**	**Depth**	**Sample Volume**	**Homogenization**	**Description**

Figure 7.6
Tissue Field Data Sheet

Sampling Event		**Date**	**Names**	**Weather Conditions**
Station No.	**Station Name**	**Time**	**Description**	

Figure 7.7
Field Instrument Sheet

Sampling Event	Date	Names	Comments

Instrument Name	Instrument Number	Probe Number	Time	Calibration Check

Figure 7.8 Sample Checklist

Sample Checklist

Sampling Event:		Sample Nos.:	
Sampling Team		Sample Matrix:	
Sampling Date / Time:		Sample Disposal Date:	
Lab Receipt Date:		Initials:	

Analyses	Analysis Date/Init	SOP/ Issue Date	Data Entry Date/Init	QA Check Date/ Init
Salinity				
D.O.				
Temp.				
Alkaline Phosp.				
Turbidity				
TOC				
Nutrients				
Total P				
Total N				
Chlorophyll				
Other Analyses:				

Figure 7.9
TOC-5000 Log Book

Date	Sample ID	MAtrix	High Std		OP
8/13/98	K13 (268-276S, 250MS)	SW	20ppm	34082	CL
8-13-98	Cleaned Syringe				PL
8/14/98	Cleaned Column				CL
8/17/98	repacked, regenerated column				CL
8/18/98	K13 (290-397, 402-403) RRBB89-124 [illegible] (549-552)	FW/SW	20ppm / 50ppm	36084 / 27802	CL
8/20/98	BB90 (C1, C2, 101-123, 104MS)	SW	20ppm	17053	CL
8/21/98	RRBB90 (C1, C2, 101-123, 104MS)	SW	20ppm	34377	CL
8/24/98	BB90 (124-135) RRmeeder [illegible] (425-2), 123MS, RRK13 (274S/B, 275S/B)	SW	20ppm	34386	CL
8/31/98	MikeB. (40-62, MS K13 241)	FW/SW	20ppm	34660	CL
9/1/98	Richards (SP17C/D-SP23C/D, MS [illegible])	FW	20ppm / 50ppm	34443 / 31260	CL
9/2/98	TT190 (C1, 51-65, MS 61)	SW	20ppm / 50ppm	34297 / 30833	CL
9/3/98	RRMIC78 (2-41, MS16)	FW	20ppm / 50ppm	34430 / 30751	CL
9/11/98	Childers Doc (4150-4213, MS FB90 17)	SW	20ppm	34318	CL
9/14/98	RR Childers Doc (4215-4236, 4500-4539, MS S13 376)	SW	20ppm	34661	CL
9/15/98	Childers Doc (4540-4571) RRmikeB. (22, 27, 34, 38), RRFB90 87, MS 6	SW	20ppm	33626	CL
9/16/98	Scioto S1 (79-140), MS MICL28I #13EB	FW	20ppm / 50ppm	33678 / 30286	CL
9/17/98	Childers (9311-9373) MS S13 376	SW	20ppm / 50ppm	33706 / 31265	CL
9/21/98	MICL28I EB, Grab (21-25), RRmikeB #521, RRChilders (9374-9378, 9417-9418) MS [illegible]	SW	20ppm / 50ppm	33321 / 30051	CL
9/22/98	cleaned syringe, changed rubber stopper				PL
9/22/98	FB91 (C1, C2, 1-14, MS17)	SW	20ppm	35401	CL
9/23/98	Richards (SP0101-SP1602C/D) MS MICL28I		20ppm / 50ppm	34545 / 30083	CL
9/30/98	RR Richards (SP1701-SP2301C/D), [illegible], RRmeeder (441-451), RRChilders (9359, 9367)	FW/SW	20ppm / 50ppm	35117 / 30034	CL
10/5/98	FB92 (C1, C2, 1-14, MS13)	SW	20ppm	35903	CL
10/6/98	WWB92 (C1, 29-43, MS32)	FW/SW	20ppm / 50ppm	35407 / 31410	CL
10/12/98	K14 (281, 318-324, 342MS)	SW	20ppm	35391	CL
10/13/98	K14 (325-332S/B, MS 333)	SW	20ppm	39021	CL
10/14/98	Cleaned Column & repacked column				CL
10/15/98	regenerated column, lubricated rollers				
10/15/98	RR K14 (325-332S/B, MS 333BC)	SW	20ppm	37498	CL

Figure 7.10
RFA Nutrient Analyzer Log Book

Date/run	N+N slope, coeff(r) STD cal., %FS@FS	NO_2^-	NH_4^+	SRP	eff	31 hours	Init
10-26-98							
Keys NE	363, .999	2198, .999	1680, .990	1680, .999	8.08		
	622, 89.2%	-, 84.0%	640, 91.1%	822, 98.8%		2592	TKN
10/27/98							
Keys14e1	361, 0.999	2194, 0.999	-, -	-, -	9.17		
	600, 88.6%	-, 91.0%	-, -	-, -		2598	TKN
10-28-98							
SJ24b	395, .999	2137, .999	-	1692, .999	8.06		
	602, 78.8%	-, 98.6%	-	802, 83.9%		2602	TK
SJ2B1	324, .996		-		8.10		
	600, 78.0%	-, 99.6%	-	802, 83.9%		2604	OK
10-29-98							
SWF10B	363, .999	2148, .999	773, .996	1761, .998	7.30	2612	
	472, 97.1%	-, 93.4%	422, 99.1%	810, 96.2%		2612	TK
10/30/98	-, -	-, -	329, 0.999	-, -	-		
Keys14e5	-, -	-, -	470, 86.0%	-, -		2619	SO
11/2/98							
MC27	333, .999	2136, .999	318, .999	1781, .999	7.32		
	560, 91.8%	-, 92.1%	430, 94.5%	840, 91.8%		2625	SO
11-5-98							
WBSQ3B3	337, .999	2113, .999	326, .999	1739, .998	6.65		
	610, 81.7%	-, 92.1%	472, 88.0%	840, 87.5%		2631	TKN
11-6-98	Changed hypochlorite tube						
MC80	328, .999	2040, .999	364, .999	1669, .999	8.26		
	672, 90.0%	-, 84.6%	400, 98.0%	840, 85.1%			TKN
11-8-98	C.d column regenerated - left on during cleaning					2639	SO
MC80E	11-9-98 changed phenolate tube						
MC80E	343, .999	2263, .999	338, .998	1724, .999	7.35		
	730, 84.2%	-, 95.2%	330, 98.9%	840, 88.6%		2646	OK

Figure 7.11
RFA Total Phosphorus Log Book

Figure 7.12
RFA Silica Log Book

DATE	SAMPLE ID	MATRIX	RANGE	slope	corr coeff	INIT.
7/15/98	K1271598 (237-246b)	SW	0.02	99.89	.999	SB
7/15/98	K12715B (242b, 244sob, 245s, 291sob)	SW	0.02	114.38	.999	SB
7/16/98	WW889 (25-50, c1, c2)	SW	0.02	111.27	0.994	SB
7/17/98	K1371798 (281ys, 318b-329b)	SW	0.02	99.37	0.999	SB
7/17/98	K137176 (329s-332s)	SW	0.02	117.75	0.999	SB
7/20/98	K137C98 (333b-345b)	SW	0.02	105.7249	0.999	SB
7/21/98	K137 2198 (345S-350%)(351-359)	SW	0.02	102.84	0.999	SB
7/21/98	K137216 shelf 13 (360-361, #187-59, K12-2394/9)	SW	0.02	120.88	0.999	SB
7/22/98	K137 2298 (Shelf 362-374)	SW	0.02	102.47	0.999	SB
7/23/98	K137-2398 (shelf 375-378)	SW	0.02	102.31	0.999	SB
7/24/98	K12 72B (229 ms-uns, 342B-S, 4sw-4sa)	SW	0.02	101.39	0.999	SB
7/23/98	K137236 (shelf 13, 385, 386, 387, 399)	SW	0.02	123.46	0.999	SB
7/29/98	FB89 (c1, c2, 1-21)	SW	0.02	105.445	0.999	SB
7/29/98	FB89b (22-28)	SW	0.02	116.4199	0.998	SB
7/30/98	BB89 (c1, c2, 101-104, 108-113, 116, 121-124, 126-133)	SW	0.02	106.05	.999	SB
7/31/98	BB89b (134-135 add 103, 130, rr)	SW	0.02	100.6672		SB
8/1/98	K138198 (sisrr 385, 386, 387, 3005-2136)	SW	0.02	88.87	0.999	SB
8/3/98	K138398 (214-230b)	SW	0.02	81.99	0.998	SB
8/4/98	K138498 (231s-245s)	SW	0.02	89.54	0.999	SB
8/5/98	K138598 (245b-246b, 264%, 400-4da)	SW	0.02	97.32	0.999	SB
8/6/98	tt189 (c1, c2, 51-73)	SW	0.02	92.86	.999	SB

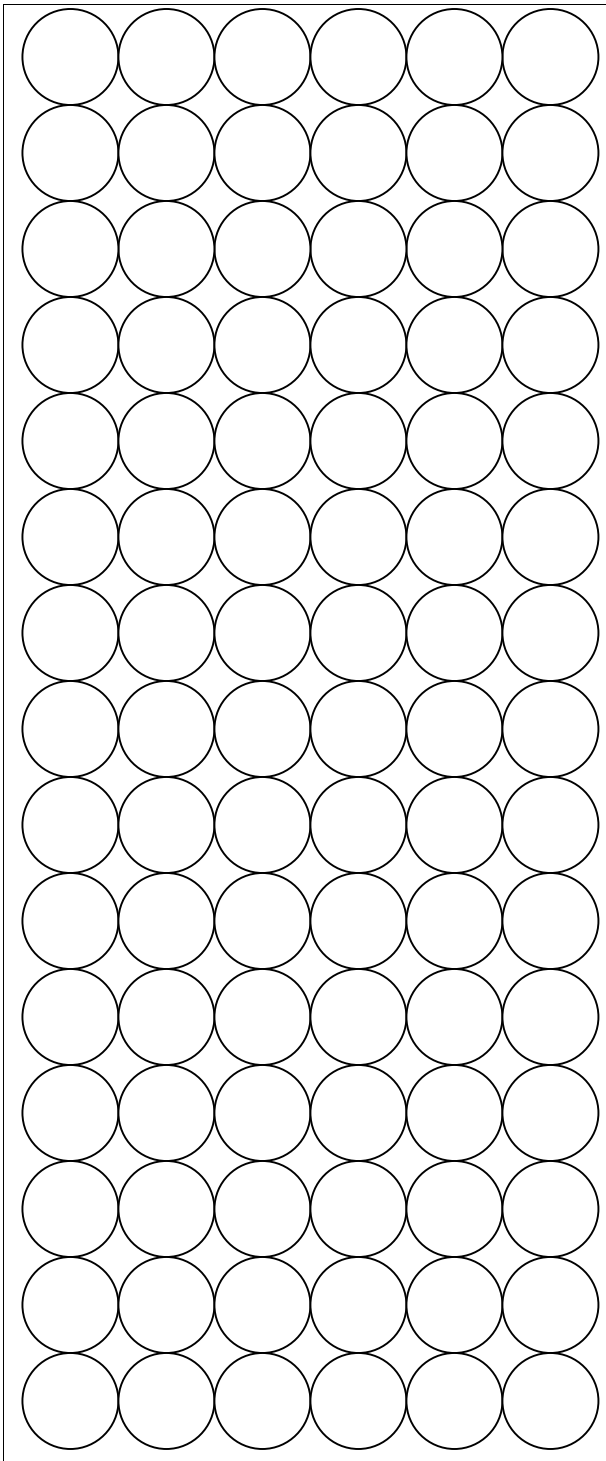
Figure 7.13
Antek Nitrogen Analyzer Log Book

Date	Description	#spl	CAL STD / QC CHK	VAC/HV	Operator
10-7-98	FB92(C1A-C2B, FB92UMS15A, FB92MS15A, 3A-13B)	30	486360/471935	25.0/300	JP
10-8-98	FB92(FB92UMS15A, FB92MS15A, 14A-26B)	30	470551/460654	25.0/300	JP
10-8-98	FB92(K14UMS283A, K14MS283A, 27A-28B) K14(265AB, 282A-291B)	30	486657/456313	25.0/800	JP
10-9-98	K14(K14UMS283A, K14MS283A, 292A-297B) WWB92(C1AB, 29A-34B)	30	482315/422656	25.0/800	JP
10-9-98	WWB92(WWB92UMS47A, WWB92MS47A, 35A-47B)	30	497158/452943	25.0/800	JP
10-15-98	WWB92(K14343BA, K14343BAMS, 48A-50B) K14(281, 318-321 SA, SB, BA, BB)	30	483031/440966	25.0/800	JP
10-15-98	K14(K14343BA, K14343BAMS, 322-327 SA, SB, BA, BB)	28	471977/446982	25.0/800	JP
10-16-98	K14(K14343BA, K14343BAMS, 328-333 SA, SB, BA, BB)	28	500768/470153	25.0/800	JP
10-16-98	K14(K14341BA, K14MS341BA, 334-339 SA, SB, BA, BB)	28	507251/475231	25.0/800	JP
10-19-98	Cleaned columns, rinsed + dried				BG JP
10-20-98	K14[K14UMS341BA, K14MS341BA) 340-345 SA, SB, BA, BB]	28	539568/515709	25.0/800	JP
10-20-98	K14(BW59UMS5, BW59MS5, 346-350 SA, SB, BA, BB) BW59(1-6)	30	503164/492839	25.0/800	JP

Figure 7.14
Fluorometer Log Book

	ANALYSIS		VOLTAGE	EXCITATION	EMISSION	CLN	SID
3/10/98	BLJS2	ChLA	700	435	667	S	168.1
3/12/98	BB85	ChL	700	435	667	S	169.6
3/18/98	Meeder	APA	425	430	507	S	164.6
3/19/98	MILLER	CHLA	700	435	667	S	164.5
3/23/98	MEEDER	ChLA	700	435	667	S	170.9
3/30/98	FB85	ChlA	700	435	667	S	185.0
3/30/98	WLB85	ChlA	700	435	667	S	188.4
4/27/98	WV6	MUF	625	365	445	S	
04/08/98	Len	MUF	625	365	445	S	
4/13/98	FB86	CHLA	700	435	667	S	194.0
4/18/98	USGS8	APA	425	430	507	S	155.3
4/20/98	WLB86	CHLA	700	435	667	S	189.0
4/20/98	BW53	CHLA	700	435	667	S	186.0
4/21/98	Meeder	APA	425	430	507	S	169.4
4/22/98	GAISER	CHLA	4700	435	667	S	185.0
4/27/98	MEEDER	CHLA	700	435	667	S	182.1
4/27/98	TTI 86	CHLA	700	435	667	S	180.0
4/27/98	GAISER	CHLA	700	435	667	S	181.0
4/27/98	USGS8	CHLA	700	435	667	S	182.5
4/28/98	FB86	CHLA	700	435	667	S	189.0
4/29/98	Gaiser	CHLA	700	435	667	S	187.0
5/6/98	Gaiser	CHLA	700	435	667	S	179.8
5/7/98	Keys12	CHLA	700	435	667	S	181.7
5/8/98	Shelf12	CHLA	700	.435	667	S	179.7
5/12/98	BW54	CHLA	700	435	667	S	184.0
5/12/98	BB87	CHLA	700	435	667	S	181.9
5-20-98	meeder	APA	425	430	507	S	165.6
5/20/98	K12	CHLA	700	435	667	S	163.0
5-21-98	WWB87	APA	425	430	507	S	163.2
5/22/98	WWB87	APA	425	430	507	S	164.6

Figure 7.15
Total Phosphorus Preparation Form



TOTAL PHOSPHORUS-WATER
SERP TP SOP 001-98
Tray contents:

_____
_____
_____

Prep (100 µl 0.17 N $MgSO_4$ + 5 ml sample per vial) and put in oven (80 ^{0}C):
(Date $MgSO_4$ was made _____)

_____ _____ _____
Date Time Init

Taken out of oven:

_____ _____ _____
Date Time Init

Put in muffle oven (550 ^{0}C)

_____ _____ _____
Date Time Init

Taken out of muffle oven:

_____ _____ _____
Date Time Init

Did the pellet melt? _____

Add acid (5 ml HCl), shake, and put in oven (80 ^{0}C):
Acid added: _____
(Date acid was made: _____)
(L=0.06N, M=0.12N, H=0.18N; if varied put the letter in each circle)

_____ _____ _____
Date Time Init

Taken out of oven:

_____ _____ _____
Date Time Init

Second shake:

_____ _____ _____
Date Time Init

Analyzed:

_____ _____ _____
Date Time Init

Vials discarded:

_____ _____ _____
Date Time Init

Comments or problems:

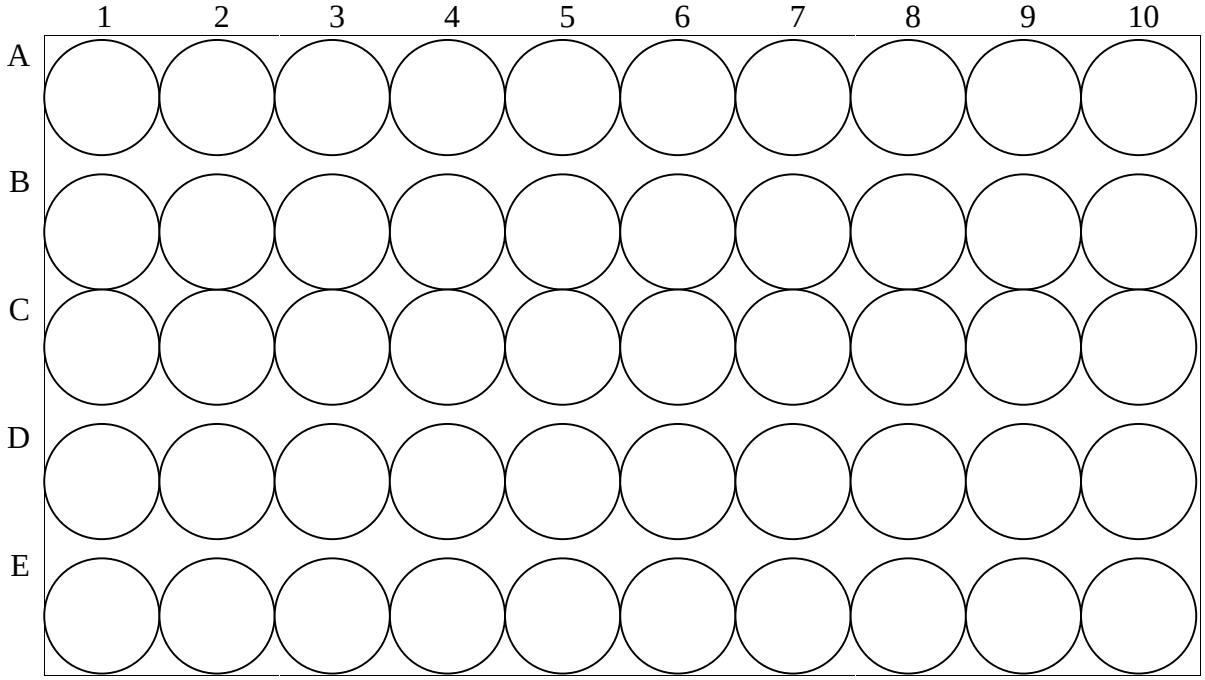
_____
_____
_____

Figure 7.16
Total Nitrogen Preparation Form

TOTAL NITROGEN PREPARATION LOG

Tray Contents:_____

Prepared by: _____ Date/time: _____ _____ HCl added? _____



preservation of samples is noted in the comments section of the checklist. Once the required analyses are performed on all of the samples collected in one sample event, the Sample Checklist is initialed and dated by the sample analyst. Sample analysis are also tracked in individual instrumentation notebooks and include sample handler name and sample number. Figures 7.9 - 7.14 are copies of pages from instrument logbooks for the TOC-5000, RFA nutrient analyzer, RFA total phosphorus analyzer, RFA silica analyzer, Antek total nitrogen analyzer, and fluorometer. Preparation of water and sediment/soil samples for total phosphorus and water for total nitrogen are recorded on sample preparation forms (Figures 7.15 and 7.16).

Ovens, refrigerators and freezers have digital temperature readouts that are monitored daily having the temperature recorded on the log posted on each oven (Figures 7.17 and 7.18). All oven, refrigerator, and freezer monitoring thermometers will be checked annually against a NIST-certified thermometer and the result of these checks and any necessary corrections will be recorded on the daily temperature logs.

The field notes, chain-of-custody/log-in form, sample checklist, and raw instrument data printouts for each sampling event are included in individual project files and kept indefinitely. These files are stored in a locked file cabinet. Once the laboratory data is entered into the computer data base and checked by the QA officer, the samples containers are directed to be either cleaned or discarded and the date is recorded in the sample Checklist form. Once the samples are discarded, the sample checklist is dated and initialed.

7.3 Electronic Data Records

Data from field measurements and laboratory analyses are compiled and summarized in computer spreadsheet format. We currently use Quattro Pro (Lotus 123 compatible) and Microsoft Excel. Separate spreadsheets for each sampling event are kept, and a compilation of all data to date is made. Spreadsheets are stored both on the hard drive of the computer, as well as onto write-protected floppy disks. In the event of computer equipment failure, the data files on the floppy disks are used as backup. The access to this electronic records is password protected. A hard copy of the spreadsheets are stored in the project files indefinitely.

All deletions or corrections will be documented on a hard copy of the spreadsheet and the person making the corrections will initial any changes.

Records of all aspects relating to changes, updates, problems and maintenance of the instrument and database software will be maintained in the instrument logbooks.

Figure 7.17 Refrigerator/freezer temperature Log

Daily refrigerator / Freezer temperature Log
Room _____ refrigerator

Date	Temp (^{0}C)	Date	Temp (^{0}C)	Date	Temp (^{0}C)

Figure 7.18 Oven temperature Log

DAILY OVEN TEMPERATURE LOG

ROOM _____ OVEN

Date	Temp (^{0}C)	Date	Temp (^{0}C)	Date	Temp (^{0}C)

8.0 Analytical Procedures

Section 5 includes the parameters and their corresponding analytical method numbers followed by SERP.

8.1 Laboratory Method Modifications

8.1.1 Autoanalyzer Methods

The methods for the inorganic nutrients (ammonium, nitrite, nitrate, and soluble reactive phosphate) are modified to be analyzed simultaneously by wet chemical analysis using a four-channel Alpkem RFA-300 (Rapid Flow Analyzer) Nutrient Analyzer (Alpkem Corp., Clackamas, OR) following the procedure for each inorganic nutrient as suggested by the Alpkem Corporation. The Alpkem methods for ammonium, nitrite, nitrate, and soluble reactive phosphate are listed in Table 5.2. Total phosphorus of water and solid samples are also determined on the Alpkem RFA following evaporation then dry ashing according to ASTM D-4638-86(9.2). Dissolved silica are also determined on the Alpkem RFA using method USGS I2700-85 (a slight modification of EPA method 370.1).

When determining concentrations of inorganic nutrients in seawater, SERP uses stock Sargasso Seawater or Gulf Stream water as analyte-free water in preparing method blanks and calibration standards. Prior to its use, the Sargasso Seawater or Gulf Stream water is analyzed to demonstrate levels of analyte less than 20% of the MDL (meaning it is not detectable).

8.1.2 Total Nitrogen in Water Samples

SERP prefers not to use Total Kjeldahl Nitrogen plus nitrate and nitrite for the determination of total nitrogen in water samples, because of the imprecision, insensitivity and tediousness of the procedure. Instead, SERP determines Total Nitrogen using an ANTEK 7000 Elemental Analyzer. This method was developed by Dr. Jones and ANTEK and involves injecting a small volume (5 μL) of sample into an oxidation furnace, where all combined nitrogen is converted to Nitric Oxide (NO). NO is then reacted with ozone to form Nitrous Oxide (N_2O), which is a chemiluminescent reaction. The light emission is detected and quantified by a photomultiplier tube. This method has been determined to produce results comparable to the Total Kjeldahl Nitrogen plus nitrate method with better estimates of precision and accuracy. A detailed description of this method along with a method validation package are included in Appendix A.

8.1.3 Alkaline Phosphatase Activity

The alkaline phosphatase activity (APA) assay measures the activity of alkaline phosphatase, an enzyme used by bacteria to mineralize phosphate from organic compounds (Hashimoto, Kitao, and Keiichiro, 1985. *Relationship between alkaline phosphatase activity and orthophosphate in the present Tokyo Bay*. Environ. Sci. Health, A20(7), 781-908). The determination of APA is currently under research by SERP in an effort to determine if APA can be used as a biological indicator. The assay is performed by adding a known concentration of an organic phosphate compound (3-*o*-

methylfluorescein phosphate (MFP)) to an unfiltered water sample. Alkaline phosphatase in the water sample cleaves the phosphate from the MFP, leaving 3-*o*-methylfluorescein (MF), a highly fluorescent compound. The concentration of MF at the end of the assay is proportional to the APA of the sample.

APA measurements are made within 12 hours of sample collection. Duplicate 3 ml subsamples from each sample bottle are pipetted into disposable cuvettes, and 30 µl of MFP solution are added to each. The MFP solution is prepared by dissolving 0.05255 g of anhydrous 3-*o*-methylfluorescein phosphate in 100 mM Tris buffer, pH=8.7. The concentration of the final stock solution is 1 mM.

The fluorescence of the subsamples are immediately measured using a Gilford Fluoro IV or Shimadzu RF-1501 Spectrofluorometer (excitation = 430 nm, emission = 507 nm) or Shimadzu RF-Mini 150 Fluorometer (filters) and recorded. The subsamples are then incubated for 2 hours in an incubator at 25 degrees Centigrade, and then the fluorescence of the samples is measured again using the same excitation and emission wavelengths. The amount of MF produced in 2 hours is quantified by comparison to a standard curve.

A stock standard solution of 3-*o*-methylfluorescein is diluted to make working standards that bracket the concentration of MF in the APA assays after 2 hours. Working standards are made up from standard stock solution and the fluorescence of the working standards is measured each day that the analyses are performed. Standard stock solution of 3-*o*-methylfluorescein is prepared by dissolving 0.0346 g of 3-*o*-methylfluorescein in 100 ml of methanol for a resulting concentration of 1 mM. Working standards of 0, 1, 2.5, 5 and 10 µM 3-*o*-methylfluorescein are then prepared by diluting the standard stock solution in analyte-free water.

8.1.4 Total Phosphorus

For the determination of total phosphorus in water, soil, sediment, and tissue samples, SERP does not use the typical ammonium persulfate digestion because of the explosive hazards and special handling requirements associated with the use of this chemical. Instead, SERP uses the sample preparation methods described by Solórzano and Sharp (1980. *Determination of total dissolved phosphorus and particulate phosphorus in natural waters.* Limnol. Oceanogr., 25(4), pp. 754-758; see Appendix B). Total phosphorus is determined in water, soil, sediment, and tissue samples by oxidizing and hydrolyzing all of the phosphorus-containing compounds in a sample to soluble reactive phosphate, and determining the soluble reactive phosphorus concentration by the EPA Method 365.1. For water samples, 100 ml of 0.17 N $MgSO_4$ is added to 5 ml of the water sample in a 8 ml glass scintillation vial and evaporated to dryness in a 80^{o}C oven (usually overnight). Once dry, the sample is ashed at 550^{o}C in a muffle furnace for 3.5 hours and allowed to cool overnight. The sample is then hydrolyzed with the addition of 5 ml of hydrochloric acid. The normality of the acid is dependent on the salinity of the sample, ranging from 0.06 N HCL for freshwater samples to 0.12 N HCl for seawater samples. The samples are then shaken, put into an 80^{o}C oven for 3 hours, shaken again, then allowed to cool in the oven overnight.

Soil, sediment and tissue samples are prepared in the same manner as the water samples except they are first dried in an 80^{o}C oven for 2 days then ground. Approximately 25 mg of sample is put into

a 20 ml glass scintillation vial with 1 ml of DIW and 200 µl of 0.17 N $MgSO_4$, then dried and ashed as described for water samples, except they are hydrolyzed with 10 ml of 0.24 N HCl.

SERP has analyzed NIST standard reference material 1572 (citrus leaves) as well as replicate samples of sawgrass according to the method described above. The results of these samples are included in Appendix B. The concentration of phosphorus in the NIST standard is reported as 1300 µg/gm ± 200 µg/gm, depending upon the analytical method. SERP's analysis of the NIST standard resulted in an accuracy range of 99% to 101% recovery. Precision ranged between 1 and 3% RSD.

8.1.5 Silica

For the determination of silica in water, SERP uses USGS method I2700-85 (a modification of the EPA 370.1 method) using the protocol outlined by Perstorp Analytical Environmental for analysis on an Alpkem RFA. This modification involves the addition acidified (with sulfuric acid) ammonium molybdate and oxalic acid to the water sample, subsequent reduction with ascorbic acid, and the spectrophotometric measurement of the resulting color development at 660 nm.

8.1.6 Chlorophyll a

A modification of the SM 10200H chlorophyll a method is used by SERP. Each sample is collected according to the protocol given in Section 6.4.1. Saturated magnesium carbonate is not added to each filter as preservation is not necessary since acetone is immediately added to the filters. Extraction of the pigment is done in a microcentrifuge tube with 1.5 ml of acetone, in the dark, at -20□C, for several days. Before analysis on a spectrofluorometer, each filter is pushed down into the tip of the tube with a stirring rod, and centrifuged for 3 minutes. In a glass cuvette, 0.75 ml of the sample and 2.25 ml of acetone are combined and the relative fluorescence at an excitation of 435 nm and emission of 667 nm is recorded.

8.2 Laboratory Operations

8.2.1 Laboratory Glassware Cleaning

All laboratory glassware is cleaned by rinsing with hot tap water, washing within Liquinox in hot tap water, rinsing with hot tap water, rinsing with 10% HCl, then rinsing three times with analyte-free (deionized water) water. Glassware used for determination of total and dissolved organic carbon analyses are also soaked in RBS35 (a dichromate-sulfuric acid mixture substitute) for 12 hours prior to rinsing. Once dried, all glassware is stored in one area of the laboratory in cabinets separate from reagents and standards. Class A volumetric glassware is not baked.

8.2.2 Reagent and Chemical Storage

All reagents and chemicals used in the laboratory are listed on Table 8.1. The method of storage for each reagent is also included on Table 8.1. Small quantities of reagents to satisfy a month or two of

analyses are kept in the laboratory. Each class of chemical is kept in its own dedicated storage area. Larger quantities of reagents are kept in a locked, outside storage area, with limited access. While being used in the laboratory, compressed gas cylinders are secured upright with straps or chains. New and empty compressed gas cylinders are also secured upright in an outside storage area that is locked with limited access. As each reagent or chemical is received it is dated and initialed by the person unpacking it. When the container is opened for the first time it is dated again and initialed by the opener.

8.2.3 Waste Disposal

Wastes produced in the laboratory include liquid acids, bases, salt mixtures and acetone. Many of these reagents are spent during sample prep and analysis. Any remaining waste acids and bases are neutralized then washed down the sink to the sanitary sewer. Non hazardous salt mixtures are diluted and washed down the sink. Small quantities of acetone is washed down the sink with large volumes of water. Empty reagent bottles are rinsed with hot tap water and disposed in trash receptacles. SERP never stores wastes; therefore, segregation and storage protocols are not needed. Since all wastes are either put down the drain or in trash receptacles, no documentation of waste disposal is needed. Dade County sewer discharge requirements and restrictions are followed.

TABLE 8.1
Reagent and Chemical Storage

Chemical	**Method of storage**
Laboratory Chemicals	
Mineral Acids (a)	Stored in original glass containers in a cabinet dedicated to acid storage.
Liquid Bases (a)	Stored in original glass containers in a cabinet dedicated to corrosive substances.
Liquid Oxidizers (a)	Stored in original plastic container in a dedicated cabinet.
Organic Solvents (a)	Stored in original glass containers in a vented cabinet designed for flammable storage. Cabinet in laboratory is locked and labeled as containing flammable substances.
Compressed Gases	Secured upright in laboratory and in outside, locked, storage area.
Dry Chemicals	Stored in original containers in alphabetical order in a dry cabinet.
pH Buffers	Stored in original containers in dedicated cabinet.

(a) Small quantities are stored in dedicated cabinets within the laboratory. Larger quantities are stored in an outside storage area that is kept locked with limited access.

9.0 Calibration Procedures and Frequency

9.1 Instrument Lists

Laboratory and field instrumentation are listed in Table 9.1.

9.2 Standard Receipt and Traceability

Primary standards traceable to NIST reference standards are purchased from reliable scientific supply firms. The standards are received by the Chief Chemist, inspected, dated, initialed, and stored in the appropriate storage area for that standard (desiccator, refrigerator, or freezer). Once opened, the standards are dated and initialled again. The manufacturer's certificates for each standard received are kept on file in a central location.

9.3 Standard Sources and Preparation

The source, preparation, and storage of standards are included on Table 9.2. Primary standards are prepared by dissolving the source standard into analyte-free deionized water. Secondary and working standards are prepared by diluting the primary standards in deionized water. Standard preparation methods are detailed in the laboratory SOP. The date, concentration, chemical vendor, lot number, and technician's initials for all standards made are recorded in the standard and reagent preparation logbook (Figure 9.1). Once prepared, the standard bottles are dated and initialed, then preserved according to the methods summarized in Table 9.2. Preservation method, storage location, and expiration date are also recorded on the standard bottles. Primary standards are produced at least quarterly, while working standards are produced daily. As no new standard or reagent is prepared until the previous one has been either completely used or expired and discarded, the logbook records link the preparation with every specific analysis.

9.4 Instrument Calibration

All field and laboratory instruments are calibrated, and checked for proper function prior to analysis. Table 9.3 summarizes the calibration procedures for field instruments, while Table 9.4 summarizes calibration procedures for the laboratory instruments. Calibration procedures for all instruments are described below.

9.4.1 Field Instruments

Field instrument calibration checks are recorded on the Field Instrument Calibration Sheet included as Figure 7.2. These sheets are kept in project specific files.

TABLE 9.1
Instrument List

Manufacturer	Model	Parameters	Matrix
Laboratory Equipment			
Alpkem	4-channel RFA 300 Rapid Flow Analyzer.	Ammonium, Nitrate, Nitrite, Ortho-phosphate, Total Phosphorus, Silica	SW, PW, SED
ANTEK	7000 Elemental Analyzer	Total Nitrogen	SW, PW
Shimadzu	TOC-5000 Total Organic Carbon Analyzer RF-1501 Spectrofluorometer RF-Mini 150 Fluorometer	Total Organic Carbon, Dissolved Organic Carbon Chlorophyll, Alkaline Phosphatase Activity Alkaline Phosphatase Activity	SW, PW
Gilford Instruments	Fluoro IV Spectroluorometer	Chlorophyll, Alkaline Phosphatase Activity	SW, PW
Carlo Erba	1500 CHN Analyzer	Total Carbon, Total Nitrogen	S, SED, T
Fisher Hewlett Packard	Models 255G and 255D IsoTemp Ovens Model 5890 Oven	Total Phosphorus Total Carbon, Total Nitrogen Bulk Density	SW,PW, S, SED S, SED S, SED
Allied	Model 7303DA Balance	All Parameters	All Matrices
Blue	LabHeat Muffle Furnace	Total Organic Carbon	S, SED
HF Scientific	DRT-15 C Turbidimeter	Turbidity	SW, PW

Table 9.1 Continued
Instrument List

Manufacturer	Model	Parameters	Matrix
Field Equipment			
Orion	140 Conductivity/Salinity/ Temperature Meter	Salinity Temperature	SW, PW
Orion	840 Oxygen Meter	Dissolved Oxygen	SW, PW
Orion	SA 250 Meter and Ross Combination Electrode	pH	SW, PW
LI-COR	LI-1000 DataLogger LI-193SA Quantum Light Sensors	Photosynthetically Active Radiation/Light Attenuation Coefficient	SW
SEA-BIRD	SEACAT SBE 19-03 Conductivity/Temperature/ Depth Meter	Temperature Conductivity/Salinity Dissolved Oxygen Turbidity Photosynthetically Active Radiation Depth	SW

Figure 9.1 Standard and Reagent Logbook

Date	Reagent	Final conc. (Vol or wt added / vol. final)	Lot #	Initials
4/22/98	Diluted HCL	5ml / 1 l	FISHER 967469	JM
4-23-98	Diluted HCL	10mL / 1 l	Fisher 967469	PL
4/27/98	Diluted HCL	10 ml / 1 l	FISHER 967469	PL
4/27/98	Diluted HCL	5ml / 1 l	FISHER 967469	PL
5-1-98	Diluted HCl	20ml / L	Fisher 967469	PL
5-5-98	Diluted HCL	20mL/L	Fisher 967469	PL
5/4/98	Diluted HCL	10ML/L	Fisher 967469	CL
5/4/98	MgSO4	10.4752 mg SO4 / 5ML H_2SO_4 / 250ml DI	Fisher 96443	CL
5/8/98	90% Acetone	900ml / 100ml DI	Fisher 982028	CL
5/11/98	Diluted HCL	5.0ml / 1l	Fisher 967469	CL
5/20/98	Diluted HCL	10ml HCl / 1l H_2O	FISHER 967469	SP
5/20/98	90% Acetone	900 mL / 100 mL DI H_2O	Fisher 982028	SM
5/21/98	Diluted HCl	10mL / 1L DH_2O	Fisher 967469	SM
6/2/98	Diluted HCl	10mL/1L	Fisher 967469	CL
6/10/98	90% Acetone	900ml / 100mL DI H_2O	Fisher 982028	CL
6/17/98	Diluted HCL	5mL / 1 L DI H_2O	FISHER 967469	SP
6/23/98	90% Acetone	900ml / 100ml DH_2O	Fisher 982028	CL
6/24/98	Diluted HCl	10mL / 1L H_2O	FISHER 967469	SB
7/6/98	Trizma	1000mL DI / 12.1081 gm	Sigma 80H5618	SM
7/6/98	MFP	0.052g / 100ml DI	Sigma 38F0245	CMM
7/7/98	Diluted HCL	10mL HCL / 1L H_2O	FISHER 967469	SP
7/9/98	90% Acetone	1980 ml / 220 ml H_2O	Fisher 982028	CMM
7/20/98	Diluted HCL	5mL HCL / 1 L DI H_2O	FISHER 967469	SP
7/20/98	Diluted HCL	10mL HCL / 1L DI H_2O	FISHER 967469	SP
7/21/98	Trizma	6.408g / 500ml H_2O	Sigma 80H5618	CMM

TABLE 9.2
Standard, Source, Preparation, and Storage.

Instrument/Parameter	Standard Sources	How Received	Source Storage	Preparation from source	Lab Stock Storage	Preparation Frequency
Alpkem RFA Auto-Analyzer ..... Ammonia Nitrite Nitrate Phosphate Total Phosphorus Silica	Fisher Scientific, Inc. ..... NIST	Dry, ACS Reagent Grade powder ....Solids..... Dry Soil and Plant Standards	Room Temperature ..... Desiccator Room Temperature	Primary from source: 5 µmol/ml NH_4^+ 1 µmol/ml NO_2^- 10 µmol/ml NO_3^- 1 µmol/ml PO_4^{3-} 5 µmol/ml SiO_2 Mixed from primary (for dissolved nutrients): 1.56 µmol/ml NH_4^+ 1.25 µmol/ml NO_3^- 0.31 µmol/ml PO_4^{3-} Working from mixed standard Working from primary for NO_2^-, SiO_2, and Total Phosphorus (see attached SOPs for lab / field nutrient analysis and for Total Phosphorus)	Room Temperature with Chloroform Room Temperature with Chloroform Not Applicable	Quarterly Quarterly or as needed Daily
Shimadzu TOC-5000 Total Organic Carbon Analyzer	Fisher Scientific, Inc.	Dry, ACS Reagent Grade powder	Room Temperature	Primary from source: 10,000 mgC/l Working from primary: 0,5,10,20 mgC/l	Refrigerate Not Applicable	Quarterly Daily
ANTEK 7000 N Total Nitrogen Analyzer Total Nitrogen	Fisher Scientific, Inc.	Dry, ACS Reagent Grade powder	Room Temperature	Primary from source: 2 mg N/l	Room Temperature w/ chloroform	Quarterly or as needed

TABLE 9.2 Continued.
Standard, Source, Preparation and Storage

Instrument/Parameter	Standard Sources	How Received	Source Storage	Preparation from source	Lab Stock Storage	Preparation Frequency
Fluorometer Chlorophyll	Sigma, Inc.	Dry, ACS Reagent Grade powder	Dessicator Freezer	Primary from source: 5 mg/l chl a in acetone Working from primary: 8 standards: 0-0.5 mg/l	Freezer Not Applicable	Quarterly or as needed
Alkaline Phosphate Activity				Primary from source: 1 mmol/l MF Working from primary: 0,1,2.5,5,10 µmol/l MF	Freezer Not Applicable	Daily
Carlo Erba Total Carbon	Fisher Scientific, Inc. NITS	Dry ACS Reagent Grade	Room Temperature	Used Directly	Desiccator	Daily
Total Nitrogen		SRM	Desiccator Room Temperature	Used Directly	Desiccator	Daily
Turbidity	HF Scientific, Inc.	Sealed 0.02 NTU reference standard sent with Instrument (calibrated in each applicable range)	Room Temperature	Not Applicable	Not Applicable	Replace annually

TABLE 9.2 Continued.
Standard, Source, Preparation and Storage

Instrument/Parameter	Standard Sources	How Received	Source Storage	Preparation from source	Lab Stock Storage	Preparation Frequency
Orion pH Meter pH	Fisher Scientific, Inc.	pH 4.0, 7.0 and 10.0 solutions	Room Temperature	Not Applicable	Not Applicable	Replace on expiration
Orion Model 840 DO meter	Orion	Calibration Sleeve with Instrument	Room Temperature	Not Applicable	Not Applicable	Not Applicable
Analytical Balances	Troemner	Stainless Steel (Class S weights)	Room Temperature	Not Applicable	Not Applicable	Daily, Semiannual Service Calibration
Pipetman and Eppendorf Pipets	DI Water Weight checked	In-house	Room Temperature	Not Applicable	Not Applicable	Daily, factory calibrated as needed

TABLE 9.3
Field Instrument Calibration

Instrument	Calibration Type	No. of Standards	Type of Curve	Acceptance/ Rejection Criteria	Frequency
Orion S/C/T Meter					
Salinity/Conductivity	Continuing Check	3	Linear	Salinity or conductivity within 5% of standard value	Daily, prior to use, every 4 hours, and end of each use.
Temperature	Continuing Check	1	Linear	Temperature within 0.1 degrees of NIST thermometer value	Daily
Orion Dissolved Oxygen Meter	Initial	1	Linear	Slope must be within 0.7-1.2	Daily, prior to use, every 4 hours, and end of each use.
	Continuing	1	Linear	Result within +/-5% of Winkler titration	Annually
Orion pH Meter	Initial	2	Linear	Reading must be with 0.05 pH units.	Daily, prior to use.
	Continuing	1	Linear	Reading must be within 0.05 pH units.	Every 4 hours, and end of each use.
HF Scientific Turbidimeter	Initial	1	Linear	Reading within 0.01 NTU	Daily, prior to use, every 4 hours, and end of each use.
	Continuing	2	Linear	Reading within 0.01 NTU	Quarterly

TABLE 9.3
Field Instrument Calibration

Instrument	Calibration Type	No. of Standards	Type of Curve	Acceptance/ Rejection Criteria	Frequency
LI-COR Light Meter	Continuing Check	0	Log	Reading between 0.95-1.05 over a non-reflective surface in air.	Daily, prior to use, every 4 hours, and end of each use.
SEA-BIRD CTD	Continuing Check	1	Linear	Salinity or conductivity within 5% of standard value	Daily, prior to use, every 4 hours, and end of each use.
		1	Linear	Temperature within 0.1 degrees of NIST thermometer value	Daily

TABLE 9.4
Laboratory Instrument Calibration

Instrument/ Analysis	Calibration Type	No. of Standards	Type of Curve	Acceptance/ Rejection Criteria	Frequency
Alpkem Rapid Flow Analyzer NH_4^+ NO_2^- NO_3^- PO_4^{3-} Total P SiO_2	Initial + Final Continuing	5 1 Blank 1 Intermediate	Linear	R>.995 Value of zero 90-110% of exp. value	Daily, Prior to use . Every 20 samples Every 20 samples
Shimadzu Total Carbon Analyzer	Initial Continuing	4 1 Blank 1 High	Linear	R>.995 90% -110% of value	Daily, Prior to use . Every 20 samples Every 20 samples
ANTEK Elemental Analyzer Total Nitrogen	Initial Continuing	5 1 Blank 1 High	Linear	R > 0.99 Value of zero 90% -110% of value	Annually or upon placement of pyrolysis tube Daily, prior to use, every 20 samples, and end of run
Gilford and Shimadzu Spectro-fluorometers	Initial Continuing	5 1-Intermediate	Linear	R>.995 90% -110% of value.	Daily, prior to use. Every 20 samples.
Carlo Erba Total Carbon, Total Nitrogen	Initial Continuing	1 1 High	Linear	 90%-110% of value.	Daily, prior to use, every 20 samples, and end of run.

9.4.1.1. Salinity/Conductivity/Temperature

The Orion model 140 Salinity/Conductivity/Temperature meter, with a 014010 4-electrode probe, is factory calibrated and compensated for temperature. Salinity and/or conductance is checked daily with a solution of known salinity or conductance, while temperature is checked daily against an NIST thermometer. The S/C/T meter probe and the NIST thermometer is inserted into 25 ml of the salinity and/or conductance standard. A conductivity and/or salinity reading within 5% of the standard value, and a temperature within 0.1 degrees are considered acceptable. Values outside these acceptance criteria will require the unit to be factory calibrated.

9.4.1.2. pH

The pH meter/probe is calibrated before each field day. We use an automatic temperature compensation (ATC) probe to adjust for differences in temperature between standards and samples. Standard pH buffers (pH 7.00, cat. no. SB108-500; and 10.00, cat. no. SB116-500) are purchased from Fisher Scientific. The two-point calibration procedure is as follows:

1. Choose pH 0.01 mode.
2. Rinse probes (pH combination and ATC) in DIW. Blot dry. Rinse with ca. 2 ml of pH 7.00 buffer. Immerse probes in pH 7.00 buffer.
3. Press Cal button. The meter will display ".1." and the pH value of the buffer; the meter automatically recognizes the pH of the buffer solution. When pH stabilizes, press Enter. The display will freeze for 3 seconds, and then display ".2.".
4. Rinse probes in DIW. Blot dry. Rinse with ca. 2 ml of pH 10.01 buffer. Immerse probes in pH 10.00 buffer.
5. Wait for pH display to stabilize, and press Enter. Display now will say "PH" and be ready for sample measurement.
6. Rinse probe in DIW, place probe in pH 7.00 buffer, and check that pH meter reading is within 0.05 pH units.

The response of the pH meter is checked with the pH 7.00 buffer after 4 hours of use and at the end of each use. If the response is outside 0.05 pH units, the two-point calibration is repeated.

In case of low pH level samples, the pH 4.00 and pH 7.00 standards will be used in the calibration procedure.

9.4.1.3. Dissolved Oxygen

The probe of the Orion model 840 Dissolved Oxygen meter is continuously polarized when attached to the meter; if it has been disconnected for over 1 h it requires 50 min to repolarize. No readings or calibration should be attempted within 50 min of connecting the probe. Calibration is preformed at the beginning of every field day. A one point calibration is done, there is no zero current on the probe. The calibration procedure is as follows:

1. Saturate the sponge in the calibration sleeve with deionized water.
2. Switch the meter on and wait 20 min for equilibration.

3. Depress and hold the Mode Key Pad until the display cursor is at Cal.
4. Depress quickly and release the Mode Key Pad. The display will show three dashes (---) and the slope of the electrode/membrane system. If the slope is outside the range 0.7 - 1.2, the probe must be serviced.
5. Remove the calibration sleeve. The probe can now be used to make field determinations of dissolved oxygen concentration.

In addition, the response of the D.O. meter is checked against a Winkler titration on an annual basis.

9.4.1.4 Turbidity Meter Calibration

A 0.02 NTU reference standard, EPA approved and shipped with the instrument, is used to calibrate the instrument before each day's analyses. Additionally, higher turbidity standards, prepared from a 4000 NTU stock Formazin solution, are used to check the instrument calibration annually. The stock Formazin is purchased from HF Scientific, Inc., Ft. Meyers, FL.

Calibration is accomplished by inserting the 0.02 NTU standard cuvette into the instrument in the proper orientation and the Reference Adjust Knob on the instrument is turned until the readout displays 0.02. The standard cuvette must be clean and unscratched. The cuvette is wiped with lint-free wipers and inserted so that the index line on the cuvette matches the instrument's index line.

9.4.1.5 Light Meter Calibration

The calibration of the LI-COR instrument is checked on a daily basis. The instrument is held in an upright position in the air over a non-reflective surface such as still water, pavement, or grass (not a white boat or concrete dock). The instrument calibration reading is recorded and should be between 0.95 to 1.05.

9.4.1.6 CTD Calibration

Salinity/conductance is checked daily with a solution of known salinity/conductance, while temperature is checked daily against a NIST thermometer. A conductivity reading within 5% of the standard value, and a temperature within 0.1 degrees are considered acceptable. Values outside these acceptance criteria will require the unit to be factory calibrated. The unit is also factory calibrated on an annual basis.

9.4.2 Laboratory Instruments

9.4.2.1 Alpkem Rapid Flow Autoanalyzer

The autoanalyzer is calibrated daily, using a five-point calibration standards of ammonium, nitrate, nitrite, and phosphate. Total phosphorus and silica are each analyzed separately on the autoanalyzer, also using a five-point calibration. Standards are prepared in the matrix to be analyzed (i.e. freshwater or seawater). The five-point calibration is checked at the beginning of each run. A linear calibration with an R square of greater than 0.995 is considered acceptable.

Blanks are inserted after every 10 samples to monitor and correct for baseline drift. A log book is kept to monitor the calibration curve parameters. The instrument is recalibrated if accuracy is not within 90 and 110 percent. If continued attempts at calibration do not meet the accuracy requirements, then the instrument is cleaned and overhauled.

9.4.2.2 Total Organic Carbon Analyzer

A four-point standard curve consisting of 0, 5, 10, and 20 mgC/l standards is run prior to every run. A linear calibration with an R square of greater than 0.995 is considered acceptable. A log book is kept to monitor the instrument calibration. The instrument is recalibrated if accuracy is not within 90 and 110 percent. If continued attempts at calibration do not meet the accuracy requirements, then the instrument is cleaned and overhauled.

9.4.2.3 Total Nitrogen Analyzer

A five-point calibration of the ANTEK Total Nitrogen Analyzer is conducted annually (see Appendix A), or upon replacement of the pyrolysis tube. A two-point calibration is prepared daily prior to every run using a 2.0 mgN/l standard. Due to the nature of the Total Nitrogen Analyzer, zero total nitrogen has a signal of 0. Intra-run drift in the calibration curve is monitored by insertion of additional 2.0 mgN/l standards after every 10 samples and at the end of the run.

9.4.2.4 Fluorometer

Calibration of the fluorometer for chlorophyll determination is done using solutions of known chlorophyll content dissolved in acetone. Chlorophyll standard is made from purified chlorophyll-a obtained from Sigma Chemical Co. The concentration of the standard solutions are measured spectrophotometrically, and a series of chlorophyll standards are prepared that bracket the range from 0 to 0.5 mg/l. Fluorescence of these standards is determined, and a standard curve is generated.

A five-point standard curve of 3-*o*-methylfluorescein is used to calibrate the fluorometer for alkaline phosphatase activity (APA), prior to and at then end of every run. An R square of greater than 0.995 is considered acceptable. Values outside this range require recalibration.

9.4.2.5 Carlo Erba

The Carlo Erba is calibrated with one standard of known total carbon and total nitrogen concentrations. The standard is run prior to every run, after every 10 samples, and at the end of a run. An accuracy between 90 a 110 % is considered acceptable.

9.4.2.6 Balances

The balances are calibrated daily using 10 mg, 1 g, and 100 g weights and these calibration checks are recorded in a logbook (Figure 9.2).

9.4.2.7 Pipettes

Pipettes are checked following the procedure in Figure 9.3 and the results of these checks are recorded in a logbook (Figure 9.4). If the pipette fails to achieve a weight / volume value within the specified range, it is recalibrated with the appropriate tools. Any needed recalibration is recorded in the logbook.

Figure 9.2 Balance Calibration Log

	Mettler AE 260			Denver XE-4000			
Date	10 mg	1g	100g	10 mg	1g	100g	INT
5-3-98	.0108	1.0024	100.008	.011	1.002	100.01	PL
5/5/98	0.0108	1.0025	100.008	0.014	1.005	100.01	[illegible]
5/6/98	0.0108	1.0025	100.008	.011	1.003	100.01	PL
5/7/98	0.0107	1.0025	100.008	0.012	1.005	100.01	[illegible]
5/8/98	0.0107	1.0024	100.008	0.012	1.005	100.00	[illegible]
5/12/98	0.0107	1.0024	100.008	0.012	1.005	100.00	SB
5/13/98	0.0107	1.0025	100.008	0.010	1.003	100.01	JL
5/14/98	0.0109	1.0025	100.008	0.012	1.003	100.01	JL
5/15/98	0.0110	1.0027	100.008	0.010	1.001	100.01	JL
5/18/98	0.0108	1.0025	100.008	0.012	1.005	100.01	JL
5/19/98	0.0107	1.0023	100.008	0.010	1.006	100.01	JL
5/20/98	0.0108	1.0025	100.008	0.011	1.004	100.01	JL
5/21/98	0.0107	1.0025	100.009	0.013	1.005	100.01	JL
5/22/98	0.0108	1.0026	100.009	0.012	1.003	100.01	JL
5/26/98	0.0108	1.0024	100.009	0.012	1.002	100.01	JL
5/27/98	0.0107	1.0024	100.009	0.008	1.004	100.01	JL
5/28/98	0.0108	1.0025	100.009	0.011	1.004	100.01	JL
5/29/98	0.0112	1.0023	100.009	0.013	1.004	100.01	JL
6/1/98	0.0107	1.0024	100.009	0.010	1.003	100.01	JL
6/2/98	0.0108	1.0025	100.009	0.011	1.003	100.01	DD
6/3/98	0.0108	1.0025	100.009	0.009	1.001	100.01	JL
6/4/98	0.0107	1.0024	100.009	0.012	1.004	100.01	JL
6/5/98	0.0106	1.0023	100.009	0.011	1.004	100.01	JL
6/8/98	0.0107	1.0025	100.009	0.011	1.005	100.01	JL
6/9/98	0.0106	1.0023	100.009	0.012	1.003	100.01	JL
6/10/98	0.0108	1.0025	100.009	0.008	1.005	100.01	JL
6/11/98	0.0107	1.0024	100.009	0.012	1.004	100.01	JL
6/12/98	0.0108	1.0025	100.009	0.012	1.002	100.01	JL
6/15/98	0.0108	1.0025	100.009	0.011	1.003	100.01	JL

Figure 9.3 Pipette Calibration Log

Date	Model	ID	Weight	OK	Initials
10-27-98	P-20	316 Hg	.0201	RECALIBRATED ✓	PL
10/29/98	P 20	321	0.0200	RECALIBRATED	BS
10/29/98	P10M1	327	9.9807	✓	MN
10/29/98	P1000	327	0.9924	✓	MN
10/29/98	P200	327	0.2004	✓	MN
10/29/98	P1000	321B	~~1.000~~ 1.003	RECALIBRATED	BS
10/30/98	P100	321	0,0989	✓	SP
10/30/98	P1000	321B	0,9929	Recalibrated	VP
10/30/98	P5000	321.B	5.0188	✓	PL
10/31/98	P5000	321A	4.9980	✓	PL
10/31/98	P100	321	.0999	✓	PL
10/31/98	P20	321	.0200	✓	PL
10/31/98	P10	327	10.0002	✓	AL
10/31/98	P200	327	0.1996	✓	AL
10/31/98	P1000	327	0.9986	✓	AL
10/31/98	P5000	327	5.0014	✓	AL
11-02-98	P 5000	321A	5.0023	✓	PL
11-02-98	P200	321B	.2001	✓	PL
11-05-98	P 5000	321A	5.0161	✓	PL
11-03-98	P100	321	.1015	✓	PL
11-03-98	P 20	321	.0200	✓	PL
11/03/98	P10	327	10.0652	✓	AL
11/03/98	P200	327	0.1994	✓	AL
11/03/98	P1000	327	0.9924	✓	AL
11/03/98	P5000	327	5.0043	✓	AL

Figure 9.4 Daily Pipette Calibration Instructions

DAILY PIPETTE CALIBRATION INSTRUCTIONS

1. Record the date, pipette model (i.e. P-5000, P-100, etc.), and pipette ID (i.e. 321A, 316 prep, etc.).

2. Set the pipette on the highest setting (e.g. 200 µl for a P-200). Always dial down to the setting (dial up a little past the setting, then dial down).

3. Dispense that volume of DIW into a tared weigh cup.

4. If the weight is within the acceptable range (see chart below or on the balance), then record the weight, put a check the OK column, and initial.

5. If the weight is not within the acceptable range, try at least two more times. If the weight is still not acceptable, record the last weight obtained, put an X in the OK column, initial, and bring the bad pipette to the QA Officer.

Accuracy range:

P-5000	4.9700 - 5.0300
P-1000	0.9920 - 1.0080
P-200	0.1984 - 0.2016
P-100	0.0992 - 0.1008
P-20	0.0199 - 0.0201
P-10	0.0099 - 0.0101

10.0 Preventive Maintenance

10.1 Routine Maintenance

Preventive maintenance is an essential part of a properly functioning laboratory. For field equipment, general maintenance includes cleaning, proper storage, check batteries and keeping the instruments fully charged. In addition, all probes are checked and replaced as necessary. The laboratory equipment receives thorough cleaning after every use. A more detailed summary of the maintenance procedures conducted on each piece of laboratory equipment is presented in Table 10.1, while field equipment is presented in Table 10.2.

10.2 Maintenance Documentation

Log books are kept on each piece of equipment. Instrument response to calibration standards, the number of samples run, and the hours of instrument use are recorded in each log book. In addition, all maintenance activities for each instrument are recorded in the log book. A record of service performed by the manufacturer or other service contractor is kept in the instrument files.

10.3 Contingency Plans

SERP maintains a stock of spare parts for all analytical instruments. Instruments which can not be fixed by SERP personnel are sent to the manufacturer or other service contractor. If equipment failure occurs, SERP will either operate backup equipment at its laboratory, or it has access to backup equipment in other laboratories at FIU. In any event, sample holding time will not be jeopardized.

TABLE 10.1 Laboratory Equipment Preventive Maintenance

Instrument	Activity	Frequency
Alpkem RFA	Clean and inspect tubing and fittings Clean Platens Wash manifold/flow cell Check cadmium column Inspect filters Change tubing Recondition pump rollers Service Maintenance	Daily Daily Daily Daily Every 200 hours Every 200 hours Every 200 hours Semiannually
Shimadzu TOC Analyzer	Check IC reagent level Check DIW level Check gases Replace tubing Replace needles Change columns	Daily Daily Daily As needed As needed Every 2000 samples
Carlo ERBA	Check gas flow Monitor Voltage Reduce copper column Repack water trap Repack oxidation column	Daily Daily Every 150 samples Every 150 samples Every 350 samples
ANTEK 7000N Nitrogen Analyzer	Replace autosampler septa Replace column septa Monitor vacuum pressure (25 in Hg) Change combustion column	Every 80 Samples Every 40 Samples Daily As Needed
Fluorometer	Clean and inspect sample chamber	Daily
Analytical Balances	Clean weighing compartment Clean interior/exterior Check calibration Factory service calibration	Daily Monthly Daily Semiannually
Ovens , Refrigerators, and Freezers	Check temperature Calibrate with NIST thermometer	Daily Annually

TABLE 10.2 Field Equipment Preventive Maintenance

Instrument	**Activity**	**Frequency**
pH meter	Check batteries - recharge Check liquid in probe Replace probes Rinse with analyte-free water	Daily Daily Every 6 to 9 months Before and after each use
Dissolved oxygen meter and S/C/T meter	Check batteries - recharge Check probes	Daily Daily
Turbidimeter	Check battery - recharge Check light source Check cuvettes are scratch-free	Daily Daily Daily
LI-COR Light Meter	Check battery - recharge Check calibration Factory service calibration	Daily Daily Annually
SEA-BIRD CTD	Check battery - recharge Check Tygon tubing is secure and filled with DIW Check all probes and connections Check calibration Factory service calibration	Daily Daily Daily Daily Annually

11.0 Quality Control Checks and Routines to Assess Precision, Accuracy and Calculation of MDLs

SERP uses both field and laboratory QC check samples. Each of these QC check samples are included on Table 11.1.

11.1 Field QC Checks

SERP's field quality control includes the collection of a duplicate sample for each parameter analyzed at every sampling location. In addition, according to FDEP-QA-001/90, one equipment blank is prepared for every 20 samples. This blank is prepared in the field prior to sampling by pouring or rinsing using analyte-free water on each piece of precleaned field sampling equipment. Equipment blanks for surface water samples are collected by pouring DIW into a syringe then into a sample bottle. For analyses requiring filtration, the equipment blank is prepared by running DIW through the filter. For pore water samples, the equipment blank is prepared by running DIW through the peristaltic pump then into sample bottles. For soil and sediment samples, the equipment blank is prepared by pouring DIW over the sampling equipment and into the appropriate sample bottles. All duplicate and blank samples are placed in appropriate bottles and preserved according to each analysis. The collection of blank samples are recorded in the field notebook. For field equipment cleaned in the field, an additional equipment blank is prepared following the field cleaning procedures at a frequency of one per sampling event or one every 20 samples, whichever is greater. The time and number of all equipment blanks are recorded in the field notebook.

Field instrument checks are completed prior to each sampling event, once every four hours of operation and at the end of the field sampling event. The results of the field QC checks are recorded on the Field Instrument Calibration Sheet (Figure 7.2). Field equipment not functioning properly are not used to collect data until they are brought back to the laboratory for maintenance. Duplicate field equipment and probes are kept on hand in the laboratory if needed.

If problems arise with the Dissolved Oxygen (D.O.) meter, and D.O. is an important parameter of the specific project, then the field sampling should be discontinued until the D.O. meter is brought back to the laboratory for maintenance. If problems arise with the S/C/T meter, samples can be collected in clean 125 ml bottles and brought back to the laboratory for salinity/conductivity determination within 24 hours. Temperature can be determined in the field with the D.O. meter. Since the D.O. meter needs to be manually adjusted for salinity, take all D.O. measurements at a salinity of 0 ppt, and record this in the field notebook. D.O. measurements are later adjusted to the sample salinity determined in the laboratory.

Table 11.1
Quality Control Checks

Type	Description	No. of Samples per event	Frequency (all parameter groups)
Field			
Equipment Blank (non-field cleaned equipment)	Fill or rinse all pre-cleaned sampling equipment (tubing, syringes, filter holders, etc.) with analyte-free water, fill appropriate sample containers and preserve according to each analysis.	< 10 > 10	1 prepared on-site at the beginning of the sampling event 1 prepared on-site at the beginning of the sampling event, and after every 20 samples or 5% whatever is greater
Equipment Blank (field cleaned equipment)	If equipment is cleaned on-site, then prepare additional equipment blank sample by filling or rinsing the field-cleaned equipment with analyte-free water, filling the appropriate sample containers and preserve according to each analysis.	< 10 > 10	1 at the end of the sampling event 1 after every 20 samples or 5% whatever is greater
Field Duplicate	A duplicate sample collected and analyzed for the same parameters as the original sample.	1 or more	Every sample is collected in duplicate
Field Measurements QC Check Standards pH meter	 Record the results of calibration check standards for all field measurement equipment. Record two or more pH readings in field notebook until sequential values are within 0.02 pH units.	 1 or more 1 or more	 Beginning of each sampling event, once every four hours, and again at end of the sampling day. Every sample.

TABLE 11.1 Continued.

Quality Control Checks

Type	Description	No. of Samples per Event	Frequency (All parameters)
Laboratory			
Method Reagent blank	Analyte-free water: DIW for freshwater samples, and Gulf Stream for seawater samples	1 or more samples	1 at beginning of a run, after every 20 samples, and at the end.
Replicate Samples	Re-analysis of a sample	1 or more samples	Every sample is analyzed in replicate.
Matrix Spikes	One sample from a set (not blanks) is split in two, and one of the duplicates is spiked with a known concentration prior to sample preparation.	1 or more samples	1 sample in a set or at a frequency of 5%, whichever is greater.
Continuing Calibration Standards	One intermediate standard and one high standard.	1 or more samples	Analyzed at the beginning of each run, and at a frequency of 5%, thereafter.
Quality Control Check Standards	Standards from an independent source that are certified and traceable (i.e. NIST standards). Can be interchanged as one of the continuing calibration check standard.	1 or more samples	Analyzed at the beginning of each run to check the initial calibration of the standard curve.
Quality Control Check Samples	Samples of known analytical concentration that are submitted blind to the analyst. These samples are either prepared in house or obtained from an independent source	1 or more samples	Analyzed in duplicate quarterly.

11.2 Laboratory QC Checks

SERP's standard laboratory QC checks includes blanks, replicates, and QC standards and QC check samples. Method reagent blanks consisting of analyte-free water (DIW for freshwater samples, and Sargasso Seawater or Gulf Stream water for seawater samples) are prepared exactly like a sample and run prior to each instrument calibration, and after every 20 samples. For the autoanalyzer, method reagent blanks are run between every sample.

As standard practice SERP collects all field samples in duplicate (see Section 11.1). In addition, SERP analyzes all samples in replicate, thereby, producing four data points for one sample location. This QA protocol allows for easy identification of unusual sample results, and provides a constant check of analytical precision and accuracy.

Continuing calibration standards (CCS) consisting of one intermediate standard and one high standard are run at the beginning of each run and at a frequency of 5% thereafter. The first CCS should be 90-110 % of expected value and the following ones should fall in a 90-110 % range of the original CCS.

Quality control check standards are certified standards from an independent source that are analyzed at the beginning of a run to check the calibration of the standard curve. The % Recovery (%R) related to the expected Quality control check standard concentration is calculated and recorded. The control limits for the %R are +/- three standard deviations of the historical average , with warning limits set at +/- two standard deviation of the historical average. New limits (both control and warning) based on historical data are calculated on a quarterly basis . These standards may be run in the place of one of the continuing calibration standards.

Matrix spikes samples are prepared by splitting a sample from the set (not a blank) into two duplicates and spiking one of the duplicates with a known concentration. The concentration from the unspiked duplicate is subtracted from the spiked result and the percent recovery by comparing the remainder to the known spike concentration. Quality control check samples are prepared in-house or from an NIST certifying source. These samples are submitted blind to the analyst on a quarterly basis to check instrument and user performance. If the blind QC check sample result is not acceptable, the results will be reported in the QA report to FDEP.

11.3 Routine Method Used to Assess Precision and Accuracy

Precision and accuracy of each analytical parameter determined in the laboratory is determined on a daily basis. Precision is defined as the agreement or closeness of two or more results. As stated above, SERP collects all field samples in duplicate, and performs a duplicate analysis on each sample, thereby, producing four results for one sample location. SERP determines the mean (X) and standard deviation (SD) of these four data points and estimates precision in terms of percent relative standard deviation (% RSD) using the following equation:

$$\% \text{ RSD} = \frac{SD}{X} * 100$$

The control limits for precision are set at +/- two standard deviations of the mean.

The Relative Percent Difference (RPD) is another parameter used to monitor the precision of our analytical results and it is calculated for Matrix Spike duplicates and/or sample duplicates. The acceptance criteria is usually RPD <= 20 %.

Accuracy is defined as the agreement between the analytical results and the known concentration. Accuracy is determined by running matrix spikes (MS) and/or standard reference materials (SRM) and is determined as percent recovery (% R) according to the following equations:

$$\%\ R = \frac{Cs - Cu}{MS} * 100.$$
$$S$$

Where:

Cs = concentration of spiked sample
Cu = concentration in unspiked sample
S = expected concentration of spike in sample
%R = percent recovery

$$\%\ R = \frac{\text{Sample Concentration}}{\text{SRM}} * 100$$
$$\text{True Value}$$

The control limits for accuracy are +/- three standard deviations of the historical percent recovery average , with warning limits set at +/- two standard deviation.

The results obtained for each quality control check are compared to their acceptable limits for precision and accuracy on a daily basis. New limits (both control and warning) based on historical data are calculated on a quarterly basis .

11.4 Method Detection Limits

Method detection limits (MDLs) have been determined according to the EPA procedure described in 40 CFR Part 136, Appendix B, revision 1.11. Specifically, seven or more replicate samples containing an analyte at a known low concentration are analyzed according to the appropriate analytical procedure for that analyte. A standard deviation for the replicates is determined and the MDL is computed as 3 times the standard deviation. The practical quantitation limit (PQL) is defined as 12 times the standard deviation. MDLs and PQLs are verified/updated once a year.

12.0 Data Reduction, Validation and Reporting

12.1 Data Reduction

Data reduction is not necessary for field data, as field measurements are read directly from the field instruments in their appropriate reportable units. The pH meter, SCT meter, and dissolved oxygen meter are automatically compensated for temperature. Salinity read from the SCT meter by the field technician and then input to the D.O. meter by the field technician, where the D.O. is automatically corrected for both salinity and temperature. Each technician is responsible for data entry of field data from the field notebook into Quattro Pro (Lotus 123 compatible) spreadsheets.

All data reduction is performed according to the protocols specified by the analytical methods listed in Section 5. Laboratory data reduction is mainly completed by computers associated with the laboratory instruments. Calculation of standard curves and sample results in comparison to the standard curves is determined by the analytical instrument computer. Analytical runs are recorded in the instrument computer under the project name and sampling event number. If a sample is outside of the standard curve, the analytical instrument automatically performs the required dilution, and calculates the sample result based on the dilution. If a sample is suspected of being far beyond the standard curve, the laboratory analyst may perform the dilution themselves. In this case, the laboratory analyst records the dilution in the instrument log book, as well as on the instrument printout. All instrument printouts are identified by their project name and sampling event number. In addition, any analytical conditions (i.e. voltage setting, wavelength, flow rate, injection volume, etc.) that deviate from those listed in the SOP are recorded on the instrument printout and logbook in detail.

Replicate sample results are further reduced to provide one data point per sampling location. SERP collects duplicate samples for all analyses at every sampling location. In addition, SERP completes a duplicate or triplicate analysis of each duplicate sample, producing at least four replicate results for each sampling location. Each of the four data points are input to a Quattro Pro or Excel spreadsheet by the analyst and the mean and standard deviation of each sample result is calculated. Any replicate result that is two standard deviation away from the mean (+ or -) is removed from the replicate data set, by crossing out the value on the instrument printout, as well as from the input spreadsheet.

12.2 Data Validation

The Chief Chemist and analytical technicians are responsible for the collection, custody, storage, and analysis of all of the samples. It is their responsibility that the samples are analyzed within the appropriate holding times. They are also responsible for the proper maintenance of all equipment and cleaning of laboratory glassware. They provide the first check on field and laboratory instrument calibration, method blanks, and equipment blank results, and ensure that all method specifications have been met. If problems arise during an analysis, such as failure of proper equipment calibration, or unusual sample results, it is their responsibility to verbally notify the laboratory director as soon as possible.

The QA Officer is responsible for a second check of instrument calibration (both laboratory and field instruments) by comparing the present instrument responses to historical values. In addition, the QA officer checks the results of method blanks, equipment blanks, and sample replicates and determines

that the instrument precision and accuracy is within the QA objectives listed in Section 5. Obvious anomalous results are subject to re-analysis.

Dr. Jones, the director, is responsible for the final review of all data and documents that are submitted to the client (FDEP; SFWMD). Due to his extensive experience in analytical chemistry, Dr. Jones can apply both objective and subjective techniques to data review. From his knowledge of nutrient chemistry, Dr. Jones can interpret the data in its environmental context. In addition, through his collection of historical data in South Florida, Dr. Jones can identify potential outliers in a data set.

12.3 Data Reporting

Once the instrument calibration and sample results have been validated, they are entered into input data files. The laboratory technicians are responsible for providing a first check of data entry. Data reports are prepared in Quattro Pro or Excel spreadsheets.

When using Quattro Pro, a standard input file is used for data entry of all field and raw laboratory data (secondary standards, sample results, and sample replicates). A second spreadsheet file performs calculations for data reduction, such as determination of replicate means and standard deviations, and conversion of sample results into reportable units of interest (i.e. moles/l, mg/l, ppm, etc.). A third spreadsheet file is then produced as output from the second spreadsheet and is the final data report in a client (FDEP; SFWMD) requested format.

The Excel workbooks consist of three sheets: one for the data input, a second one where raw data is filtered eliminating those individual values that are outside the mean +/- 2 SD acceptance range, and a third one where the final printout steps are performed. The second sheet contains two Visual Basic macros that perform all the calculations, unit conversions and format adjustments needed for the data report, producing the final report printouts that are sent to the customer.

For data reports issued to the client for DEP-related work, or for reports issued directly to DEP, the following information will be included:

a. Laboratory name, address, and phone number
b. Client name and/or site name
c. CompQAP number
d. Client or field identification number
e. Sample identification number
f. Method number of each analysis
g. Analytical result with applicable data qualifiers
h. Date of sample preparation
i. Time of sample preparation if holding time is in hours
j. Date of sample analysis
k. Date and time of sample collection
l. Identification of all laboratories providing analytical results, including their CompQAP number

The QA officer provides a second check of the input data, spreadsheet calculations, and output file

formats. Once all of the data has been validated, the QA officer will provide a written statement of validation along with the data report. An example of a final data report form is included as Figure 12.1.

12.4 Data Storage

Data input files and final report files are stored on hard drive and write-protected floppy disks using names that readily identify a sampling event. Files labeled by sampling event are stored in a locked file cabinet with limited access by SERP employees only. These files contain hard copies of the file input and output as well as all raw laboratory data sheets and field notebook sheets. Raw laboratory output data sheets are identified with a date, analysis, analyst initials, and sampling event number. SERP plans to maintain all records indefinitely, but will at minimum comply with the Chapter 62-160 F.A.C. requirement of 3 years.

Figure 12.1
Final Data Report

Southeast Environmental Research Program
Florida International University OE 148
Miami, Florida 33199
Phone: (305) 348-3095

Client Name:_____
Site Name:_____
CompQAP #:_____
Project Code:_____

Sample ID	Field No.	Station Code	FQC Code	Sampling Date	Sampling Time	Sampling Depth (m)	Parameter/ SOP #	Storet Code	Method Name	Sample Prep Date	Sample Prep Time	Sample Analysis Date	Sample Analysis Time	PQL	MDL	Result	Unit	Remark

Other Laboratories providing analytical results:

Lab :_____ CompQAP # :_____ Analyses:_____

Lab :_____ CompQAP # :_____ Analyses:_____

Lab :_____ CompQAP # :_____ Analyses:_____

13.0 Corrective Action

Corrective action is taken whenever the quality assurance objectives have not been met. A summary of the corrective actions for the laboratory and for the field are included in Tables 13.2 and 13.3, respectively.

The analyst, either the Chief Chemist or the technicians, are responsible for providing a first check for compliance, and initiating corrective action procedures as described in Table 13.1. The QA officer is responsible for a second check for compliance, and initiating corrective action as appropriate. If problems continue, then the analyst and/or QA officer will notify the laboratory director immediately, who may initiate further steps in solving the problem.

Any corrective action taken will be documented in the instrument log books, sample reanalysis sheets, and/or sample checklist within the project-specific files.

FDEP recommended corrective action will be initiated as a result of systems or performance audits, split samples, or data validation review.

TABLE 13.1
Corrective Actions for the Laboratory

QC Activity	Acceptance Criteria	Recommended Corrective Action
Initial Instrument Blank	Instrument response <MDL	Prepare new blank, if same response determine cause of contamination: reagents environment, equipment failure; notify Dr. Jones and QA Officer
Initial Calibration Standards	Linear response with R>0.995	Reanalyze standards, if same response, reoptimize instrument, if same response, prepare new standards, notify Dr. Jones and QA Officer
QC Check Continuing Calibration Standards	Historical average +/- 3 SD 90-110 % from initial calibration	Reanalyze check standard, if same response, prepare new check standard, if same response, prepare new primary and calibration standards, notify Dr. Jones and QA Officer.
Matrix Spikes	Historical average +/- 3 SD	Reanalyze matrix spike, if same response, prepare and run new matrix spike, is same response, notify Dr. Jones and QA Officer.
Replicate Sample	RPD < 20 %	Determine cause: baseline drift, carryover, etc. Reanalyze all samples between duplicates, notify Dr. Jones and QA Officer.
Duplicate Sample	RPD < 20 %	Reanalyze duplicates, reanalyze all samples between duplicates; notify Dr. Jones and QA Officer

TABLE 13.2
Corrective Actions for the Field

QC Activity	Acceptance Criteria	Recommended Corrective Action
Initial Calibration Standards	Value within +/- 5% of expected value	Reanalyze standards, if same response, optimize instrument, if same response, use new standards; notify Dr. Jones and QA Officer.
QC Check Standards	Value within +/- 3 standard deviations of the historical value	Reanalyze QC check standard, if same response, prepare new QC check standard, if same response, recalibrate; notify Dr. Jones and QA Officer.
Equipment/Trip Blank	Value <MDL	Reanalyze blanks: if same response, check recorded cleaning procedures and mark sample trip results for affected and related parameters questionable or invalidate data, as required; notify Dr. Jones and QA Officer.
Duplicate Samples	RPD < 20 %	Reanalyze duplicates: if same response, mark sample trip results for affected and related parameters questionable or invalidate data as required. If reanalysis show Field Collection to be acceptable, reanalyze all samples analyzed with the Field samples the first time. Notify Dr. Jones and QA Officer.

14.0 Performance and System Audits

Dr. Jones supervises all aspects of field and laboratory activities. He requires the laboratory and all instrumentation to be clean and working at optimum conditions. He is knowledgeable on the inner-workings of each instrument and checks on their performance as well as on the performance of the laboratory personnel continually.

14.1 Field Audits

An internal system audit is conducted on an annual basis by the QA officer. During these audits, the QA officer will review and evaluate the various components of the measurement system to determine their proper selection and use. Specifically, the auditor will review sampling technique, field instrument calibration, and field notebook documentation. The checklist included as Figure 14.1 will be used during the audit, and any discrepancies or deviations will be noted in the checklist and corrected immediately. At the end of the audit, the QA officer will date and sign the checklist stating that the audit was completed, and a copy of the checklist will be put in the project-specific files.

14.2 Laboratory Audits

Internal laboratory system audits are conducted on a semiannual basis by the QA officer. This audit is conducted with the use of the checklist included as Figure 14.2. In addition to these audits, instrument performance is checked continually by the analyst with analysis of standard curves, sample replicates, method blanks, and equipment blanks. The QA officer checks the instrument log books on a monthly basis to check that instruments are running within their appropriate QA objectives. Many of the analyses are performed and checked within 24 hours to seven days of sample collection, allowing for any deficiencies to be corrected and samples re-analyzed if needed. Documentation associated with each audit including the checklist, calculation checks of standard curves, sample replicates, equipment blanks, spikes, and QA check samples and standards are kept in the QA officer notebook.

Laboratory performance audits are conducted on a quarterly basis. The performance audit consists of at least two of the following samples:

- blind samples prepared by the QA Officer
- split samples with another laboratory
- QC samples from an independent certifying source (NIST)
- blind spike samples

Currently, SERP is not involved in a regular external audit program; however, we are available to receive on-site audits by FDEP at any time.

Figure 14.1 Field Audit Checklist

Field Audit Checklist

Auditor: _____ Date of Audit: _____

Y	N	Sample Collection	Comments
		* Sampling equipment & bottles rinsed 3 times before sample collected.	
		* Samples collected near the bow of the boat away from the engine.	
		* Samples collected for dissolved constituents are filtered.	
		* Filter is sparged with at least 30 ml of air prior to removal from filter holder.	
		*Filters are placed in microcentrifuge tube and acetone is added to the top line of the tube.	
		* Microcentrifuge tubes are stored in the dark in a cooler with ice.	
		* Dissolved nutrient bottles kept in a cooler with ice.	
		* Total nutrient bottles stored in the dark in a cooler without ice.	
		* The appropriate number of QC samples are collected at the appropriate times.	
		* Samples are collected at the correct project locations.	
Y	**N**	**Field Notebook**	**Comments**
		The following are recorded in the field notebook:	
		* Names of the field crew	
		*Weather conditions	
		* Time of sample collection	
		* Time of QC sample collection	
		*Temperature, Salinity, D.O.	
		* Volume of water filtered	
		* Date	
		* Station names	

Figure 14.1 Field Audit Checklist (Continued)

Field Audit Checklist (cont.)

Auditor: _____ Date of Audit: _____

Y	N	Field Instruments	Comments
		* Meter number and probe number recorded on the field instrument sheet.	
		* The D.O. meter is turned on at least 50 minutes prior to first reading.	
		* Slope of the D.O. meter is checked with the sleave on and the results are recorded.	
		* Salinity/Conductivity meter checked against Sal/Cond. Standard with results recorded.	
		* Instrument calibration is checked at the beginning of the day, 4 hours later, and at the end of the day.	
		* Spare instruments are available in the field.	
		* D.O. meter repair kit is available in the field.	
		* Temperature checked against NIST thermometer.	
Y	**N**	**Other**	**Comments**
		* Main office (and NPS Dispatch) is notified before and after sampling trip by phone or radio.	

Figure 14.2 Laboratory Audit Checklist

Laboratory Audit Checklist

Auditor: _____ Date of Audit: _____

Y	N	Instrument Response	Comments
		* Instrument notebook is up to date.	
		* High standard within 10 % of expected value.	
		* Calibration curve correlation coefficient better than 0.995	
		* Instrument blank is less than MDL.	
		* QC check standard within control limits.	
		* Matrix Spike samples within control limits.	
		* Replicate samples show RPD < 20%	
		* Duplicate samples show RPD < 20%	
		* Instrument maintenance up to date.	
		* Equipment Blank samples less than MDL.	
Y	**N**	**Sample Tracking**	**Comments**
		* Sample Checklist is filled out correctly.	
		* Samples are kept in proper storage	
		* Samples are analyzed within holding times.	
		* Samples are not discarded until QA checked.	
Y	**N**	**Labware**	**Comments**
		* Reagents are stored appropriately.	
		* Waste is disposed properly.	
		* Glassware and bottles are cleaned appropriately.	
		* Check the age of sample bottles.	
		* Standards are properly labeled and stored appropriately.	
		* Check dates of standards.	

Figure 14.2 Laboratory Audit Checklist (Continued)

Laboratory Audit Checklist (cont.)
Auditor: _____ Date of Audit: _____

Y	N	Data Management	Comments
		* Project Files up to date.	
		* Data input up to date and correct.	
		* Calculations performed correctly.	
		* Output files in correct format.	
		* Data values within 2 standard deviations of historical mean.	
		* Data reports up to date.	

15.0 Quality Assurance Reports

SERP will submit quality assurance reports for all Quality Assurance Project Plans at a frequency according to Table V of Appendix D of the QA Manual. The Quality Assurance Officer is responsible for the preparation of these reports. In general, if no audits were performed and no significant QA/QC problems have been identified, then SERP will prepare a brief letter stating these facts in lieu of a detailed quality assurance report.

A detailed QA report will be prepared when:

1. Activities were conducted in a manner other than those described by the CompQap or QAPP.
2. Preservation or holding requirements were not met.
3. Quality control checks were unacceptable.
4. Precision, accuracy, or MDL objectives were not met.
5. Corrective action was taken.
6. Internal or external audits were conducted and discrepancies were noted.

According to FDEP guidelines, these QA reports will include the following:

1. Title Page including the time period of the report, the QA Project Plan Title and Plan number, the laboratory name, address and phone number, and the preparer's name and signature.
2. Table of contents if the report is over 10 pages long.
3. The results of performance or system audits to include, date of audit, system tested, name of auditor, parameters analyzed, results of tests, deficiencies or failures, and an explanation of the problem and the corrective action taken.
4. Significant QA/QC problems.
5. Corrective actions taken.

APPENDIX A

Method Validation for Micromolar Concentrations of Total Nitrogen in Natural Waters

Limited Use Method Validation

Prepared by and for:

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25 April 1994
Date

Rene' M. Price
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SERP Quality Assurance Officer

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Date

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FDEP QA Officer

1/5/95
Date

1.0 Scope and Application

This method covers the determination of total nitrogen in fresh and saline surface waters. The method is capable of measuring total nitrogen at concentrations between 2.1 µmol/l (0.03 mg/l) to 250 µmol/l (3,500 µg/l) at an precision of 5% relative standard deviation (%RDS) or better, and an accuracy between 95 and 105%. The method is also applicable for analysis of dissolved nitrogen of filtered water samples.

2.0 Summary of Method

The procedure is a modification of the classical Dumas (1831) method of determining nitrogen by combustion technique with the addition of chemiluminescence. The method involves converting all forms of nitrogen into nitric oxide (NO) upon combustion of a sample with oxygen at a temperature in excess of 1000°C. The NO is reacted with ozone (O_3) to form a metastable form of nitrogen dioxide (NO_2^*). As the metastable form of nitrogen dioxide decays, a quanta of light is emitted in an amount directly proportional to the amount of nitrogen in the sample. The chemiluminescent emission is detected by a photomultiplier tube at a specific wavelength.

$$R\text{-}N + O_2 \text{ -----> } NO + H_2O + \text{Oxides}$$
$$NO + O_3 \text{ ----> } NO_2^* + O_2 \text{ ----> } NO_2 + h\nu$$

An ANTEK Instruments, Inc. Model 7000N Nitrogen Analyzer (Figure A-1) is used to determine total nitrogen of 5µl of a preserved water sample. The instrument is run according to the Installation/ Operation/Service Manual provided by ANTEK Instruments, Inc., except that Oxygen gas is used as a carrier gas instead of Argon to promote complete recovery of the nitrogen in the water samples. Total nitrogen is determined on unfiltered samples, while total dissolved nitrogen is determined on filtered samples. An autosampler is used to inject the samples into the analyzer.

3.0 Interferences

There are no known interferences with this procedure as long as all glassware is cleaned properly, and deionized water (DIW) is used to make the standards. The emission wavelength is completely specific for nitrogen, therefore, there is no interference with other compounds. In addition, the method is specific for chemically bound nitrogen and does not detect dinitrogen (N_2). Matrix interferences are eliminated by analyzing only water samples.

Care must be taken not to contaminate samples or laboratory glassware. Given the small size of the autoanalyzer vials,

cleaning is not practical, therefore, autoanalyzer vials are obtained clean directly from the manufacturer, used once, then discarded. All laboratory glassware used for standard preparation are washed with hot tap water and liquinox, rinsed with hot tap water, rinsed with hydrochloric acid, then triple rinsed with analyte-free water. The inside of all glassware, sample bottles, autoanalyzer vials or the autoanalyzer injection needle should not be touched since human contact can contaminate the samples with nitrogen.

4.0 Safety Precautions

In general, this method is environmentally safe. Strong chemicals or acids are not used, and no hazardous wastes are produced. The use of fume hoods or protective clothing is not required. Care is advised during handling of the high pressure gases as well as the pyrotech tube. All gas lines, regulators, gas filters, etc. need to be specified for high pressure oxygen gas. In addition, the pyrotech tube operates at combustion temperatures of over 1000°C, and care should be taken during maintenance to avoid serious thermal burns.

5.0 Apparatus and Materials

An ANTEK Instruments, Inc. Model 7000N Nitrogen Analyzer equipped with the following:

- Autosampler
- Chemiluminescent Nitrogen Detector
- Gas/Liquid Inlet System
- Pyro Tech Furnace and accessories
- Printer
- Oxygen Supply regulated to 20 psig

Standard laboratory glassware is used for preparation of standards. Glass vials, 1.5µl, with teflon/silicon septa crimp seals are used for the autoanalyzer.

6.0 Reagents

Reagents are limited to primary standards, secondary standards, and hydrochloric acid (used as a preservative). All standards are prepared with DIW.

6.1 Primary Standards

Primary standards are made by dissolving 0.3612 g of anhydrous Potassium Nitrate (KNO_3) in 100 ml of DIW in a volumetric flask. This primary standard has a nitrogen concentration of 500 mg/l. Preserve with 1 ml of chloroform and store at room temperature for no more than one year.

6.2 Secondary Standards

Using microliter pipets, add the required volumes of primary standard into 100 ml flasks and dilute to the mark with DIW to produce the following secondary standards:

Volume of Primary Standard added to 100 ml flask	Concentration of N in Secondary Standard
0 µl	0 mg/l (0 µmol/l)
100 µl	0.5 mg/l (35.7 µmol/l)
200 µl	1.0 mg/l (71.4 µmol/l)
400 µl	2.0 mg/l (142.9 µmol/l)
1000 µl	5.0 mg/l (357.1 µmol/l)

These secondary standards are used to check instrument calibration on an annual basis or upon replacement of the pyrolysis tube.

6.3 Working and Continuing Calibration Standards

A working standard of 2.0 mg/l (described above) is prepared identical to samples. Specifically, 1.5 ml of this standard is placed into a glass autoanalyzer sample vial and acidified with 10 µl of 3 N HCl. Duplicate vials of this standard are run in triplicate prior to each run, after every 20 samples, and at the end of the run to check instrument calibration.

6.4 Hydrochloric Acid

ACS reagent grade 3 N HCl is made by adding 125 ml of concentrated 12 N HCl into a 500 ml flask and diluting to the mark with DIW.

7.0 Calibration

Low level calibration curves have been performed in triplicate for both freshwater and seawater and yield the following results:

Low Level Calibration

µM Concentrations	Instrument Counts Freshwater	Instrument Counts Seawater
0	204, 193, 199	1003, 987, 1003
0.5	1002, 993, 974	2113, 1987, 2223
1.0	2304, 2403, 2311	3034, 3081, 3003
2.0	4686, 4688, 4803	5396, 5388, 5203
5.0	10101, 10591, 10188	11201, 11903, 11514

Low level linear calibration curves determined by plotting instrument response count against standard concentration are illustrated on Figure A-2. Note that the slopes of the curves for freshwater and seawater are similar with correlation coefficients of greater than 0.99.

High level calibration curves prepared in triplicate for both freshwater and seawater yield the following results:

High Level Calibration

µM Concentrations	Instrument Counts Freshwater	Instrument Counts Seawater
0	794,656,600	715,682,701
25	45808,48213,48218	47541,46822,47849
50	99402,93619,96219	94880,97103,96847
100	192390,190000,187230	186458, 188999, 190506
125	239200,248870,233380	238024, 242743, 241527
150	287070,297870,291080	293001, 290901, 292006
200	389000,385330,382110	380841, 387848, 387095

High level linear calibration curves are illustrated on Figure A-3 and indicate identical slopes (5.17×10^{-4}) for both matrices with correlation coefficients of 0.9999.

Linearity of instrument response only needs to be checked on an annual basis or whenever the pyrolysis tube is replaced. Once the instrument calibration is determined to be linear (regression coefficient >0.98), then only a one-point instrument calibration is required on a daily basis. The ANTEK instrument is programmed such that a zero nitrogen concentration produces an instrument response of zero. The one-point calibration curve is obtained with a high working standard, usually 2.0 mgN/l (143 µM N). Prior to each run, duplicate vials of this standard are run in triplicate. The mean and standard deviation of the six results are determined. If the precision of these results is less than 5 %RSD, then a standard curve is determined by using the mean of the high standard and forcing the intercept through zero.

8.0 Quality Control

8.1 Calibration Check Standards

Calibration check standards are run at the beginning and end of each run, with each run consisting of no more than 20 samples. Calculate accuracy and determine it to be within 95 to 105%. If results fall outside this acceptable range, then all samples within the run must be analyzed again.

8.2 Duplicate and Replicate Analyses

Every sample is collected in duplicate and analyzed in triplicate to produce six results for each sample. Triplicate analysis is recommended given the small injection volume (5 µl) as well as the possibility of injecting an air bubble into the instrument. A mean and standard deviation of the six replicate results is determined, and any replicate result determined to be beyond 1 standard deviation away from the mean is discarded. A sample precision of 5 %RSD is considered acceptable. Values outside this range results in a re-analysis of the samples.

9.0 Sample Collection, Preservation, and Handling

Surface water samples are collected directly into clean, 120 ml plastic sample containers. Prior to sample collection, sample containers are triple-rinsed with the sample water. Samples are stored in the dark until delivery to the laboratory. Within 12 hours of sample collection, 1.5 ml of sample is placed into a glass autoanalyzer sample vial and acidified with 10 µl of 3 N HCl. The vial is sealed with a teflon/silicon lined crimp cap and stored in a refrigerator at 2°C until analyzed. When preserved by this method, samples have a shelf life of three months without deterioration; however, sample analysis within 28 days is recommended.

10.0 Sample Extraction/Preparation

Other than the preservation procedures described above, there are no sample extraction or preparation procedures.

11.0 Sample Cleanup and Separation

There are no additional sample cleanup or separation protocol necessary to separate the nitrogen from the sample matrix. Combustion of the sample and its reaction with ozone is completed by the ANTEK Instruments, Inc. Model 7000N Nitrogen Analyzer.

12.0 Sample Analysis

The ANTEK Instruments, Inc. Model 7000N Nitrogen Analyzer is generally operated at the following conditions:

Pyrolysis Temperature	1100°C
Pyrolysis Oxygen flow:	2.0 psig
Oxygen to Ozone flow:	2.0 psig
System Pressure:	1.0 psig
Vacuum:	25 in/Hg
PMT Voltage:	800
Gain:	X50 Hi
Sample volume:	5 µl

Using pipets, 2 ml of sample is put into autoanalyzer vials and capped. The first two vials consist of the blank water that is used to rinse the instrument. The next two vials are high standards followed by a vial of blank water. Samples are loaded into the following vials separated by vials containing blanks to rinse the injection system between samples. The autoanalyzer collects 5µl from each sample vial and injects it into the instrument. The instrument is programmed to perform a triplicate analysis of each sample. A calibration check standard is analyzed at the end of every run, which does not exceed 20 samples.

13.0 Calculations

No special calculations are required. Instrument peak height values are converted directly to sample concentrations by comparison to the standard curve. Method precision, accuracy, and detection limit are determined according to the equations presented in Section 11. An example of the instrument raw data is attached along with determination of precision, accuracy, and method detection limit for that run.

14.0 Confirmation

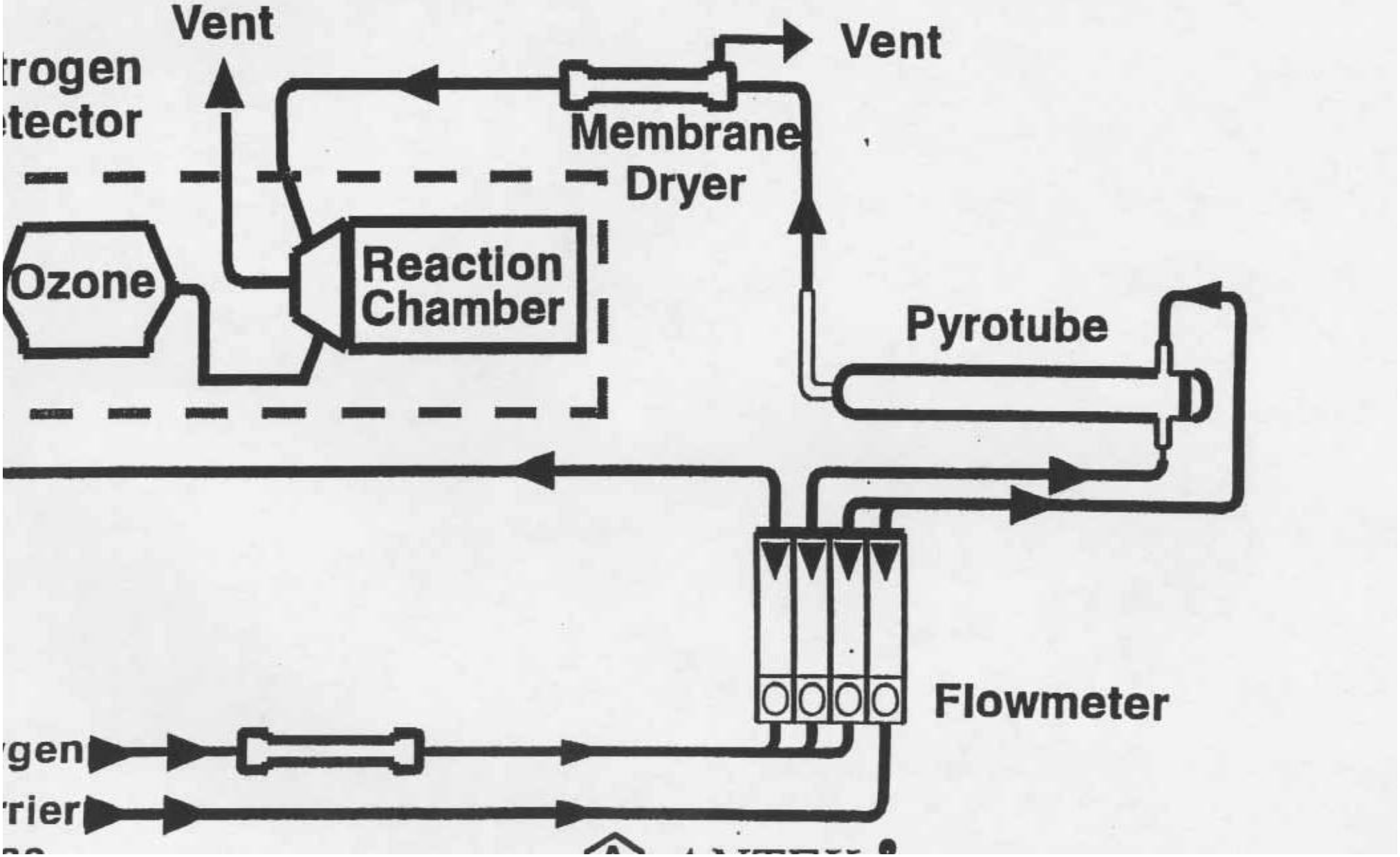
Confirmation of the presence of nitrogen in each sample can be confirmed by performing Total Kjeldhal Nitrogen method plus nitrate or by UV persulfate digestion.

15.0 Method Performance

This method measures total nitrogen of fresh and saline water samples at concentrations between 2.1 µmol/l (0.03 mg/l) to 250 µmol/l (3,500 µg/l) at a precision of 5% relative standard deviation (%RDS) or better, and an accuracy between 95 and 105%. There are no interferences with this method. In addition, there is no required sample extraction procedure and sample preparation is minimal.

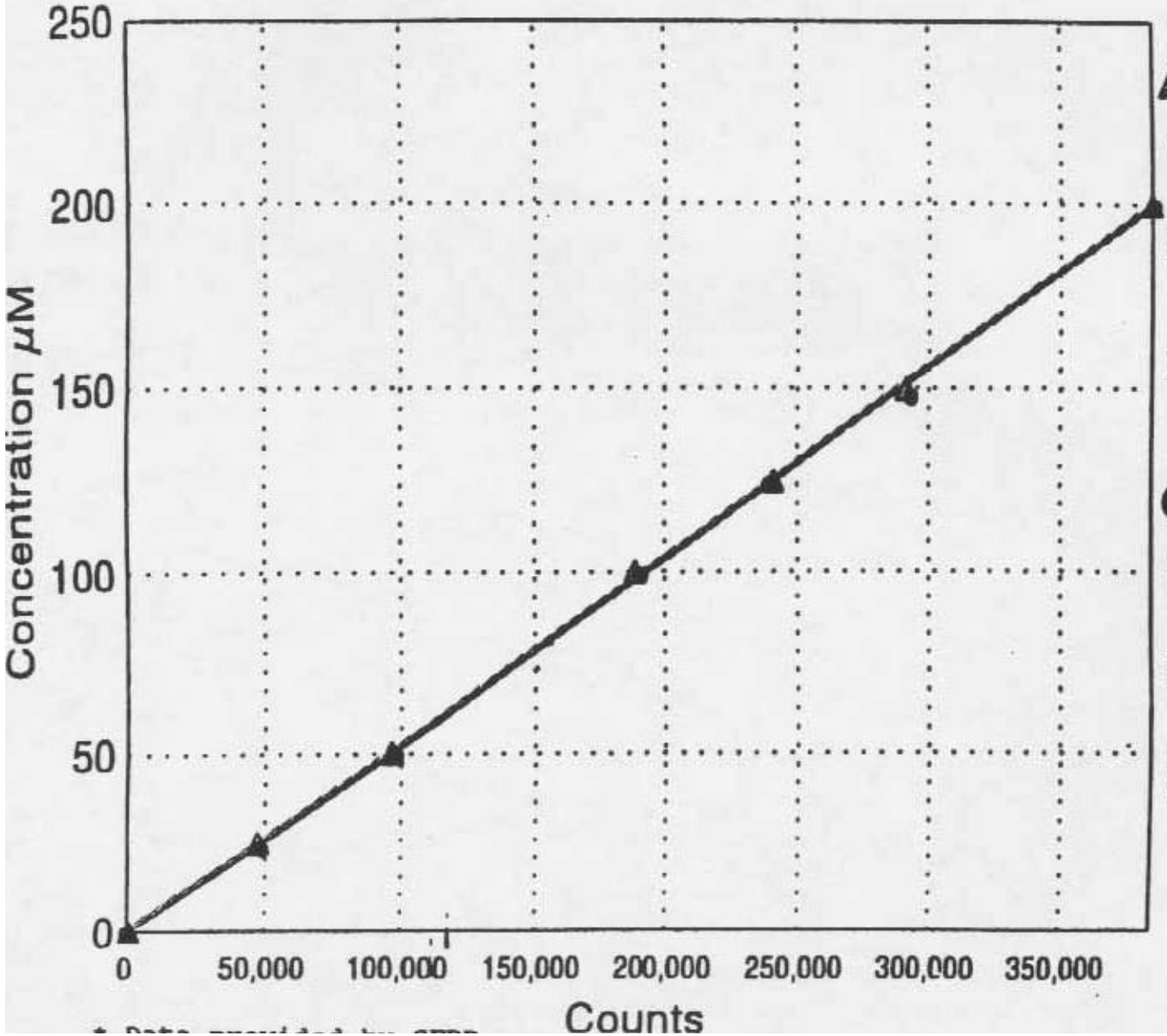
Figure A-1.

Flow Diagram Antek Nitrogen System



Calibration Data (High Level)

Freshwater & Seawater



▲Seawater:

correlation coefficient:
r=0.9999
slope:
m=5.1787 x 10-4
y-intercept:
b=3.0416 x 10-1

●Freshwater:

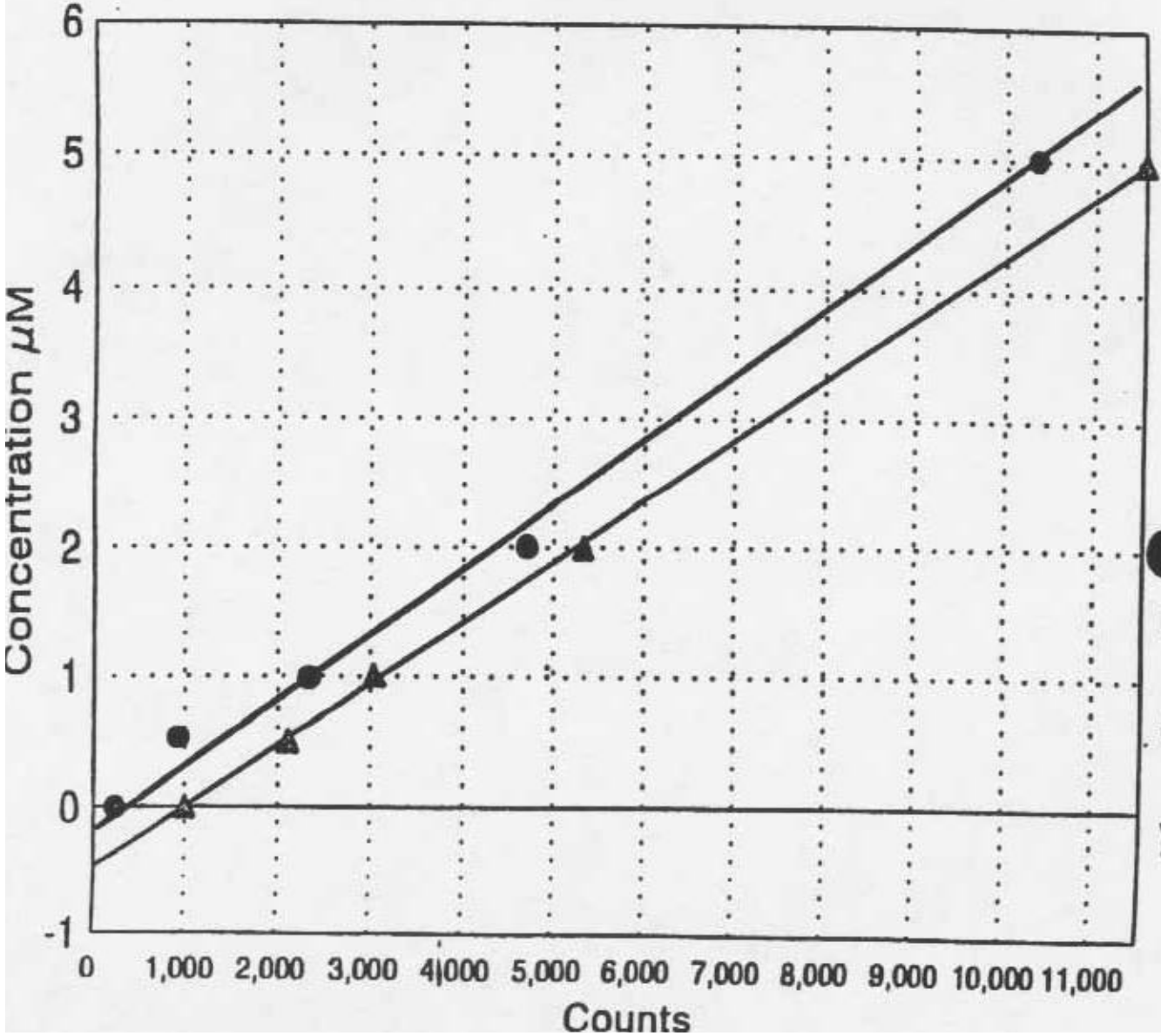
correlation coefficient:
r=0.9999
slope:
m=5.1773 x 10^{-4}
y-intercept:
b=2.3147 x 10^{-1}

ANTEK INSTRUMENTS, INC

Figure A-3.

Calibration Data (Low Level)

Freshwater & Seawater



▲Seawater:

correlation coefficient:
r=0.9998
slope:
$m=4.7401 \times 10^{-4}$
y-intercept:
$b=-4.8167 \times 10^{-1}$

●Freshwater:

correlation coefficient:
r=0.9980
slope:
$m=4.8840 \times 10^{-4}$
y-intercept:
$b=-1.1166 \times 10^{-1}$

ANTEK

Table A-1.
Instrument Raw Data

High Standard

Peak Height	Mean	S.D.
328068	316981	9961
326476		
315434		
307124		
307802		

Y Coef.: 6.31E-06

Sample No.	Peak Height	Conc. mg/l	Mean	S.D.
18	68059	0.43		
18	73595	0.46		
18	76348	0.48		
18	72947	0.46		
18	53989	0.34		
18	56782	0.36	0.46	0.06
19	63046	0.40		
19				
19	63181	0.40		
19	58140	0.37		
19	56652	0.36		
19	69979	0.44	0.39	0.03
20	101584	0.64		
20	88086	0.56		
20	93045	0.59		
20	93943	0.59		
20	103294	0.65		
20	88642	0.56	0.59	0.04
21	143391	0.90		
21	129645	0.82		
21	87297	0.55		
21	146675	0.93		
21	143873	0.91		
21	124534	0.79	0.80	0.14
22	143138	0.90		
22	126648	0.80		
22	129014	0.81		
22	124290	0.78		
22	122970	0.78		
22	117537	0.74	0.83	0.05

Table A-1 Continued.
Instrument Raw Data

Sample No.	Peak Height	Conc. mg/l	Mean	S.D.
23	113338	0.72		
23	97275	0.61		
23	109933	0.69		
23	114808	0.72		
23	85867	0.54		
23	109311	0.69	0.69	0.07
24	103168	0.65		
24	101434	0.64		
24	89431	0.56		
24	98077	0.62		
24	94929	0.60		
24	95829	0.60	0.62	0.03
25	51495	0.32		
25	50899	0.32		
25	48765	0.31		
25	46430	0.29		
25	48899	0.31		
25	51564	0.33	0.31	0.01
26	51896	0.33		
26	54313	0.34		
26	50917	0.32		
26	56950	0.36		
26	53244	0.34		
26	55425	0.35	0.34	0.01
27	37557	0.24		
27	36379	0.23		
27	36302	0.23		
27	36827	0.23		
27	38671	0.24		
27	126059	0.80	0.23	0.23
28	64364	0.41		
28	60120	0.38		
28	56844	0.36		
28	62889	0.40		
28	60725	0.38		
28	52691	0.33	0.39	0.03

Table A-1 Continued.
Instrument Raw Data

Sample No.	Peak Height	Conc. mg/l	Mean	S.D.
29	97467	0.61		
29	95358	0.60		
29	94148	0.59		
29	94593	0.60		
29	89004	0.56		
29	96449	0.61	0.60	0.02
30	113388	0.72		
30	120673	0.76		
30	105595	0.67		
30	87972	0.56		
30	113795	0.72		
30	115067	0.73	0.67	0.07
31	161965	1.02		
31	199033	1.26		
31	175726	1.11		
31	164179	1.04		
31	173689	1.10		
31	170957	1.08	1.11	0.08
32	142926	0.90		
32	138120	0.87		
32	130854	0.83		
32	136164	0.86		
32	121559	0.77		
32	129145	0.81	0.86	0.05
33	159314	1.01		
33	144694	0.91		
33	168716	1.06		
33	179937	1.14		
33	186718	1.18		
33	165232	1.04	1.03	0.09
34	150815	0.95		
34	159985	1.01		
34	153592	0.97		
34	145461	0.92		
34	154167	0.97		
34	157292	0.99	0.96	0.03

Table A-1 Continued.
Instrument Raw Data

Continuing Calibration Check Standards	
Peak Height	Conc. mg/l
330896	2.09
311059	1.96
324867	2.05
315522	1.99
329304	2.08

Table A-2.
Sample and QC Calculations

High Standard

Peak Height	Mean	S.D.
328068	316981	9961
326476		
315434		
307124		
307802		

Coef. of 6.31E-06

Continuing Calibration Check Standards

Peak Height	Conc. mg/l	Accuracy
330896	2.09	104
311059	1.96	98
324867	2.05	102
315522	1.99	100
329304	2.08	104

Sample No.	Peak Height	Conc. mg/l	Mean	S.D.	Precision
18	68059	0.43			
18	73595	0.46			
18	76348	0.48			
18	72947	0.46			
18					
18			0.46	0.02	5
19	63046	0.40			
19					
19	63181	0.40			
19	58140	0.37			
19	56652	0.36			
19			0.39	0.02	5
20					
20	88086	0.56			
20	93045	0.59			
20	93943	0.59			
20					
20	88642	0.56	0.58	0.02	3
21	143391	0.90			
21	129645	0.82			
21					
21	146675	0.93			
21	143873	0.91			
21			0.88	0.05	5

Table A-2.
Sample and QC Calculations

Sample No.	Peak Height	Conc. mg/l	Mean	S.D.	Precision
22					
22	126648	0.80			
22	129014	0.81			
22	124290	0.78			
22	122970	0.78			
22	117537	0.74	0.80	0.03	3
23	113338	0.72			
23					
23	109933	0.69			
23	114808	0.72			
23					
23	109311	0.69	0.71	0.02	2
24	103168	0.65			
24	101434	0.64			
24					
24	98077	0.62			
24	94929	0.60			
24	95829	0.60	0.64	0.02	4
25	51495	0.32			
25	50899	0.32			
25	48765	0.31			
25	46430	0.29			
25	48899	0.31			
25	51564	0.33	0.31	0.01	4
26	51896	0.33			
26	54313	0.34			
26	50917	0.32			
26	56950	0.36			
26	53244	0.34			
26	55425	0.35	0.34	0.01	4
27	37557	0.24			
27	36379	0.23			
27	36302	0.23			
27	36827	0.23			
27	38671	0.24			
27			0.23	0.01	3

Table A-2.
Sample and QC Calculations

Sample No.	Peak Height	Conc. mg/l	Mean	S.D.	Precision
28	64364	0.41			
28	60120	0.38			
28	56844	0.36			
28	62889	0.40			
28	60725	0.38			
28			0.39	0.02	5
29	97467	0.61			
29	95358	0.60			
29	94148	0.59			
29	94593	0.60			
29	89004	0.56			
29	96449	0.61	0.60	0.02	3
30	113388	0.72			
30	120673	0.76			
30	105595	0.67			
30					
30	113795	0.72			
30	115067	0.73	0.71	0.03	5
31	161965	1.02			
31					
31	175726	1.11			
31	164179	1.04			
31	173689	1.10			
31	170957	1.08	1.06	0.04	4
32	142926	0.90			
32	138120	0.87			
32	130854	0.83			
32	136164	0.86			
32					
32	129145	0.81	0.86	0.04	4
33	159314	1.01			
33					
33	168716	1.06			
33					
33					
33	165232	1.04	1.03	0.03	3

Table A-2.
Sample and QC Calculations

Sample No.	Peak Height	Conc. mg/l	Mean	S.D.	Precision
34	150815	0.95			
34	159985	1.01			
34	153592	0.97			
34	145461	0.92			
34	154167	0.97			
34	157292	0.99	0.96	0.03	3

Method Detection Limit for the run determined on the sample with lowest concentration.

Sample	Peak Height	Conc. mg/l	Mean	S.D.	MDL (3*S.D.)
27	37557	0.24			
27	36379	0.23			
27	36302	0.23			
27	36827	0.23			
27	38671	0.24			
27			0.23	0.01	0.03

15.0 Quality Assurance Reports

SERP will submit quality assurance reports for all Quality Assurance Project Plans at a frequency according to Table V of Appendix D of the QA Manual. The Quality Assurance Officer is responsible for the preparation of these reports. In general, if no audits were performed and no significant QA/QC problems have been identified, then SERP will prepare a brief letter stating these facts in lieu of a detailed quality assurance report.

A detailed QA report will be prepared when:

1. Activities were conducted in a manner other than those described by the CompQap or QAPP.
2. Preservation or holding requirements were not met.
3. Quality control checks were unacceptable.
4. Precision, accuracy, or MDL objectives were not met.
5. Corrective action was taken.
6. Internal or external audits were conducted and discrepancies were noted.

According to FDEP guidelines, these QA reports will include the following:

1. Title Page including the time period of the report, the QA Project Plan Title and Plan number, the laboratory name, address and phone number, and the preparer's name and signature.
2. Table of contents if the report is over 10 pages long.
3. The results of performance or system audits to include, date of audit, system tested, name of auditor, parameters analyzed, results of tests, deficiencies or failures, and an explanation of the problem and the corrective action taken.
4. Significant QA/QC problems.
5. Corrective actions taken.

APPENDIX B

Results of QC Check Samples for Total Phosphorus in Soil/Tissue

Solórzano L. and J.H. Sharp. 1980. Determination of Total Dissolved Phosphorus and Particulate Phosphorus in Natural Waters. 25(4). pp. 754-758.

Attached are the results of SERP's analysis of NIST standard reference material 1572 (citrus leaves) and sawgrass leaves for total phosphorus. Each of the samples were prepared according to the preparation procedures described in Section 8 and developed by Solórzano and Sharp (1980; see attached). Following the sample preparation, total phosphorus concentrations were determined according to the EPA Method 365.1.

Eight NIST standards (CL1 - CL8) were prepared each day and analyzed in replicate, while three samples of the sawgrass leaves (L1 - L3) were prepared each day and analyzed in replicate. The mean values of each data set are summarized below along with the resulting accuracy and precision estimates.

Sample Mean (µg/gm)	Standard Deviation	Accuracy %R	Precision %RSD
Citrus Leaves			
1378	46	99.7	3
1398	31	101	2
1384	20	100	1
1381	18	100	1
1363	20	99.0	1
X=1381	S.D.= 13		%RSD=1
Sawgrass Leaves			
1780	18	98.3	1
1747	18	99.8	1
1748	37	99.9	2
1751	20	100	1
1727	10	98.7	1
X=1750	S.D.= 19		%RSD=1

The NIST sample is certified to 1300 µg/gm ± 200 µg/gm (see enclosed certificate of analysis) for an accuracy range of 85% to 115% recovery. SERP's accuracy for the NIST standard ranged from 99 to 101% recovery, is well within the NIST limits. For the sawgrass leaves, SERP's accuracy ranged from 98 to 100% recovery. Precision for both the NIST standard and the sawgrass leaves varied between 1 and 3% RSD.

Total Phosphorus Sediments RFA

Citrus Leaves

Date: May 22, 1989

DW Blank = 0 Baseline offset = 0
100uM STD-3097ug/l 64.88 STD height = 64.88

Sample Site	Sediment Dry Wt.	Base OFST	PK HGT	Cor Avg	PO4 Conc. (ug P/gm)
CL1	0.0094	0	27.00		
			27.00	27.00	1371
CL2	0.0098	0	29.50		
			29.50	29.50	1437
CL3	0.0111	0	33.00		
			33.00	33.00	1419
CL4	0.0093	0	26.25		
			26.25	26.25	1347
CL5	0.0098	0	27.00		
			25.75	26.38	1285
CL6	0.0103	0	29.75		
			29.50	29.63	1373
CL7	0.0100	0	28.75		
			29.00	28.88	1378
CL8	0.0095	0	27.75		
			27.75	27.75	1394
L1	0.0099	0	36.50		
			36.50	36.50	1760
L2	0.0109	0	41.75		
			40.25	41.00	1796
L3	0.0109	0	40.75		
			40.75	40.75	1785

Total Phosphorus Sediments RFA

Citrus Leaves - B

Date: May 22, 1989

DW Blank = 0 Baseline offset = 0
100uM STD-3097ug/l 64.88 STD height = 64.88

Sample Site	Sediment Dry Wt.	Base OFST	PK HGT	Cor Avg	PO4 Conc. (ug P/gm)
CL1	0.0096	0	28.25		
			28.50	28.38	1411
CL2	0.0110	0	32.25		
			32.25	32.25	1400
CL3	0.0100	0	29.25		
			29.00	29.13	1390
CL4	0.0106	0	29.75		
			30.00	29.88	1345
CL5	0.0122	0	41.00		
			35.75	38.38	~~1502~~
CL6	0.0110	0	31.75		
			32.00	31.88	1383
CL7	0.0100	0	30.25		
			30.25	30.25	1444
CL8	0.0105	0	31.00		
			31.25	31.13	1415
L1	0.0120	0	45.25		—
			43.50	44.38	1765
L2	0.0098	0	36.00		
			35.75	35.88	1748
L3	0.0096	0	34.75		
			34.75	34.75	1728

Total Phosphorus Sediments RFA

Citrus Leaves - B

Date: May 23, 1989

DW Blank =	0	Baseline offset =	0
100uM STD-3097ug/l	65.00	STD height =	65.00

Sample Site	Sediment Dry Wt.	Base OFST	PK HGT	Cor Avg	PO4 Conc. (ug P/gm)
CL1	0.0105	0	31.00		
			31.00	31.00	1407
CL2	0.0096	0	27.25		
			27.50	27.38	1359
CL3	0.0094	0	27.75		
			27.75	27.75	1407
CL4	0.0101	0	28.75		
			28.75	28.75	1356
CL5	0.0094	0	27.25		
			28.00	27.63	1400
CL6	0.0094	0	27.25		
			27.50	27.38	1388
CL7	0.0104	0	29.50		
			30.50	30.00	1374
CL8	0.0093	0	27.00		
			27.00	27.00	1383
L1	0.0119	0	44.50		
			44.50	44.50	1782
L2	0.0109	0	39.75		
			40.50	40.13	1754
L3	0.0113	0	40.50		
			40.50	40.50	1706

Total Phosphorus Sediments RFA

Citrus Leaves

Date: May 23, 1989

DW Blank = 0 Baseline offset = 0
100uM STD-3097ug/l 65.00 STD height = 65.00

Sample Site	Sediment Dry Wt.	Base OFST	PK HGT	Cor Avg	PO4 Conc. (ug P/gm)
CL1	0.0104	0	29.25		
			29.75	29.50	1352
CL2	0.0103	0	29.75		
			29.75	29.75	1376
CL3	0.0105	0	31.00		
			31.00	31.00	1407
CL4	0.0092	0	26.50		
			26.50	26.50	1372
CL5	0.0104	0	30.00		
			30.25	30.13	1380
CL6	0.0095	0	27.50		
			30.25	28.88	~~1443~~
CL7	0.0110	0	31.50		
			31.50	31.50	1364
CL8	0.0102	0	29.75		
			29.75	29.75	1370
L1	0.0096	0	34.75		
			35.00	34.88	1731
L2	0.0099	0	36.50		
			36.50	36.50	1757
L3	0.0106	0	39.50		
			39.25	39.38	1770

Total Phosphorus Sediments RFA

Citrus Leaves

Date: May 25, 1989

DW Blank = 0 Baseline offset = 0
100uM STD-3097ug/l 66.81 STD height = 66.81

Sample Site	Sediment Dry Wt.	Base OFST	PK HGT	Cor Avg	PO4 Conc. (ug P/gm)
CL1	0.0095	0	27.25		
			28.00	27.63	1348
CL2	0.0097	0	29.00		
			29.00	29.00	1386
CL3	0.0094	0	27.25		
			27.25	27.25	1344
CL4	0.0104	0	30.75		
			30.75	30.75	1371
CL5	0.0096	0	28.00		
			28.00	28.00	1352
CL6	0.0112	0	33.50		
			33.50	33.50	1387
CL7	0.0093	0	27.50		
			27.75	27.63	1377
CL8	0.0098	0	28.25		
			28.25	28.25	1336
L1	0.0114	0	42.50		
			42.75	42.63	1733
L2	0.0099	0	36.50		
			36.75	36.63	1715
L3	0.0111	0	41.50		
			41.50	41.50	1733

U. S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1572

Citrus Leaves

This Standard Reference Material (SRM) is intended primarily for use in calibrating instrumentation and evaluat the reliability of analytical methods for the determination of major, minor, and trace elements in botanical materi agricultural food products, and similar matrices.

Certified Values of Constituent Elements: The certified values for the constituent elements are shown in Table 1. Th are based on results obtained either by definitive methods of known accuracy or by two or more independent analyti methods. Non-certified values, which are given for information only, appear in Table 2.

Notice and Warnings to Users:

Expiration of Certification: This certification is invalid 5 years after the shipping date. Should it be invalidated befo then, purchasers will be notified by NBS.

Stability: The material should be kept in its original bottle and stored at temperatures between 10-30 °C. It should not exposed to intense sources of radiation. Ideally, the bottle should be kept tightly closed in a desiccator in the dark at t temperature indicated.

Use: The bottle should be shaken well before each use. A minimum sample of 500 mg of the dried material (see Instru tions for Drying) should be used for any analytical determination to be related to the certified values of this certificat

Statistical consultation was provided by K. Kafadar of the Statistical Engineering Division.

The overall direction and coordination of the analyses leading to this certification were performed under the chairman ship of E.L. Garner, Chief of the Inorganic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Referenc Material were coordinated through the Office of Standard Reference Materials by R. Alvarez.

Washington, D.C. 20234
December 20, 1982
(Revision of Certificate
dated 2-22-82)

George A. Uriano, Chief
Office of Standard Reference Material

(over)

Table 1. Certified Values of Constituent Elements

Major and Minor Constituents

Element	Content,[1] (Wt. Percent)
Calcium	3.15 ± 0.10
Magnesium	0.58 ± 0.03
Phosphorus	0.13 ± 0.02
Potassium*	1.82 ± 0.06
Sulfur	0.407 ± 0.009

Trace Constituents

Element	Content,[1] µg/g	Element	Content,[1] µg/g
Aluminum	92 ± 15	Manganese	23 ± 2
Arsenic	3.1 ± 0.3	Mercury	0.08 ± 0.02
Barium	21 ± 3	Molybdenum	0.17 ± 0.09
Cadmium	0.03 ± 0.01	Nickel	0.6 ± 0.3
Chromium	0.8 ± 0.2	Rubidium*	4.84 ± 0.06
Copper	16.5 ± 1.0	Sodium	160 ± 20
Iodine	1.84 ± 0.03	Strontium*	100 ± 2
Iron	90 ± 10	Zinc	29 ± 2
Lead*	13.3 ± 2.4		

[1] Based on dry weight: For drying instructions, see the section of this certificate on Instructions for Drying. The uncertainties are based on judgment and represent an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability for samples weighing 500 mg or more.

*For those elements determined by definitive methods, the uncertainties are given as 95%/95% statistical tolerance intervals. See The Role of Standard Reference Materials in Measurement Systems, NBS Monograph 148, 1975 p 14.

Table 2. Non-certified Values for Constituent Elements

NOTE: The following values are not certified because they are not based on the results of either a definitive metho known accuracy or two or more independent methods. These values are included for information only.

Major Constituent

Element	Content,[1] (Wt. Percent)
Nitrogen	(2.86)

Trace Constituents

Element	Content,[1] µg/g	Element	Content,[1] µg
Antimony	(0.04)	Samarium	(0.052)
Bromine	(8.2)	Scandium	(0.01)
Cerium	(0.28)	Selenium	(0.025)
Cesium	(0.098)	Tellurium[a]	(0.02)
Chlorine	(414)	Thallium	(≤0.01)
Cobalt	(0.02)	Tin	(0.24)
Europium	(0.01)	Uranium	(≤0.15)
Lanthanum	(0.19)		

[1] Analytical values are based on the "dry weight" of material (See Instructions for Drying).

[a] Not sufficiently homogeneous for certification.

Instructions for Drying: Samples of this SRM must be dried before weighing and analysis by either of the followin procedures:

1. Drying for 2 hours in air in an oven at 85 °C.
2. Drying for 24 hours at 20 to 25 °C and at a pressure not greater than 30 Pa (0.2 mm Hg).

Additional Information on Analyses: This SRM contains siliceous material, which is an integral part of the sample. Th values in Tables 1 and 2 are based on analyses performed on the *entire* sample. Therefore, dissolution procedures should be capable of complete dissolution of the sample but should not result in losses of volatile elements, such as arsenic and mercury.

Source and Preparation of Material: The plant material for this SRM was collected and prepared under the direction of A. L. Kenworthy, Michigan State University. Its source was the Lake Alfred area of central Florida. The material was air-dried, ground in a comminuting machine to pass a 425-µm (No. 40) sieve, dried at 85 °C, and thoroughly mixed in a feed blender. After packaging the material in polyethylene-lined fiber drums, it was sterilized in situ with cobalt-60 radiation. The sterilization procedure was carried out at the U.S. Army Research and Development Command, Natick, Mass. under the direction of A. Brynjolfsson.

Analytical Methods Used and Analyses

Analytical Methods:

A. Atomic absorption spectrometry
B. Atomic emission spectrometry, flame
C. Atomic emission spectrometry, inductively coupled plasma
D. Ion chromatography
E. Isotope dilution thermal source mass spectrometry
F. Isotope dilution spark source mass spectrometry
G. Kjeldahl method for nitrogen
H. Neutron activation
I. Photon activation
J. Polarography
K. Spectrophotometry

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Limnol. Oceanogr., 25(4), 1980, 754–758

Determination of total dissolved phosphorus and particulate phosphorus in natural waters[1]

Abstract—Procedures are presented for separate analyses of total dissolved phosphorus and total particulate phosphorus in natural waters. The method for both procedures involves drying a sample with magnesium sulfate and baking the residue at a high temperature to decompose organic phosphorus compounds. The residue is then treated with hydrochloric acid to hydrolyze polyphosphates and the orthophosphate is measured by the molybdate method. The method gives 100% recovery with refractory phosphorus compounds, is usable on undiluted samples with up to 18 μmol of phosphorus per liter (6 μg P per sample), and has a mid-range precision of ±1%.

Dissolved and particulate organic phosphorus in seawater are not often measured, although some quantitative studies have been done (e.g. Ketchum et al. 1955; Strickland and Austin 1960; McGill et al. 1964; Holm Hansen et al. 1966). Attempts have also been made to quantify fractions of the dissolved phosphorus (Watt and Hayes 1963; Strickland and Solórzano 1966) and particulate phosphorus pools (Correll 1965). The dissolved organic phosphorus pool has been indicated as a potentially important source of phosphorus for phytoplankton (Chu 1946; Harvey 1953). Part of the reason that more is not known about the abundance, distribution, and cycling of organic phosphorus in the sea is analytical inadequacy.

Several methods developed for the analysis of organic phosphorus depend on acidic oxidation of organic molecules to release orthophosphate (Redfield et al. 1937; Harvey 1948; Hansen and Robinson 1953). Menzel and Corwin (1965)

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used potassium persulfate as the oxidant of organic phosphorus. Oxidation to orthophosphate is also the mechanism in the ultraviolet irradiation method (Armstrong et al. 1966). All these methods, measuring total phosphorus, also detect initially nonreactive phosphorus compounds that are not necessarily organic. The last two methods are used at present in seawater analysis. We have found incomplete yield with the UV method when adenosine-5′-triphosphate (ATP) was used as a test compound and poor precision with the persulfate method. The UV method requires high intensity ultraviolet lamps and heat for efficient yield of phosphate from ATP (Armstrong and Tibbitts 1968; Goossen and Kloosterboer 1978).

We have developed procedures suitable for both total dissolved phosphorus and total particulate phosphorus. Our separation between particulate and dissolved is arbitrary (*see* Solórzano and Sharp 1980). The method is essentially a high temperature combustion of dried samples (Assoc. Official Agric. Chem. 1936) that has been used for particulate matter from freshwater (Stockner and Armstrong 1971). The procedure for particulate phosphorus is modified from that of Stockner and Armstrong because we found poor recovery of orthophosphate without the addition of magnesium sulfate. The procedure for total dissolved phosphorus was developed from the particulate one. A somewhat similar method, without $MgSO_4$, was used by Levine et al. (1955). Since the forms of the samples are different and different concentrations of reagents are required, the dissolved and particulate methods are described separately. In both cases, reagent grade chemicals and high quality distilled water *must* be used (we used reverse osmosis/deionized water from a Millipore-Milli-RO/Milli-Q system).

This work and that on dissolved organic nitrogen (Solórzano and Sharp 1980) are offered as simple, accurate, and precise methods for routine use on samples from fresh, estuarine, or oceanic waters. The difficulties with the UV method and the special equipment it requires justify the method presented here.

Total dissolved phosphorus

Magnesium sulfate, 0.17 M—dissolve 10 g of $MgSO_4$ (or 21 g of $MgSO_4 \cdot 7H_2O$) in 500 ml of distilled water and add 1 ml of concd H_2SO_4. This solution can be stored for months in a glass bottle.

Hydrochloric acid, 0.75 M—dilute 65 ml of concd HCl to 1 liter with distilled water. This solution can be stored for months in a glass bottle.

Mixed reagent—this is the mixed reagent from the standard method for soluble reactive phosphorus (Murphy and Riley 1962) as outlined by Strickland and Parsons (1972). As specified by Strickland and Parsons, it should be made immediately before use and not stored for more than about 6 h.

The procedure given is for total dissolved phosphorus on samples with >15‰ salinity; for samples of lower salinity, the variation given below should be used. Dissolved organic phosphorus is determined by subtracting the ambient soluble reactive phosphorus concentration from the measured total.

The sample should be filtered to remove particulate matter. We use pretreated GF/C filters (*see below*). Measure 10 ml of sample into a small Pyrex container and add 0.2 ml of 0.17 M $MgSO_4$. The ideal container is a 40- × 50-mm weighing bottle with outside ground glass cover. Evaporate the sample to dryness in a clean oven at 95°C; this step takes about 2.5 h. Transfer the weighing bottle to a muffle furnace and bake at 450°–500°C for 2 h. After cooling, add 3 ml of 0.75 M HCl and heat the sample in an oven at 80°C for 20 min. Then add 7 ml of distilled water and continue heating for an additional 10 min. The evaporation step should be done without the covers, the baking step should be done with loose covers, and hydrolysis must be done with covers on tightly. After hydrolysis, cool and transfer the sample to a test tube, add 1 ml of the mixed reagent and after 10 min read the absorbance at 885 nm in a 1- or 10-cm cuvette.

For samples with salinity <15‰, a modification must be used to ensure complete recovery of phosphate. After adding the 3 ml of acid, heat the samples without covers in a 80°C water bath for 10 min. Then add 7 ml of distilled water and heat the samples without covers for an additional 10 min. After cooling, transfer samples to a graduated cylinder and bring the volume to 10 ml. Thereafter, treat the sample as above.

Particulate phosphorus

Sodium sulfate, 0.17 M—dissolve 12 g of anhydrous Na_2SO_4 in 500 ml of distilled water. This solution can be stored for months in a glass bottle.

Magnesium sulfate, 0.017 M—dissolve 2 g of $MgSO_4$ in 1 liter of distilled water. This solution can be stored for months in a glass bottle.

Hydrochloric acid, 0.2 M—dilute 16 ml of concd HCl to 1 liter with distilled water. This solution can be stored for months in a glass bottle.

Table 1. Yield of phosphate from organic and inorganic salts with ($MgSO_4$) and without (no $MgSO_4$) addition of magnesium sulfate and with (hydrol) and without (no hydrol) final acidic hydrolysis step. All yields as percentage of theoretical value; 100 indicates theoretical yield (95–105%).

Compound	No $MgSO_4$, No hydrol	No $MgSO_4$, Hydrol	$MgSO_4$, Hydrol
Sodium phosphate			
monobasic	30	90	100
dibasic	15	58	100
Riboflavin phosphate	29	66	100
Phosphoryl chloride	53	90	100
Dexametasone phosphate	22	59	100
Sodium glycerophosphate	10	100	100
ATP	12	40	100
Disodium phenylphosphate	16	84	100

Collect the particulate matter on a pretreated (baked at 450°–500°C for 0.5–1 h, not acid-rinsed) glass-fiber filter. We have used Whatman GF/C filters of 24- and 42.5-mm diameter and have used mild suction (<20 cm of Hg) in filtering. After filtration, rinse the filter twice with 2-ml aliquots of 0.17 M Na_2SO_4 and then place it in a weighing bottle (*see above*). Soak the filter in the bottle with 2 ml of 0.017 M $MgSO_4$ and then dry the mixture in a 95°C oven. Next, transfer the weighing bottle to a 450°–500°C muffle furnace and bake for 2 h. After cooling, add 5 ml of 0.2 M HCl and heat in an 80°C oven for 30 min. As with the total dissolved phosphorus method, the evaporation step should be done with bottles unsealed and the baking and hydrolysis steps with the covers on tightly. After the hydrolysis step, cool, and pour the supernatant into a centrifuge tube. Rinse the bottle with 5 ml of distilled water and add to the centrifuge tube. Add 1 ml of mixed reagent and centrifuge at about 2,000 rpm for 5 min. Read the optical density at 885 nm in a 1- or 10-cm cuvette.

Solutions of varying concentrations of guanosine-5′-monophosphate, guanosine-5′-diphosphoglucose, adenosine-5′-triphosphate (ATP), deoxyribonucleic acid, riboflavin phosphate, potassium glycerophosphate, sodium glycerophosphate, glucosamine-6-phosphate, phosphoenol pyruvate, disodium phenylphosphate, tris-p-nitrophenyl phosphate, dexametasone phosphate, phosphoryl chloride, potassium orthophosphate (monobasic), and sodium orthophosphate (both monobasic and dibasic salts) have been analyzed by the dissolved organic phosphorus method at concentrations from 0.12 to 18.0 μM phosphorus. Yields of phosphate from these 15 compounds were 95–103%; this indicates that the method is accurate. Five replicates of 6 μM ATP were run which gave a relative standard error of the mean (1 SD) of 1%. When magnesium sulfate was omitted from distilled water samples of phosphorus compounds, recovery was variable. Table 1 shows yields of a series of standards with and without the $MgSO_4$ addition and with and without the final hydrolysis. The $MgSO_4$ is used as an acidic solution (after addition to the seawater sample, the pH was about 3) to minimize silicate leaching from the glassware during evaporation. The acid and heating are necessary to hydrolyze any condensed phosphates in the final mixture. The modification for freshwater and low salinity samples is needed because without it the acidity of the sample can retard color development of the phosphomolybdate complex. We found this problem somewhat evasive at first because of the variability in strength of concd HCl in older bottles of the acid. Seawater salts apparently buffer the rehydrated sample sufficiently, and the partial evaporation in the modification also places the pH in the correct range (>0.7) for optimal color development.

We have used the procedure for dissolved phosphorus on estuarine to oceanic samples (salinity 0–36‰) with ambient phosphate concentrations ranging from <0.05 to 3.0 μM, and dissolved organic phosphorus concentrations from <0.05 to 0.8 μM. We have also used it on samples from a recirculating seawater system (salinity ca. 30‰) with phosphate concentrations ranging from 0.30 to 3.70 μM and dissolved organic phosphorus concentrations ranging from 0.08 to 0.55 μM.

Because the particulate phosphorus determination follows the same steps as that for dissolved organic phosphorus, it is also considered to be accurate. The thoroughness and precision of this method have been checked by analysis of filters on which varying volumes were collected of cultures of the marine diatoms *Skeletonema costatum* and *Thalassiosira pseudonana*. The linearity (r^2 = 0.9992)

was similar to that found with varying concentrations of ATP. Absorbance from a reagent blank and from a filter blank were 0.015 and 0.020 in 10-cm pathlength cells.

For work at sea, we have found it convenient to use liquid scintillation vials for both dissolved and particulate phosphorus samples, processing the samples through the drying step and then storing them capped at room temperature for later analysis on land.

As with all organic analyses, special care including prior acid-washing is required with the glassware. The glassware should be used only for the organic phosphorus determinations. The weighing bottles should be used only for the evaporation, baking, and hydrolysis steps with transfer before the mixed reagent is added. Disposable scintillation vials can be used throughout the procedure (capped with foil for the baking step).

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APPENDIX C

An Equivalency Study on the Preservation of Nutrient Samples by Freezing or Refrigeration

Clementson, L.A. and S.E. Wayte. 1992. The Effect of Frozen Storage of Open-Ocean Seawater Samples on the Concentration of Dissolved Phosphate and Nitrate. Water Resources Research. 26(9). pp. 1171-1176.

INTRODUCTION

On January 5, 1995, Florida Department of Environmental Protection approved SERP's Comprehensive Quality Assurance Project Plan (CompQAP). The approval did not include the freezing of samples for ammonium analysis. FDEP recommends storage of ammonium samples at 4°C. In response to their comment, SERP has completed an equivalency study demonstrating no significant difference in the results of ammonium following storage by freezing or refrigeration. The results of this study are presented herein.

METHODS

On 2 March 1995, two 2-liter bottles of surface water were collected from Florida Bay Station 16 (Murray Key). The bottles were filled according to the protocols described in SERP's CompQAP. Specifically, the bottles were rinsed three times with sample water prior to being filled. The bottles were then stored in a dark cooler with ice and transported to SERP's laboratory on the same day of sample collection.

In the laboratory each bottle was vacuum-filtered through a Whatman GF/F glass fiber filter. From each filtered sample, 10 60-ml sample bottles were filled, for a total of 20 subsamples (10 labelled as F for frozen, and 10 labelled as R for refrigerated). Each of the sample bottles were rinsed three times with the sample prior to filling. The 10 F samples were stored in a freezer and the 10 R samples were stored in a refrigerator at 4°C until analyzed.

Sample analysis was performed according to EPA method 350.1 as described in SERP's CompQAP. Frozen samples were allowed to thaw slowly to room temperature and shaken thoroughly prior to analysis. Both a frozen and refrigerated sample were analyzed over the course of 35 days from sample collection.

RESULTS

Ammonium concentrations varied from 1.98 to 3.19 uM (Table C-1). A paired t-test was used to test the null hypothesis that there is no difference between the ammonium concentrations of the frozen and refrigerated samples (that they are from the same common population). The results of the paired t-test confirmed the null hypothesis at the 0.05 probability level ($t=-0.09$, $p=0.93$), indicating no difference between the ammonium concentrations of the frozen and refrigerated samples. In addition, there was no significant difference between the concentrations of the frozen and refrigerated samples for nitrate+nitrite ($t=0.61$, $p=0.56$), nitrite ($t=0.71$, $p=0.49$), and soluble reactive phosphorus ($t=2.10$, $p=0.07$).

Table C-1.
Results of Frozen (F) and Refrigerated (R) Nutrient Samples.

Sample	Days	Date	NH4-R (µM)	NH4-F (µM)	N+N-R (µM)	N+N-F (µM)	NO2-R (µM)	NO2-F (µM)	SRP-R (µM)	SRP-F (µM)
1	0	03-Mar-95	2.09	2.06	1.44	1.40	0.25	0.25	0.06	0.06
2	6	09-Mar-95	2.58	2.64	1.42	1.41	0.25	0.24	0.12	0.10
3	7	10-Mar-95	2.37	2.92	1.56	1.61	0.34	0.38	0.07	0.06
4	20	23-Mar-95	3.01	2.23	1.67	1.48	0.32	0.26	0.09	0.09
5	21	24-Mar-95	2.73	3.19	1.52	1.54	0.25	0.25	0.04	0.07
6	24	27-Mar-95	2.60	2.31	1.53	1.51	0.30	0.28	0.08	0.03
7	25	28-Mar-95	1.98	2.19	1.48	1.50	0.27	0.27	0.08	0.05
8	26	29-Mar-95	2.05	2.13	1.50	1.50	0.26	0.25	0.04	0.03
9	27	30-Mar-95	2.10	2.19	1.47	1.50	0.27	0.25	0.14	0.09
10	35	07-Apr-95	2.74	2.50	1.48	1.49	0.24	0.26	0.08	0.06

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THE EFFECT OF FROZEN STORAGE OF OPEN-OCEAN SEAWATER SAMPLES ON THE CONCENTRATION OF DISSOLVED PHOSPHATE AND NITRATE

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Abstract—Filtered seawater samples which were stored at −20°C in containers of different surface area to volume ratios were analysed for the effects of surface adsorption of nitrate and phosphate during storage for a maximum of 24 months. The results for nitrate showed no significant change in concentration over the duration of the experiment. However, the concentration of phosphate steadily decreased in samples stored for longer than 4 months. The loss of phosphate after this time cannot be explained at present, but this study showed that it was not due to the effects of surface adsorption.

Key words—seawater samples, frozen storage, storage time, surface adsorption, nitrate phosphate, nutrients.

INTRODUCTION

The storage of seawater samples for nutrient analyses has been a problem that has plagued researchers for more than 40 years. Harvey (1948) was one of the first researchers to deal with the problem. Ideally samples should be analysed immediately after collection; however, this is not always possible, and when analytical equipment fails during an intensive field program or sample collection is from a remote location, samples must be stored. Oceanographic studies are an example of an intensive field program where limited personnel, insufficient time to analyse samples between sample collections and weather conditions may all mean that samples have to be stored. It is therefore necessary to store samples so as to minimize any changes in the concentrations of nutrients with time.

The literature on storage techniques for nutrient samples describes an array of techniques. Some of these techniques and the corresponding results are summarized in Table 1. Samples have been stored by freezing, quick freezing with dry ice and the addition of "preservatives" (chemicals such as mercuric chloride, acid or chloroform). In addition some of the samples were filtered and others were not. Filtering can introduce contamination from the filters, but it does eliminate particulate matter that may cause adsorption or desorption of nutrients on standing and also removes some bacteria and plankton that may alter nutrient concentrations through metabolic processes.

One conclusion which is evident from all the studies examined is that the effectiveness of any one storage technique can be dependent upon the type of sample water tested, whether it be estuarine, coastal or open ocean water. The biological and physical characteristics of these water types are quite different and could therefore affect the concentration of nutrients in different ways.

Because of the contradictory results in the literature, we needed to determine how the samples we worked with would behave if stored. Kremling and Wenck (1986) and Hagebø and Rey (1984) showed that samples stored in a cool dark atmosphere (0–5°C) resulted in greater and more rapid loss of nutrients with time than samples that had been stored frozen. For this reason samples used in this study were frozen for storage. Filtered samples from the deep open ocean were stored in 4 containers of different volumes and surface areas for up to 2 years. The aims of this study were to determine how long samples can be stored frozen without affecting the reliability of analytical results for nitrate and phosphate and to examine the effects, if any, of container volume/surface area on changes in nutrient concentrations during storage.

MATERIALS AND METHODS

The sample was collected from a depth of 300 m at a site off the east coast of Tasmania (position: 41 30.5 S 148 46 E) during a cruise of FRV *Soela* in July 1986. The water depth at this position was 1260 m and the salinity at 300 m was 35.04. The sample was collected in three 8 l. PVC Niskin bottles on a CTD rosette and emptied into a 25 l acid-washed, seawater-conditioned high-density polyethylene carboy. Immediately after collection the sample was filtered through a Millipore 0.45 μm filter that had been soaked in 10% HCl solution. Approximately 600 ml of sample seawater was filtered and discarded before the seawater was filtered and retained for the experiment. Subsamples were taken and stored in 4 containers of different volume (1

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Table 1 Literature review of storage experiments from the past 30 years

Authors	Nutrients analyzed	Sample type	Filtered or unfiltered	Storage	Storage bottle	Maximum storage time	Conclusions
Krenkel and Wench (1956)	PO_4^{3-}, NO_3^-, SiO_3^{2-}	Open ocean 800 and 2000 m	NI	10 C, 4 C with without HgCl	Polypropylene, 60 ml	100 days	Frozen samples showed slight decrease with time Cooled samples showed marked increase with time
Hagebo and Rey (1984)	PO_4^{3-}, NO_3^-, SiO_3^{2-}	Open ocean 50 and 250 m	F (Whatman GF C)	10 C, 1 3 C with without $CHCl_3$	Polyethylene	51 days	Cooled samples showed no decrease with time for first 6 days Frozen samples showed variability
MacDonald and McLaughlin (1982)	PO_4^{3-}, NO_3^-, SiO_3^{2-}	Coastal and estuarine	F (0 45 µm Millipore) and NF	Quick frozen, freezer storage	Polystyrene, glass, 15 ml	365 days	Quick freezing reliable for PO_4^{3-} Both freezing techniques cause slight decrease in NO_3^-
Morse et al (1983)	PO_4^{3-}, NO_3^-, SiO_3^{2-}, NH_4^+	Open ocean 0 and 500 m	F (0 45 µm Nuclepore)	Frozen, cooled with without $HgCl_2$ with without acid	Polyethylene, Teflon and glass, 125 ml	60 days	No significant trend in concentration with time
Thayer (1970)	N, P	Estuarine	F (No 10 bolting cloth)	Quick frozen, cooled with without $CHCl_3$	Polyethylene bags, 1000 ml	10 days	Quick freezing alone stabilized nutrient concentrations at all times of the year
Gilmartin (1967)	PO_4^{3-}	Estuarine	F (Whatman No 1) and NF	Quick frozen, room temp with without $CHCl_3$	Polyethylene, 100 ml	4 days	Rate of change of PO_4^{3-} in quick frozen samples with $CHCl_3$ is significantly less than in frozen or unfrozen samples without $CHCl_3$
Murphy and Riley (1956)	PO_4^{3-}	Seawater	F (Whatman No 1)	Dark, 20 C with without NaF with without $CHCl_3$ with without $Al(OH)_3$ and $TrCO_3$	Soda glass and polyethylene	28 days	$CHCl_3$ was the only effective preservative Large losses of PO_4^{3-} when stored in polyethylene

250 ml bottles [high-density polyethylene with leakproof seal and screw cap (Kartell); surface area/volume = 0.87]; (2) 50 ml bottles (as for 1; surface area/volume = 1.46); (3) 20 ml scintillation vials [high-density polyethylene with polypropylene screw cap (Kartell); surface area/volume = 1.84] and (4) 15 ml tubes [polypropylene with push-on cap (Disposable Products Australia); surface area/volume = 2.78]. Except for the 15 ml tubes all containers had been soaked in 10% HCl solution for at least 48 h, rinsed 3 times with water from a Millipore Milli-RO/Milli-Q system and rinsed twice with the filtered seawater sample. The tubes were usually used as received from the manufacturer and therefore were not acid-washed, but were rinsed twice with the filtered seawater sample. Two replicates for each container for each storage period were taken. The containers were filled at random and to only 7/8 of their total volume. After all containers for the entire experiment had been filled they were frozen at −40°C in a blast freezer. All containers were in the freezer within 3 h of sample collection. After the cruise the subsamples were stored at −20°C. Subsamples were allowed to thaw and warm to room temperature over 2–3 h and analysed for nitrate and phosphate within a further 3–4 h after 9, 16, 30, 67, 94, 129, 212, 247, 302, 367, 590, 645 and 729 days.

Two weeks after this sample was collected a second sample was collected from a coastal site (position: 43°11'S 147°31'E; water depth 50 m; collection depth 10 m; salinity 34.01). The sample was filtered and stored frozen in acid-washed scintillation vials only. These subsamples were analysed each time a set of oceanic subsamples was analysed.

Phosphate and nitrate were determined using a Tecator 5020 flow injection analyser with a Tecator 5023 spectrophotometer and a Hewlett-Packard 7123A chart recorder. The method for nitrate analysis was based on those recommended by Anderson (1979) and Johnson and Petty (1983). For phosphate analysis the reagents used were based on those in Koroleff (1976) with slight modification to allow their use in a flow injection analysis system. A more detailed description of the methods can be found in Clementson *et al.* (1989). The detection limits and relative standard deviation of the methods were $0.1 \pm 0.01\ \mu M$ for nitrate and $0.15 \pm 0.01\ \mu M$ for phosphate.

The results were subjected to a two-factor analysis-of-variance with an F-test for linear trend in days. The linear trend is tested against mean square for deviations from regression rather than the residual mean square because there is a large amount of heterogeneity about the regression line (Sokal and Rohlf, 1981). In order to estimate the time at which the phosphate concentration began to decrease, a least-squares function-fitting routine (Miller 1981) was employed to fit a two phase linear model to the data.

RESULTS AND DISCUSSION

The variation in concentration of nitrate and phosphate in frozen oceanic seawater samples with time is shown in Figs 1 and 2. Before the statistical analysis was performed, one "maverick" data point was removed from the data set as only contamination could have accounted for it.

The total variability in the data from storage studies is due to the combination of several factors and not to storage time or type alone (MacDonald and McLaughlin, 1982; Kremling and Wenck, 1986). The total variability (S_{tot}) is equal to the variability within a group of replicates (S_r) plus the variability between identical samples analysed on different days

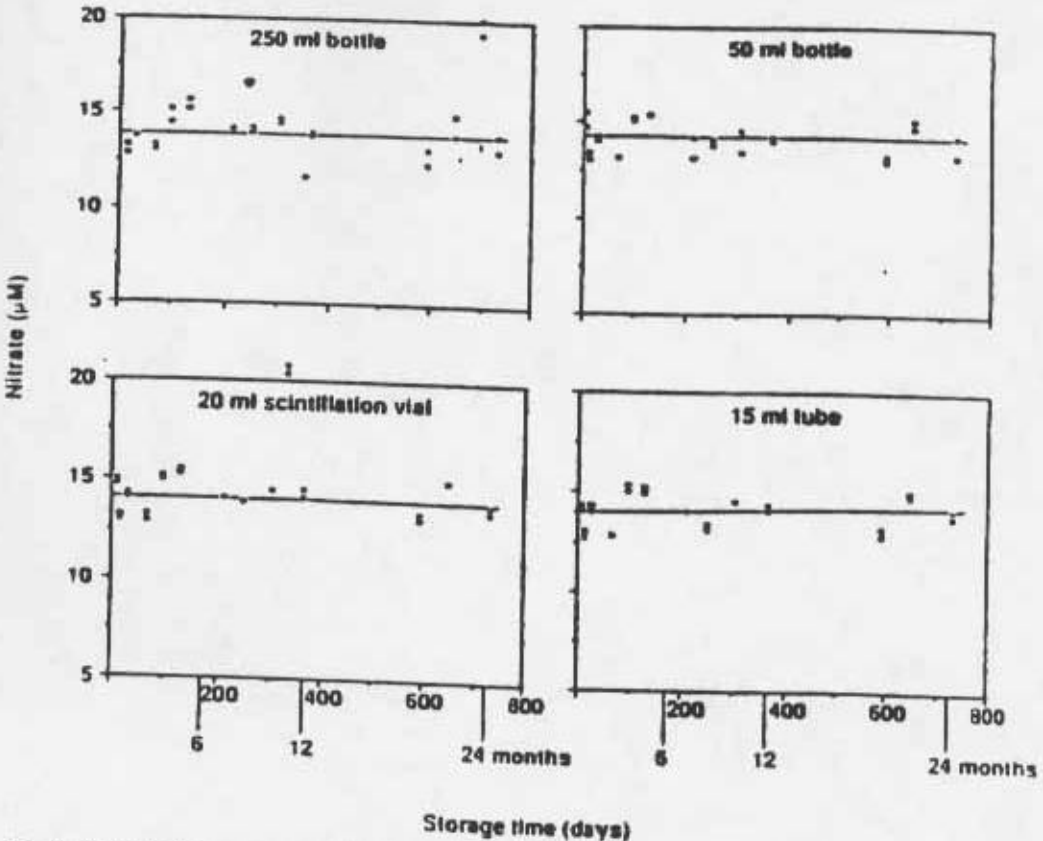

Fig. 1. Variation in concentration of dissolved nitrate with storage time for bottles of different volume and surface area.

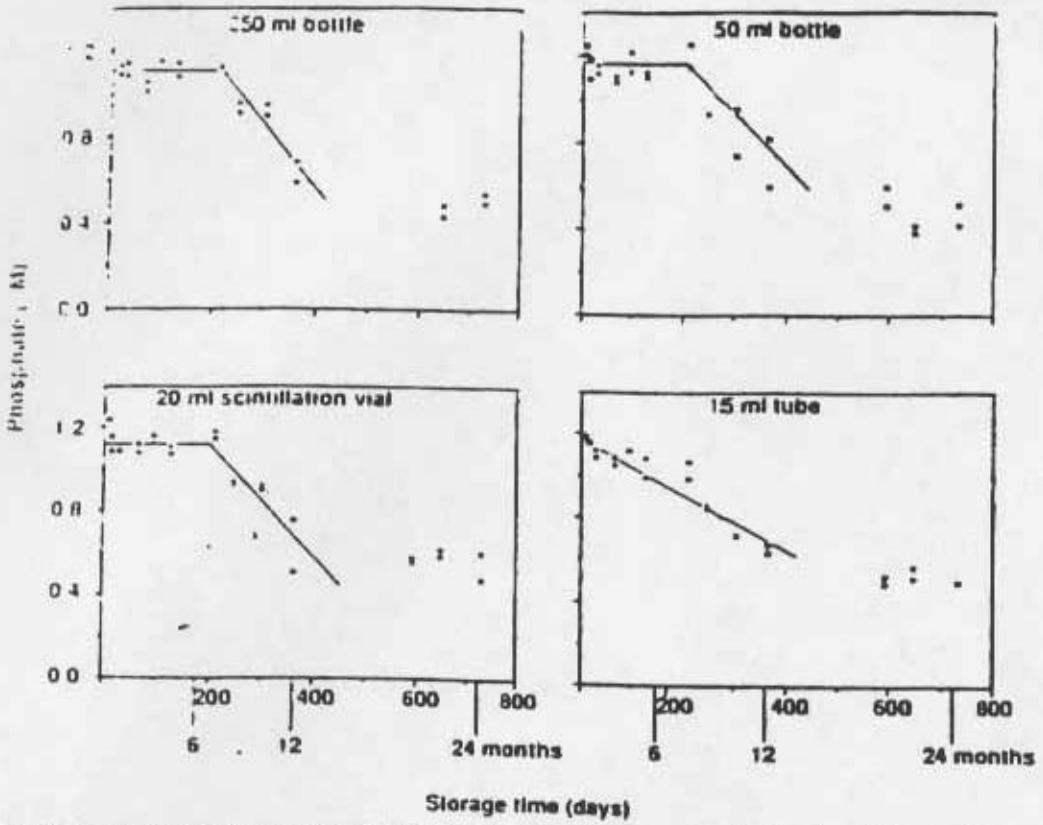

Fig. 2. Variation in concentration of dissolved phosphate with storage time for bottles of different volume and surface area.

S_r) plus the variability due to storage time and storage type (S_t).

$$S_{rr} = S_r - S_b + S_t.$$

The within-replicate variability is equal to the analytical precision of a set of sequentially analysed replicates and should remain the same throughout the experiment. The day to day variability is due to different operators, different blanks, recalibration effects and environmental effects. The analysis of variance examines the variability attributable to the two "treatments" storage time and storage type (S_t). The part of this variability attributable to storage time is confounded with S_b. However we would expect S_b to appear as random variation, whereas any effect on concentration due to the length of storage time would appear as a consistent trend over time.

Nitrate

The analysis of variance found no significant difference between the storage types [F-test (F) = 2.66; degrees of freedom (d.f.) = 3,50; probability (P) > 0.05]. The difference in concentration between storage times was significant (F = 53.91; d.f. = 12,50; $P < 0.001$), but as the concentration did not change linearly with storage time (F = 0.134; d.f. = 1,11; $P > 0.05$), and the storage types responded similarly at each storage time (no significant interaction: F = 1.43, d.f. = 35,50; $P > 0.05$), this difference must be due to the day to day variability (S_b).

The variability due to calibration effects ranges from 0.3 to 1.2% of the average concentration. The observed variability in concentration is greater than 1.2%, thus the day to day variability is mostly attributable to factors other than calibration uncertainty. The variability in samples does not appear to increase with storage time as found by other workers (MacDonald and McLaughlin, 1982; Kremling and Wenck, 1986).

The overall slope estimate from the regression of nitrate concentration on time was −0.00025 (SE = 0.000323). This implies a decrease in nitrate concentration of 1.2% over the 729 days of the study. Given the level of variability in this data, a slope of −0.00063 or a drop in concentration of 3% should have been observed to detect a statistically significant decrease in nitrate concentration with storage time.

Phosphate

The difference in concentration between storage times was highly significant (F = 201.83; d.f. = 12,50; $P < 0.001$), and this was largely attributable to a significant linear trend over time (F = 116.176; d.f. = 1,11; $P < 0.001$). A non-significant interaction (F = 1.54; d.f. = 34,50; $P > 0.05$) showed that storage types responded similarly over time. Figure 2 shows that the data for all storage types, except the 15 ml tube, fell into three distinct phases; the phosphate concentration remained constant for approximately the first 200 days and then decreased linearly for some

time before appearing to remain relatively constant for the remainder of the study. One of the prime aims of this study was to find out how long samples could be stored frozen without changes in phosphate concentration. Therefore it was important to determine the storage time at which the phosphate concentration started to decrease rather than try and fit a model to the entire set of data points. For this reason the data points corresponding to 590, 645 and 773 days storage time were omitted from further analysis (these were the data points that appeared to remain constant at a lower concentration). To determine the point at which the concentration began to decrease a model consisting of two straight lines, the first with zero slope, was fitted to the data (see Fig. 2). The estimated times, for each storage treatment, at which the phosphate concentration began to decrease and their standard errors are shown in Table 2. These results indicate that in the 250 ml bottle, 50 ml bottle and the scintillation vial, the phosphate concentration remained unchanged for the first 210 days of frozen storage and then steadily decreased at the same rate with increasing storage time until levelling off at approx. 400 days. The exact time at which the phosphate concentration stopped decreasing cannot be determined from this study. Due to equipment failure there was a period of 200 days in which no samples were analysed and it was during this period that the phosphate concentration stopped decreasing and started to remain at a relatively constant concentration. The decrease in phosphate concentration after 210 days cannot be explained at the present time. However, as the different surface area to volume ratios of the containers did not affect the concentrations, wall adsorption effects can be discounted as a possible reason for the loss of phosphate.

The analysis-of-variance also indicated a significant difference between storage types ($F = 4.55$; d.f. = 3,50; $P < 0.01$), but t-tests showed this to be entirely due to the tube having a lower mean phosphate concentration than the other containers. Furthermore a linear model ($R^2 = 0.89$) was the best fit to the data points for the tube (see Fig. 2) which indicates that the phosphate concentration began decreasing immediately on freezing. Why the tube behaves differently in the storage of phosphate samples is uncertain. Although the tube had the greatest surface area to volume ratio, the early loss of phosphate cannot be attributed to wall adsorption because this effect was not found in the other containers. However, the tube was the only container which was made of polypropylene rather than high-density polyethylene and also had not been acid-washed before the start of the experiment. Possibly one or both of these factors are responsible for the significant difference found in storing samples for phosphate analysis in the tubes rather than the other containers.

Kremling and Wenck (1986) suggest that phosphate could be taken up during the freezing and thawing periods by proliferating micro-organisms that attach themselves to the walls of the containers. However, it is not known why the micro-organisms remain in a "dormant" state for approx. 210 days before proliferating and then later return to a "dormant" state. If they attach themselves to the walls of the container when proliferating, this study, in which containers of different surface area to volume ratios were used, should have shown varying phosphate loss with storage type.

The above statistical results apply only to the oceanic seawater samples, but similar results were obtained for the samples from the coastal site. No linear trend was found between nitrate concentration and storage time. For phosphate a significant linear trend was found to exist between concentration and storage time.

In an attempt to resolve the behaviour of phosphate in stored samples a second experiment was initiated. Samples were collected in April 1989 from approximately the same position (41 40'S 148 45 E) and depth as those collected in July 1986. As there had been no difference between the 250 ml, 50 ml bottles and the scintillation vial in the first experiment, only 50 ml bottles and tubes were used in the second experiment. The bottles and tubes were divided into two sets: those that had been acid-washed as previously described and those that were used as received from the manufacturer. The containers were filled and stored in the same manner as the containers in the first experiment and were analysed after 16, 30, 62, 90, 120, 150, 180, 214, 247 and 367 days.

As in the first experiment the analysis-of-variance indicated that the concentration of phosphate responded differently over time for the acid-washed 50 ml bottle and the acid-washed tube ($F = 2.63$; d.f. = 9,20; $0.01 < P < 0.05$). For the tubes the analysis-of-variance showed a significant interaction between storage time and acid washing ($F = 3.81$; d.f. = 9,20; $P < 0.01$). A linear model was the best fit to the data points for both the acid-washed and non-acid washed tubes; however the slope of the line was greater for non-acid-washed tubes indicating that acid-washed tubes were preferable to

Table 2. Results of fitting a 2-phase linear model to phosphate concentrations

Storage type	R^2	Time of decrease (days)		Slope (μM day	
		Estimate	SE	Estimate	SE
250 ml bottle	0.891	211	13	-0.0028	0.0003
50 ml bottle	0.819	210	16	-0.003	0.0005
Scintillation vial	0.847	210	15	-0.003	0.0004
Tube	2-phase model could not be fitted				

... acid-washed bottles for the storage of seawater samples for phosphate analysis. That is the concentration of phosphate decreases more slowly in acid-washed bottles. For the 50 ml bottle the model consisting of two straight lines, the first with zero slope, was the best fit to the data points for both acid-washed and non-acid-washed bottles. No significant difference was found between these parameters ($F = 0.31$, $df = 1,20$, $P = 0.587$). However the time at which the phosphate concentration started to decrease was determined to be different in the two experiments, 210 days for the first experiment and 147 days for the second experiment. This difference may be explained by the different composition of the seawater samples used in the two experiments and could be tested in a future study with samples collected from several different locations.

CONCLUSION

The results from these storage experiments indicate that freezing can be a reliable and effective means of storing oceanic and coastal samples for nutrient analysis. The concentration of nitrate showed no significant trend with increasing storage time. For reliable phosphate analyses, the frozen samples must be analysed within approx. 4 months of collection. After this time the phosphate concentration will start to decrease steadily with increasing storage time. From the containers used in this study the preferable storage treatment is acid-washed, high-density polyethylene bottles. Storage containers with different surface area to volume ratios showed similar trends in nutrient concentration with storage time, thus discounting the loss of phosphate due to the effects of surface adsorption.

Acknowledgement—The authors would like to thank the Captain and crew of FRV *Soela* for their assistance with the sample collection.

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APPENDIX D

An Equivalency Study on the Preservation of Total Organic Carbon Samples With and Without Acid

INTRODUCTION

On January 5, 1995, Florida Department of Environmental Protection approved SERP's Comprehensive Quality Assurance Project Plan (CompQAP). The approval did not include the analysis of total organic carbon (TOC) by EPA Method 415.1 without pH preservation of samples, unless SERP demonstrated through an equivalency study, that non-pH preserved samples produced equivalent results. In response to their comment, SERP has completed an equivalency study demonstrating no significant difference in the results of TOC samples with and without pH preservation. The results of this study are presented herein.

METHODS

On 2 March 1995, two 2-liter bottles of surface water were collected from Florida Bay Station 16 (Murray Key). The bottles were filled according to the protocols described in SERP's CompQAP. Specifically, the bottles were rinsed three times with sample water prior to being filled. The bottles were then stored in a dark cooler without ice and transported to SERP's laboratory on the same day of sample collection.

In the laboratory one of the bottles was acidified with 0.5 ml of concentrated hydrochloric acid. The pH of this sample was checked to be less than 2.0 by pouring some of the sample onto a strip of pH paper. Both the acidified and non-acidified samples were split into fifteen 125 ml sample bottles, for a total of 30 subsamples (15 labelled Acid TOC and 15 labelled No Acid TOC). Each of the sample bottles were rinsed three times with the sample prior to filling. These samples were stored in a refrigerator at 4°C until analyzed.

Sample analysis was performed according to EPA method 415.1 as described in SERP's CompQAP. Both an acidified and non-acidified sample were analyzed over the course of 33 days from sample collection, corresponding with EPA's recommended holding time for organic carbon analyses.

RESULTS

TOC results varied from 5 to 8 mg/l (Table D-1). Although the sample results varied from day to day, the results of the acidified and non-acidified samples varied similarly. A paired t-test was used to test the null hypothesis that there is no difference between the TOC results from the acidified and non-acidified samples (that they are from the same common population). The results of the paired t-test confirmed the null hypothesis at the 0.05 probability level ($t=1.34$, $p=0.20$), indicating no difference

between the TOC concentrations of the acidified and non-acidified samples.

Table D-1.
Results of Acidified and Non-Acidified TOC Samples.

Sample	Date	Days	TOC (mg/l) No Acid	TOC (mg/l) Acid
1	02-Mar-95	0	8.395	8.412
2	03-Mar-95	1	6.395	6.145
3	09-Mar-95	7	7.489	7.420
4	10-Mar-95	8	7.196	7.084
5	15-Mar-95	13	7.541	7.763
6	23-Mar-95	21	6.271	5.935
7	24-Mar-95	22	7.559	7.038
8	25-Mar-95	23	5.130	4.814
9	26-Mar-95	24	6.626	6.130
10	27-Mar-95	25	5.616	5.888
11	28-Mar-95	26	6.885	6.921
12	29-Mar-95	27	7.348	7.392
13	03-Apr-95	32	6.385	6.568
14	04-Apr-95	33	5.513	5.550

APPENDIX E

SERP Standard Operating Procedures

Standard Operating Procedures for Total Phosphorus Analysis

Southeast Environmental Research Program
Florida International University

Version 1

September 1997

Table of Contents

Total Phosphorus in Soils and Sediments

Method ID: R4 Phosphorus.Sed M1

1. Scope and Application

This method is used for the determination of total phosphorus in soils and sediments. The working range is from 0.5 to 10 mg/L of prepared sample (instrument cal. range).

2. Sample Preparation

2.1 A portion of the sample is dried at 105° C.

2.2 The dried sample is ground. Discard rocks.

3. Reagents

3.1 Sulfuric acid solution (H_2SO_4): Add 25 Ml of conc. H_2SO_4 to DI water and dilute to 1 liter.

3.2 Potassium persulfate ($K_2S_2O_8$): Dissolve 4 grams $K_2S_2O_8$ in DI water and dilute to 1 liter.

4. Digestion Procedure

4.1 Do not use commercial detergents for glassware used in this determination.

4.2 Weigh 0.1 to 0.2 grams dried sample into an autoclavable phosphorus-free container and add 5 mL DI water.

4.3 Add 5 mL of the sulfuric acid solution (3.1) and 10 mL of the potassium persulfate (3.2).

4.4 Prepare a 100 ppm PO_4 as P intermediate standard. A set of standards (suggested range: 0.5 to 10 mg/L PO_4 as P) are digested in the same manner as the samples.

4.5 Cover the container loosely with a cap or foil and autoclave for 30 minutes at 250°F (120° C) and 15 psi.

5. Analysis

5.1 After digestion, phosphorus is determined by the automated single reagent method (Methods for the Chemical Analysis of Water and Wastes, EPA, Method 365.1).

5.2 The wash water is 6.2 mL H_2SO_4 per liter.

6. Calculations

Dry weight basis:

$$\text{Phos mg/kg} = \mu\text{g/g} = \frac{\mu\text{g/mL phos} \times 5\text{ mL}}{\text{grams sa} \times \%\text{solids (decimal)}}$$

1.0 Total Phosphorus Methods

For the determination of total phosphorus in water, soil, sediment, and tissue samples, SERP does not use the typical ammonium persulfate digestion because of the explosive hazards and special handling requirements associated with the use of this chemical. Instead, SERP uses a modification of the sample preparation methods described by Solórzano and Sharp (1980, *Determination of total dissolved phosphorus and particulate phosphorus in natural waters.* Limnol. Oceanogr., 25(4), pp. 754-758 (see attached). Total phosphorus is determined in water, soil, sediment, and tissue samples by oxidizing and hydrolyzing all of the phosphorus-containing compounds in a sample to soluble reactive phosphate. Soluble reactive phosphate then is determined by reacting phosphate with molybdenum (VI) and antimony (III) in an acid medium to form a phosphoantimonylmolybdenum complex; this complex is reduced with ascorbic acid to form a colored dye.

Analysis for soluble reactive phosphorus is performed by wet chemical analysis using a single-channel Alpkem RFA-510 Nutrient Analyzer following EPA Method 365.1 and the procedure suggested by the Alpkem Corporation, modified for optimum conditions in our laboratory. SERP analyzes total phosphorus on a separate autoanalyzer from that used for soluble inorganic nutrient analysis.

1.1 Sample Preparation

Sample preparation for total phosphorus determination is done as soon as possible. Water samples should be prepared within 24 hours of sample collection, and are often prepared immediately upon return to the laboratory.

1.1.1 Sample Preparation Reagents

Magnesium sulfate ($MgSO_4$), 0.17 N: 10.475 g magnesium sulfate is dissolved in 250 ml DIW. 0.5 ml concentrated sulfuric acid is then added.

Hydrochloric acid (HCl):

0.06 N - 5 ml conc. hydrochloric acid in 1 L total volume DIW.
0.12 N - 10 ml conc. hydrochloric acid in 1 L total volume DIW.
0.18 N - 15 ml conc. hydrochloric acid in 1 L total volume DIW.
0.24 N - 20 ml conc. hydrochloric acid in 1 L total volume DIW.

(Use caution when making acid solutions. Always add acid to water. The mixing of acid in water may generate heat.)

1.1.2 Water Sample Preparation

1. Prepare a tray with two or three 8 ml scintillation vials (without the aluminum-lined caps) per sample bottle plus two vials for DIW method reagent blanks, two vials for SPEX standards, and two vials for each matrix spike. Add 100 µl of 0.17 N $MgSO_4$ to each vial using the 5.0 ml dispenser tip (1 = 100 µl) and an Eppendorf pipet setting of

1. Fill out a total phosphorus preparation log sheet with the placement and contents of the vials on the tray. Record the date the $MgSO_4$ used was made.
2. Add 5 ml of sample water into each of the vials.
3. Place tray in an 80□C oven and evaporate to dryness (usually overnight). Record the date and time the samples were placed in the oven and removed from the oven on the log sheet.
4. Ash the samples at 550□C in a muffle furnace for 3 hours and allow to cool overnight. Transfer the vials from the plastic trays to the metal trays, keeping the vials in the exact same order as outlined on the sample preparation log sheet. A melt pellet (melting point of 550□C) should be placed in an empty vial in an empty space on one of the trays to confirm that the furnace reached 550□C. Record the date and time the samples were placed in the furnace and removed from the furnace and whether or not the pellet melted. Once cool, return the vials from the metal trays to the plastic trays, keeping the vials in the exact same order as outlined on the sample preparation log sheet.
5. Hydrolyze each sample with the addition of 5 ml of hydrochloric acid. The normality of the acid is dependent on the salinity of the sample according to the following table:

Salinity	HCl concentration
0.0 - 15 ppt	0.06 N
16 - 38 ppt	0.12 N
39 - 55 ppt	0.18 N
56 - 80 ppt	0.24 N

Record the date and time the acid was added, the acid concentration(s) used, and the date(s) the acid(s) was/were made on the sample preparation log sheet.

6. Cap each sample tightly with polylined caps, shake using a vortexer, and put into an 80□C oven overnight. After removing from the oven, allow to cool and shake again. Record the dates and times the vials were taken out of the oven and shaken on the sample preparation log sheet.

1.1.3 Soil, Sediment and Tissue Sample Preparation

1. Dry sample in an 80□C oven for 2 days, then grind.
2. Prepare a tray with two 10 ml glass scintillation vials (without the aluminum-lined caps) per sample plus two vials for citrus leaf standards. Fill out a total phosphorus preparation log sheet with the placement and contents of the vials on the tray.
3. Add 25 mg of sample, 200 µl of 0.17 N $MgSO_4$, and 1 ml DIW to each vial. Record the date the $MgSO_4$ was made.
4. Place tray in an 80□C oven and evaporate to dryness (usually overnight). Record the date and time the samples were placed in the oven and removed from the oven on the log sheet.
5. Ash the samples at 550□C in a muffle furnace for 3 hours and allow to cool overnight. Transfer the vials from the plastic trays to the metal trays, keeping the vials in the exact

same order as outlined on the sample preparation log sheet. A melt pellet (melting point of 550□C) should be placed in an empty vial in an empty space on one of the trays to confirm that the furnace reached 550□C. Record the date and time the samples were placed in the furnace and removed from the furnace and whether or not the pellet melted. Once cool, return the vials from the metal trays to the plastic trays, keeping the vials in the exact same order as outlined on the sample preparation log sheet.

6. Hydrolyze each sample with the addition of 10 ml of 0.24 N hydrochloric acid. Record the date and time the acid was added and the date the acid was made on the sample preparation log sheet.
7. Cap each sample tightly with polylined caps, shake using a vortexer, and put into an 80□C oven overnight. After removing from the oven, allow to cool and shake again. Record the dates and times the vials were taken out of the oven and shaken on the sample preparation log sheet.
8. Analyze at a 1:10 dilution (200 μl of sample with 1800 μl DIW).

1.2 Analysis

1.2.1 Autoanalyzer Reagents

All reagents are made with the high reagent-grade chemicals dissolved in double-deionized water.

Blank and wash water: The salinity of the blank water and the samples effect the shape of the peaks from the autoanalyzer, therefore we match the salinity of the blank and wash water to the salinity of the samples. Samples with salinities of 15 ppt or less are run with nutrient-free DIW acidified with sulfuric acid (2.0ml/15L). Samples with salinities greater than 15 ppt are run with nutrient-free seawater acidified with sulfuric acid (3.0ml/15L). The nutrient-free seawater is obtained from the Sargasso Sea and stored in carboys fitted with ammonia traps. Blank and wash water are pumped directly from these carboys to the autoanalyzer.

Total phosphorus analysis requires five reagents which are mixed just prior to the analysis to make a working reagent.

Antimony potassium tartrate: 0.75 g antimony potassium tartrate is dissolved in 250 ml DIW.

Ammonium molybdate: 20 g ammonium molybdate is dissolved in 500 ml DIW. Do not refrigerate.

Sulfuric acid solution: 140 ml conc. sulfuric acid is added to 900 ml DIW.
(Use caution when making acid solutions. Always add acid to water. The mixing of acid in water may generate heat.)

Ascorbic acid: 6.0 g ascorbic acid is dissolved in 200 ml acetone and 200 ml DIW. Refrigerate.

Sodium dodecyl sulfate (SDS), 15% w/w: 15 g sodium dodecyl sulfate is added to 85 ml DIW.

Working mixed reagent: Combine 50 ml sulfuric acid solution, 5 ml antimony potassium tartrate solution, 15 ml ammonium molybdate, 30 ml ascorbic acid, and 2 ml SDS.

1.2.2 Total Phosphorus Standards

The primary standard for total phosphorus is the same as that used for soluble reactive phosphate.

Phosphate primary standard, 1000 μM: Dissolve 0.1360 g potassium dihydrogen phosphate in 1 L DIW. Add 2 ml chloroform. Final concentration is the equivalent of 1.0 μmoles/ml.

SPEX standard, 0.50 μM: 15.5 μl of SPEX concentrate (1000 ppm) in 1 L total volume DIW.

Working standards for water samples are prepared from the primary standard in DIW or Sargasso Seawater depending upon the salinity of the samples. For water samples with salinities of 15 ppt or less, standards are prepared in DIW. Sargasso Seawater is used for water samples with salinities greater than 15 ppt. The working standards are made to bracket the expected concentration of the samples as shown in Table 3. A log book is kept by the total phosphorus autoanalyzer. In the log book record the slope and correlation coefficient of the calibration curve, the water matrix (freshwater or seawater), the instrument range setting, the technician's initials, and the preparation dates of the primary phosphorus standard and five reagents.

Table 1. Working standards for total phosphorus determination.

Working Standard	Volume of Primary Standard in 100 ml of DIW or Seawater	Phosphorus Standard Concentration (μM)
Low Standard Curve		
S1	10 μl	0.10
S2	50 μl	0.50
S3	100 μl	1.00
S4	150 μl	1.50
S5	200 μl	2.00
High Standard Curve		
S1	125 μl	1.25
S2	250 μl	2.50
S3	500 μl	5.00
S4	750 μl	7.50
S5	1000 μl	10.00
Standard Curve for Solid Samples		
S1	125 μl	3.87 μg/g
S2	250 μl	7.74 μg/g
S3	500 μl	15.48 μg/g
S4	750 μl	23.22 μg/g
S5	1000 μl	30.97 μg/g

1.2.3 Autoanalyzer Instrument Parameters

Alpkem Rapid Flow Analyzer (RFA; Alpkem Corp., Clackamas, OR)) Model 510 with Sampler Model 301
Flowcell: 5.5 mm
Filter: 660 nm
Heat bath: Omega Model CN9000A, 45□C
Rise time: 3 sec.
Absorbance range: 0.02 (for low standard curve) or 0.1 (for high or solid sample standard curves) AUFS
Sample time: 35 sec.
Wash time: 65 sec.

1.2.4 Autoanalyzer Calibration and Operation

After power is turned on to all units and the tubes are reconnected to the rollers, start wash water (DIW or sea water, depending on the samples to be analyzed) and mixed reagent flowing through the autoanalyzer. Then flush the instrument for at least 10 minutes or until the baseline is stable, then press autozero. Load a few vials of high standard (S5) to be analyzed until 60% fl scale is achieved. The computer table is then created with the samples to be loaded. When the samples are loaded, press
Alt-1, wait 20 seconds, then press start on the sampler.

Each run of the autoanalyzer begins with the running of a SYNC cup (S5) then a wash water blank. A complete set of blanks and the 5 working standards described in Table 1 are then run. Following the standard curve is a carryover (which is the low standard S1), a low standard (S1), and a check cal (S3). Additionally, a blank sample and an S4 are placed after every 10 sample vials in an autoanalyzer run to monitor baseline and intra-run calibration drift. At the end of the run, an S5 cup is run again.
Samples that are out-of-range of the standard curve are diluted and reanalyzed.

1.2.5 Autoanalyzer Shutdown

The following reagents are needed:

Sodium hydroxide (NaOH), 1 N: 40 g of sodium hydroxide is added to 1 L of DIW.

Hydrochloric acid (HCl), 10% v/v: 100 ml of conc. hydrochloric acid in 1 L total volume.

After the last samples are analyzed, the instrument should be flushed with fresh wash water for 5 min, 1 N NaOH for 5 min, 10% HCl for 5 min, DIW for 5 min, and air for 5 min. Turn off the power at the power strip (heat bath remains on all the time). Remove the tubes from the rollers.

1.2.6 Autoanalyzer Preventative Maintenance

All spills are immediately cleaned up. Following solid sample analyses, NaOH and HCl are flushed through the tubing longer (10 min each) or are pushed through with a syringe for greater pressure. Any worn tubing is replaced immediately.

1.3 Autoanalyzer Calculations

Alpkem SoftPac Plus version 1.07 release 1.0 software is used. Absorbance is directly proportional to phosphorus concentration and is measured as peak height units. The peak heights on the output have been corrected by the software for carry over and baseline. The slope, intercept, and correlation coefficient are calculated by the software by first order regression. The value for any given sample is then calculated by (peak height units - intercept)/slope. The intercept will impart a false concentration to all the values (i.e. on the low scale, the calculation will result in blanks with 0 peak height units having around a 0.07 μM concentration). As the software is not able to automatically correct for this, the false blank value must then be subtracted from all results. The correction will be done in the data entry of the values.

2.0 QA/QC and Corrective Action

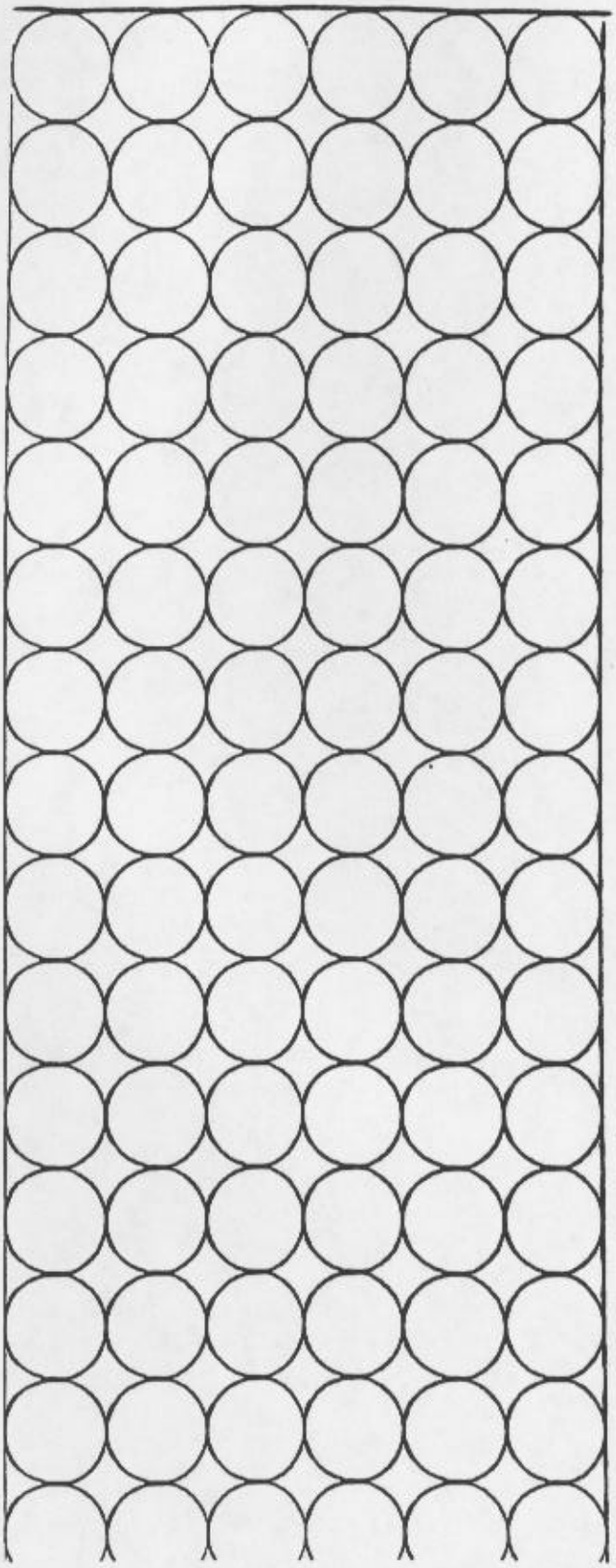
The standard curve should have a correlation coefficient ❒ 0.995 and the slope of the line should fall within ± 2 S.D. of the historic mean (the allowable range is listed on the inside cover of the logbook). A failing slope or correlation coefficient indicate the need for either preventative maintenance on the autoanalyzer or re-making of the standards.

To carry over (CO) should be ❒ 3%. The S4 continuing calibration checks should be ± 5% of the S4 value. Equipment blanks and method reagent blanks should be ❒ MDL (0.02 μM). QC check standards (SPEX or citrus leaf standards) should be ± 5% of the expected value. Matrix spikes should be ± 5% of the expected value. The precision of the 2 - 4 values produced for every sample should be ❒ 5%. Outliers (> 1 S.D. of the mean) can be discarded. Any samples and runs which do not meet these criteria may need to be reanalyzed. If a problem continues, preventative maintenance on the autoanalyzer may be necessary.

3.0 Total Phosphorus Forms

The sample preparation log sheets for water samples and solid (soil, sediment, and tissue) samples are shown in Figures 1 and 2. Figure 3 is a copy of a page from the instrument logbook.

Figure 1. Sample preparation log sheet for water samples.



TOTAL PHOSPHORUS - WATER
SERP 77 SOP v.1 Form v.3 (9/97)
Tray contents:

_____

_____

_____

_____

Prep. (100ml 0.17% MgSO, + 5 ml sample per vial) and put in oven (80°C):
(Date MgSO, was made _____)

_____ _____ _____
Date Time Init.

Taken out of oven:

_____ _____ _____
Date Time Init.

Put in muffle oven (550°C):

_____ _____ _____
Date Time Init.

Taken out of muffle oven:

_____ _____ _____
Date Time Init.

Did the pellet melt? _____

Add acid (5 ml HCl), shake, and put in oven (80°C):

Acid added: _____
(Date acid was made: _____)
(L=0.06N, M=0.12N, H=0.18N; If varied, put the letter in each circle)

_____ _____ _____
Date Time Init.

Taken out of oven:

_____ _____ _____
Date Time Init.

Second shake:

_____ _____ _____
Date Time Init.

Analyzed:

_____ _____ _____
Date Time Init.

Vials discarded:

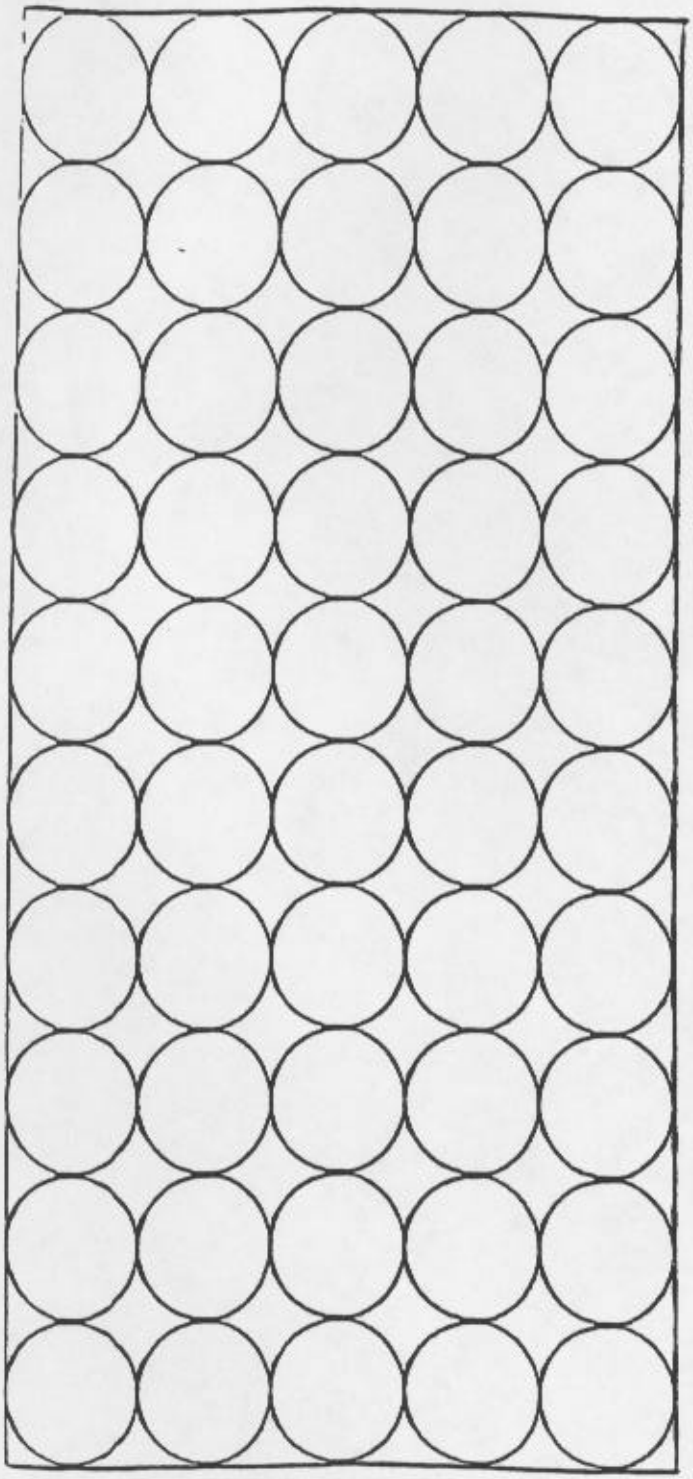
_____ _____ _____
Date Time Init.

Comments or problems:

_____

_____

Figure 2. Sample preparation log sheet for solid samples.



TOTAL PHOSPHORUS - SOLIDS
SERP-TP-SOP v.1 Form v.2 9/97)

Tray contents:

_____

_____

_____

_____

Prep. (200 µl 0.17N MgSO, + 1 ml DIW = ~25 mg sample per vial) and put in oven (80°C):
(Date MgSO, was made: _____)

_____ _____ _____
Date Time Init.

Taken out of oven:

_____ _____ _____
Date Time Init.

Put in muffle oven (550°C):

_____ _____ _____
Date Time Init.

Taken out of muffle oven:

_____ _____ _____
Date Time Init.

Did the pellet melt? _____

Add acid (10 ml 0.24N HCl), shake, and put in oven (80°C):
(Date acid was made: _____)

_____ _____ _____
Date Time Init.

Taken out of oven:

_____ _____ _____
Date Time Init.

Second shake:

_____ _____ _____
Date Time Init.

Analyzed:

_____ _____ _____
Date Time Init.

Vials discarded:

_____ _____ _____
Date Time Init.

Comments or problems:

_____

_____

_____

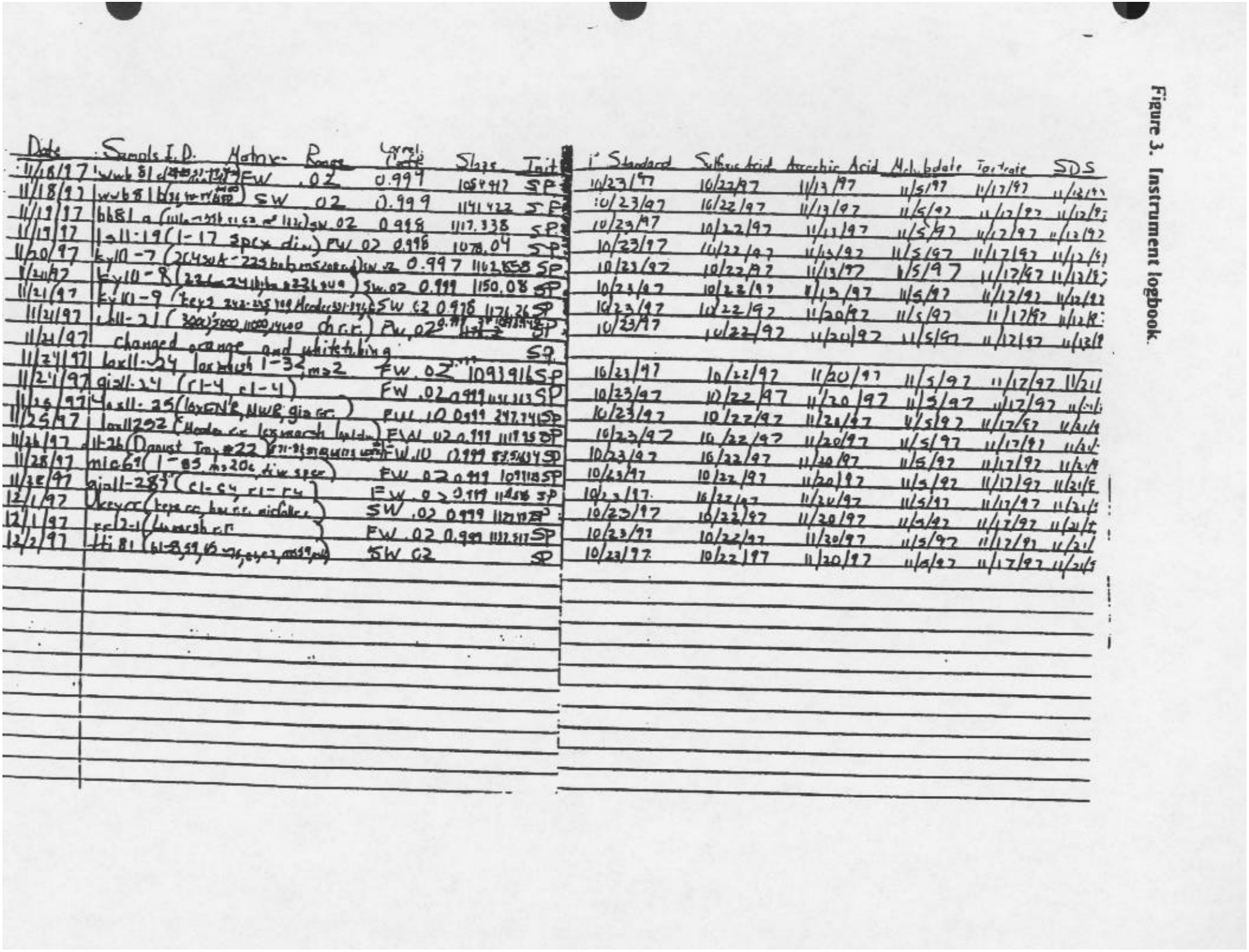


Figure 3. Instrument logbook.

Date	Sample I.D.	Matrix	Range	Correl. Coeff	Slope	Init	1° Standard	Sulfuric Acid	Ascorbic Acid	Molybdate	Tartrate	SDS
11/18/97	wwb 81 a([illegible])	FW	.02	0.999	1054.917	SP	10/23/97	10/22/97	11/13/97	11/5/97	11/17/97	11/12/9
11/18/97	wwb 81 b([illegible])	SW	.02	0.999	1141.422	SP	10/23/97	10/22/97	11/13/97	11/5/97	11/17/97	11/12/9
11/19/97	bb81 a ([illegible])	SW	.02	0.998	1117.338	SP	10/23/97	10/22/97	11/13/97	11/5/97	11/17/97	11/12/97
11/19/97	la11-19 (1-17 spcx dia)	FW	.02	0.998	1078.04	SP	10/23/97	10/22/97	11/13/97	11/5/97	11/17/97	11/12/9
11/20/97	ky11-7 ([illegible])	SW	.02	0.997	1162.858	SP	10/23/97	10/22/97	11/13/97	11/5/97	11/17/97	11/12/9
11/21/97	ky11-8 ([illegible])	SW	.02	0.999	1150.08	SP	10/23/97	10/22/97	11/13/97	11/5/97	11/17/97	11/12/9
11/21/97	ky11-9 (keys [illegible])	SW	.02	0.998	1176.26	SP	10/23/97	10/22/97	11/20/97	11/5/97	11/17/97	11/12/9
11/21/97	b11-21 (3000, 5000, 11000, 14400 ch. c.r.)	FW	.02	0.999	[illegible]	SP	10/23/97	10/22/97	11/20/97	11/5/97	11/17/97	11/12/9
11/24/97	changed orange and white tubing					SP						
11/24/97	lax11-24 laxmouth 1-32, ms2	FW	.02		1093.916	SP	10/23/97	10/22/97	11/20/97	11/5/97	11/17/97	11/21
11/24/97	gia11-24 (c1-4 e1-4)	FW	.02	0.999	[illegible]	SP	10/23/97	10/22/97	11/20/97	11/5/97	11/17/97	11/21
11/25/97	ya11-25 (laxCN'R, NWR, gia cr.)	FW	.10	0.999	247.741	SP	10/23/97	10/22/97	11/20/97	11/5/97	11/17/97	11/21
11/25/97	lax11292 (Hoeder cr. lexmarsh [illegible])	FW	.02	0.999	1119.35	SP	10/23/97	10/22/97	11/20/97	11/5/97	11/17/97	11/21
11/26/97	11-26 (Dqnust Tray #22 [illegible])	FW	.10	0.999	835.434	SP	10/23/97	10/22/97	11/20/97	11/5/97	11/17/97	11/21
11/28/97	mic69 (1-85, ms20c, tiw spec)	FW	.02	0.999	1091.18	SP	10/23/97	10/22/97	11/20/97	11/5/97	11/17/97	11/21
11/28/97	gia11-28 (c1-c4, r1-r4)	FW	.02	0.999	1148.8	SP	10/23/97	10/22/97	11/20/97	11/5/97	11/17/97	11/21
12/1/97	keyrc (keys cr. [illegible])	SW	.02	0.999	1121.172	SP	10/23/97	10/22/97	11/20/97	11/5/97	11/17/97	11/21
12/1/97	rc12-1 (lexmarsh cr.	FW	.02	0.999	1112.517	SP	10/23/97	10/22/97	11/20/97	11/5/97	11/17/97	11/21
12/2/97	Hi 81 ([illegible])	SW	.02			SP	10/23/97	10/22/97	11/20/97	11/5/97	11/17/97	11/21

Standard Operating Procedures for Laboratory and Field Nutrient Analysis

Southeast Environmental Research Program
Florida International University

Table of Contents

1.0 Introduction

This document describes SERP's standard operating procedures for the collection and analysis of nutrient samples. The purpose of the document is to provide a cookbook of procedures and instrument operation used on a daily basis by SERP employees. SERP typically collects and analyzes both fresh and saline surface waters for nutrient analyses. Soil/sediment and tissue (plant, fish) samples are processed and analyzed on a limited basis, and are also described in this document. Standard operating procedures used in SERP's mercury laboratory are presented in another document.

The document is organized into three main sections: 1) field procedures; 2) laboratory procedures; and 3) QA/QC samples. A detailed description of SERP's QA/QC procedures for the nutrient laboratory is presented in SERP's CompQAP. In general, most QA/QC procedures are overseen by SERP's QA Officer. Section three of this document describes only QA/QC samples that need to be processed by the laboratory technicians on a day-to-day basis.

2.0 Field Procedures

2.1 Equipment Preparation

Preceding a trip to the field, the personnel responsible for collection of the samples are required to ensure that everything is prepared for the expedition. This entails making sure that all sample containers are clean and properly labelled, and that all sampling and field measurement equipment are properly cleaned, charged and functioning within acceptable limits. Table 1 is an equipment checklist prepared for the sampling team.

TABLE 1
Field Equipment Checklist

Surface Water Sampling Equipment

1. Labeled and cleaned sample bottles (narrow-mouth plastic)
   - 60 mL (2 per site)
   - 125 mL (2 per site)
2. 140 mL clean plastic syringes
3. Microcentrifuge tubes
4. 2.5 cm in-line filter holders
5. 2.5 cm Whatman GF/F glass fiber filters
6. Filter forceps
7. 2-5 gallon plastic bucket
8. Niskin sampler

Field Measurement Equipment

1. S/C/T meter and probe
2. Dissolved oxygen meter and probe
3. Spare meters and probes
4. Salinity/Conductivity Check Standard
5. Light Sensor
6. CTD
7. pH meter and buffers
8. Instrument manuals

Sample Preservation

1. Acetone (90%)
2. Disposable polyethylene bottle
3. Ice
4. Coolers
5. DI water (1L) for Equipment Blanks.

Boat Equipment

1. GPS
2. Radio

Miscellaneous Equipment

1. Field data sheets
2. Pencils

3. Portable Phone
4. Life Vests
5. Depth Finder
6. Boat hook
7. Emergency Flares
8. Charts
9. Tool Box
10. Fire Extinguisher

3. Label tape and waterproof pens
4. DIW squeeze bottle (filled)

2.2 Water Sample Containers and Cleaning

SERP typically collects three types of water samples: 1) filtered soluble nutrient samples; 2) unfiltered total nutrient samples; and 3) chlorophyll-a samples. Clean 60 mL HDPE bottles are used for filtered nutrient samples. These bottles are cleaned by first rinsing three times with distilled water, then rinsing once with acetone to aid in drying and to remove organics. The bottles are shaken dry then capped. Clean 125 mL HDPE bottles are used for total nutrient samples. These bottles are cleaned by rinsing three times with distilled water. Acetone is not used to clean the 125 mL bottles for totals analysis. 1.8 mL microcentrifuge tubes are used to store the chlorophyll samples. These are used once then discarded. Filter holders and syringes used to collected the filtered and chlorophyll samples are rinsed well with distilled water. Syringes are allowed to air-dry in the dish drainer. Filter holders are placed right side up on a plastic tray and placed in an 80°F oven overnight to dry. Once dry, Whatman GF/F 25 mm filters are placed in each holder. At the end of each field day, all field instruments and coolers need to be wiped down with fresh water and dried before storing.

2.3 Water Sample Collection

Specific sampling locations are project specific. In general, surface water, sediment, and plant tissue samples are collected from a boat, helicopter, airboat, or by a SCUBA diver. To ensure collection of undisturbed samples, the boat is advanced toward a sampling station from the downstream direction. Surface water samples are collected as grab samples away from the outboard engine. Sediment or tissue samples are collected by SCUBA diver or by wading toward the sampling location from the downstream location. If surface water samples and sediment and/or tissue samples are collected at one location, then the surface water samples are collected prior to the collection of sediment or tissue samples. In areas of suspected high concentrations such as downgradient of a landfill, samples are collected in order of suspected low concentration to higher concentration.

2.3.1 Filtered Water Samples (for soluble nutrient determinations)

a. Use clean 140 mL polypropylene syringes to collect filtered surface water samples.
b. Place the syringes to draw water 10 cm below the surface of the water into the direction of water flow (if applicable).
c. Partially fill the syringe and rinse with sample water three times.
d. Fill the syringe with 120 or 140 mL of water.
e. Attach a filter holder (containing a new filter) to the end of the syringe and force about 10 mL of sample through the filter to rinse.

f. Use the remaining filtrate from the syringe to rinse a 60 mL HDPE sample bottle three times.
g. Fill syringe with sample water again (if necessary), and re-attach filter holder with filter.
h. Fill sample bottle to bottom of neck. Multiple syringe volumes may contribute to a single sample bottle.
i. Repeat steps *a* through *h* to collect a duplicate sample.
j. Check that the sample bottles are properly labelled.
k. Record date and time of sample collection in the field note book.
l. Place the samples in a cooler with ice.

2.3.2. Chlorophyll-a Samples

a. Using the syringe and filter from the previously collected filtered nutrient samples, force a known amount of air (30-40 mL) through the filter to aid in drying it.
b. Using forceps, transfer the filter to a 1.8 mL microcentrifuge tube.
c. Add 90% acetone to the top line of the tube (1.5 mL) with a disposable polyethylene pipet. Acetone extracts the chlorophyll from the cells collected on the filter.
d. Check that the tube is properly labelled.
e. Place the tube immediately in a dark bottle in the cooler with ice.
f. Record the volume of water filtered through the filter on the field data sheet (preferably 120 or 140 mL, but do not force too much water through that the filter breaks).
g. Repeat steps *a* through *f* to collected a duplicate sample.

2.3.3. Unfiltered Water Samples (for total nutrient determinations)

a. Unfiltered surface water samples are collected directly into clean 125 mL HDPE bottles.
b. Submerge the bottles neck first to about 10 cm below the surface of the water.
c. Invert the bottle with neck upright and pointing into the direction of water flow (if applicable).
d. Partially fill the bottle (at least 25 percent filled) cap, and shake, and pour the rinse water downstream of the sampling location.
e. Repeat this procedure two more times for a total of three rinses.
f. Fill the bottle to the neck and cap.
g. Repeat procedures *a* through *f* to collect a duplicate sample.
h. Check that the sample bottles are properly labelled.
i. Record date and time of sample collection in the field note book.
j. Place the samples in a cooler in the dark. Alkaline phosphatase activity is a microbiological parameter, therefore, these samples can not be stored on ice. Once alkaline phosphatase activity has been determined on the samples, the remaining sample for the inorganic parameters are stored in a refrigerator.

2.3.4 Samples Collected by Bucket or Niskin Sampler

When access to the surface water can not be made by boat or wading, such as from a bridge or side of canal, then a clean plastic bucket attached to a line is used to collect the surface water sample in bulk. This bucket is rinsed with sample water three times, with the rinse water poured downstream of the

sampling location, prior to collection of the sample. Sample bottles, syringes, and filters are then rinsed and filled from the water collected in the bucket following the procedures described above.
For water samples collected from a specific depth in the water column, a Niskin Sampler is used. A metered line is attached to the Niskin sampler and, while open, the sampler is lowered to the appropriate depth. The sampler is considered to be rinsed as it is lowered through the water column. Once at the desired depth, a weighted messenger is sent down the line to activate the closing of the sampler. Water in the sampler is extracted from a sample port at the bottom of the sampler. Water collected from one cast of the sampler must be used to rinse and fill all sample bottles and syringes.

2.4 Soil/Sediment/Tissue Sample Collection

The collection of soil/sediment/tissue samples are not commonly done, but are project specific. For collection of these samples, they are stored in a cooler with ice while in the field, and upon return to the laboratory they are stored in a freezer.

2.4.1 Surface Soil Samples

Surface soil samples are collected from the upper 10 cm of an undisturbed location. Surface detritus is removed prior to sample collection. The surface soil samples are collected with a stainless steel trowel, spade, PVC core, polycarbonate core or by hand and placed into plastic, wide-mouth specimen cups. The physical parameters of the soil, including color, moisture content, presence of biota, and texture are described in the field notebook, if required to satisfy the project objectives. The sample depth, date and time of sample collection, and the amount of sample (or subsamples) collected are also recorded in the field notebook. Roots may or may not be removed from the soil samples depending upon the project objectives.

Soil samples are homogenized either in the field or in the laboratory, depending upon the project objectives. If homogenized in the field, the soil sample is placed into a polypropylene mixing tray and homogenized by slicing, mixing, and remixing of the sample. The homogenized soil sample is then placed into a wide-mouth specimen cup and stored in a cooler in the dark for transport to the laboratory. In the laboratory, soil samples are homogenized by mixing the entire sample in a blender.

2.4.2 Subsurface Soil Samples

Subsurface soil samples are collected using either polycarbonate or PVC core tubes, pushed into the soil or sediment by hand by twisting the tube in a circular clock-wise and then in a counterclock-wise movement. The depth of the soil surface on the outside and on the inside of the core tube is measured and recorded to determine compaction.

Once the core is extracted, plastic caps or neoprene rubber stoppers are inserted and taped to the end of the tube to prevent slippage and spillage. The top direction of the core tube is marked on the tube along with the sample number and the tube is stored in an upright position during transport to the laboratory. In the laboratory, the soil or sediment is extracted from the core tube and using a stainless steel knife, a sample for analysis is collected from the center of the tube, away from the sides. The physical characteristics of the soil are described in the field notebook, along with the approximate

amount of sample (or subsample collected).

In general, soil sample compositing or splitting in the field is not preferred due to potential contamination concerns; the collection of duplicate samples in the field by collecting soil from the same sample source and homogenization of the samples in the laboratory with a blender, is preferred. If samples are to be homogenized in the field, then the samples will be extracted from the core tube onto a polypropylene tray and mixed with a stainless steel or Teflon spatula. The homogenized samples are then placed into plastic, wide-mouth specimen cups and stored in a cooler in the dark for transport to the laboratory.

2.4.3 Sediment Sample Collection

Sediment is collected using either polycarbonate or PVC core tubes or with an Ekman Dredge. The sediment sample is removed from the tubes or dredge and placed in a polypropylene tray. A stainless steel knife is used to collect a section of soil from near the center of the sample container. These samples may be homogenized in the field by mixing with a spatula or homogenized in the laboratory using a blender. Samples are stored in plastic wide-mouth specimen cups and stored in a cooler in the dark for transport to the laboratory. The amount of sediment collected, all equipment used, the method of homogenization, and the amount of sample stored are documented in the field notebook.

2.4.4 Tissue Sample Collection

Plant tissue samples are collected by gathering the plants by hand and placed into plastic bags. The plant samples are kept in a cooler on ice until transported to the laboratory.

2.5 Field Measurements

SERP typically measures temperature, salinity/conductivity, and dissolved oxygen at the surface and bottom of the water column at each station. For some projects, pH and light measurements are measured. All field measurements are taken contemporaneously with the sample collection to ensure direct correlation of laboratory results with field measurements. The water depth is determined from either a depth finder on the boat or from a weighted, non-stretch line that is marked in 10 cm increments. The calibration of all field instruments needs to be checked at the beginning of each day, after every four hours of operation, and at the end of each day. Instrument performance at each calibration check needs to be recorded on the field instrument calibration sheet (see attached). All instruments need to be fully charged overnight prior to their use in the field.

A new Field Data Sheet has been prepared (see attached). This sheet must be copied onto bond-water resistent paper and put onto a clipboard to take to the field. The sheets may be filled out in pencil; however, errors are not allowed to be erased. If an error is made, corrections must be made by drawing a single line through the error and entering the corrected information next to the error, then initialling.

2.5.1 Field Instrument Corrective Actions

A primary and backup meter and probe for each field instrument are brought on every sampling event. If both the primary and backup field instruments fail during the trip, then the sampling event should be discontinued until proper functioning equipment can be obtained. Salinity measurements are an exception to this rule. If the S/C/T meter is not functioning properly, the D.O. meter can still be used in the following manner. Adjust the D.O. meter to a salinity of zero. Record the temperature and D.O. for each station on the Field Data Sheet, noting which stations the D.O. was measured using a salinity of zero. Note the malfunction of the S/C/T meter on the instrument calibration sheet. Back at the laboratory, use a functioning salinity meter and probe and record the salinity of sample remaining in the total nutrient bottle on the field instrument sheet. The D.O. of each sample will then be corrected for the appropriate salinity.

2.5.2 Salinity/Conductivity/Temperature

Salinity and/or conductance is checked daily with a solution of known salinity or conductance, while temperature is checked daily against an NIST thermometer. The S/C/T meter probe and the NIST thermometer is inserted into 50 - 100 mL of the salinity or conductance standard. A salinity reading within 5% of the standard value, and a temperature within 0.1 degrees are considered acceptable. If Sargasso Seawater is used as the salinity standard, a value of 36.1 should be obtained, but values between 34.3 and 37.9 ppt are considered acceptable. Values outside these acceptance criteria will require the unit to be factory calibrated and the QA Officer or Dr. Ron Jones needs to be notified.

Surface temperature and salinity/conductivity is measured by submersing the probe of the salinity/conductivity/temperature (SCT) meter 10 cm under water. After the digital readout stabilizes (less than 5 minutes), temperature is recorded in °C and salinity is recorded in parts per thousand (ppt). Conductivity is recorded in units of μmhos/cm. The probe is then lowered to 10 cm from the bottom of the water column. After the digital readout stabilizes, the bottom parameters are recorded in the field notebook. Rinse the probe with DIW between stations, and shut the instrument off.

2.5.3 Dissolved Oxygen

The probe of the Orion model 840 Dissolved Oxygen meter is continuously polarized when attached to the meter; if it has been disconnected for over 1 hr, it requires 50 min to repolarize. No readings or calibration should be attempted within 50 min of connecting the probe. The calibration procedure is as follows:

1. Saturate the sponge in the calibration sleeve with deionized water and wait 50 min for equilibration.
2. Switch the meter on.
3. Depress and hold the Mode Key Pad until the display cursor is at Cal.
4. Depress quickly and release the Mode Key Pad. The display will show three dashes (---) and the slope of the electrode/membrane system. A slope reading between 0.7 and 1.2 is considered acceptable.

If calibration can not be properly obtained, take the probe out of the sleeve and look at the membrane on the bottom. Check that there are no air bubbles and that the silver ring is silver color and the gold

cathode is gold in color. If air bubbles are present, remove the cap at the bottom of the probe, fill the cap with new electrolyte solution and replace making sure there are no air bubbles. If the membrane is damaged or loose, replace the entire membrane cap. If the silver ring and/or gold cathode is tarnished, scape them carefully with a small glass fiber brush. Soak for two hours in cleaning solution, then for two more hours in distilled water. Any time that the membrane cap is removed from the electrode, a minimum of 50 min is required for the instrument to repolarize prior to use.

To take D.O. readings in the field:

1. Remove the sleeve from the probe. Be sure not to drop the sleeve in the water.
2. Submerse the probe 10 cm beneath the water surface.
3. Use the Mode Key Pad to select the Cal mode.
4. Adjust the salinity display with the up and down arrow keys to match the previously-measured station salinity.
5. Use the Mode Key Pad to select readings in mg/l.
6. Gently stir the probe until a constant reading is obtained.
7. The probe should be rinsed with DIW between each station, and the instrument can be turned off between stations.

2.5.4 Light Measurements

Light measurements are made utilizing two-4□ sensors mounted on a PVC pipe and attached by cables to a meter. The distance between the sensors can be adjusted to 0.5 m or 1.0 m. The 1.0 m distance is prefered if the depth allows. Calibration of the instrument should be as follows:

1. Plug the cables from the sensors into the meter (LI-1000). Check that the numbered tags on the cables match the channels of the meter, and that the Sensor probe # matches the # on the meter.
2. Turn the meter on, and wait for the display. Hit the Channel button. It should read MA for math, if not, continue pressing the Channel button until MA is displayed. The number following MA, ending in the letters PC, is the ratio of light at the bottom sensor to the top sensor. This is the number that you will be recording.
3. Check the calibration of the instrument by holding the PVC pipe with the sensors in a vertical position over a non-reflective surface such as still water, pavement or grass (not a white boat bottom or concrete dock). Record the instrument calibration reading on the instrument calibration check sheet.
4. The calibration reading should be between 0.98 to 1.02 in air over a non-reflective surface.

If the calibration is within this range, check that the multiplier for each sensor is correct:

1. Hit the CFG key once.
2. Using the ENTER key scroll through the options for light=1 until MULT= is obtained. Enter the negative multiplier number for sensor 1 in water. The multiplier number should be on the tag attached to each sensor.
3. Hit enter and scroll through the options for light=2 until MULT= is obtained. Enter the multiplier number for sensor 2 in water.

4. Hit the CHAN key until MA is displayed. Check that the calibration reading is between 0.98 and 1.02.

If the calibration is still outside this range, instrument readings need to be made according to the following procedures:

1. At each station, determine the depth with the sounding line. If the depth is greater than 1m, adjust the sensors to be 1m apart. Record this distance as z on the data sheet.
2. Stand on the sunny side of the boat. Be careful when advancing to a station, that sediment is not stirred-up into the water column. If necessary, move the boat to a sunny, undisturbed location.
3. Turn the instrument on. Hold the PVC pipe with the sensors in a vertical position to ensure that the distance between the sensors is indeed 1m (or 0.5m, if necessary). Submerge the sensors so that the top sensor is completely submerged. Be careful not to hit the sediment bottom and stir-up mud, as this will invalidate the reading. Also check that the bottom sensor is not covered with seagrass or algae.
4. If the instrument calibration was within the excepted range, then the ratio I_z/I_o can be read directly from the meter on the MA (math channel).
5. If the instrument is out of calibration, readings can not be made using the MA (math) channel. Instead, the incidence of light needs to be recorded for each sensor individually.
6. Depress the channel button until 1A appears. Record reading for the first channel on the data sheet. Press the Channel button again until 2A appears, and record the reading for the second channel. Minimize the time between recording the readings between the channels.
7. Turn the instrument off between readings.

2.5.5 pH

An automatic temperature compensation (ATC) probe on the pH meter adjusts the pH reading for temperature differences between standards and samples. The pH meter/probe is calibrated using a two-point calibration as follows:

1. Choose pH 0.01 mode.
2. Rinse probes (pH combination and ATC) in DIW. Blot dry. Rinse with 2 mL of pH 7.00 buffer. Immerse probes in pH 7.00 buffer.
3. Press Cal button. The meter will display ".1." and the pH value of the buffer; the meter automatically recognizes the pH of the buffer solution. When pH stabilizes, press Enter. The display will freeze for 3 seconds, and then display ".2.".
4. Rinse probes in DIW. Blot dry. Rinse with 2 mL of pH 10.00 buffer. Immerse probes in pH 10.00 buffer.
5. Wait for pH display to stabilize, and press Enter. Display now will say "PH" and be ready for sample measurement.
6. Rinse probe in DIW, place probe in pH 7.00 buffer, and check that pH meter reading is within 0.05 pH units.

The response of the pH meter is checked with the pH 7.00 buffer after 4 hours of use and at the end of each use. If the response is outside 0.05 pH units, the two-point calibration is repeated. If instrument calibration can still not be achieved, then the instrument needs to be factory calibrated. pH is determined at each station by collecting a sample of surface water in a clean, 400 mL polyethylene beaker after it is rinsed three times with sample water. The pH probe and ATC probe are submersed in the beaker, and the pH is recorded in the field note book. Successive aliquots of surface water are collected until the pH of three successive aliquot agrees within 0.02 pH units.

2.5.6 SEA-BIRD CTD (Model: SEACAT SBE 19-03)

Field measurements determined with the CTD include temperature, salinity, dissolved oxygen, photosynthetic active radiation, turbidity, and depth. These measurements will be made using the SEA-BIRD CTD in profile mode. At the beginning of every day prior to going out in the boat, set up the CTD according to the following procedures:

1. Plug the computer cable into the CTD by removing the rubber waterproof cap first (do not lose this) and lining up the fat prong with the fat hole.
2. Plug the other end of the computer cable into the back of the computer.
3. The CTD should be turned off. Check this.
4. Turn on the computer. After the C:\ prompt type **cd\seasoft**. Once in the seasoft directory type **TERM19**. Any time that you ever want the SEA-BIRD to talk to the computer, you have to be in the TERM19 program.
5. Once in TERM19, type **DS**. This checks the status of the instrument. Check that the instrument is in the PROFILE mode. If not, then type **MP**. Check that the pump delay is on 45 seconds. If not, type **SP** and set minimum conductivity to 2500 and pump delay at 45 seconds. Check that the time and date are correct. If not, type **ST** and set the current date and time.
6. Check that the battery charge is 7.0 volts or greater. If less than 7.0 volts, then the batteries need to be changed. Six D cell batteries are needed. Read the instrument manual on how to change the batteries.
7. Once you are comfortable with the instrument settings, and there is no data in the CTD that you want to save, press **IL** to initialize logging. The instrument will ask if you are sure. Initialize logging will erase all of the stored data in the instrument's memory, so do not initialize logging if there is data you still want to retrieve. If you are sure you want to initialize logging, type **Y**, then control **Y**.
8. Type **QS** for quiescence mode. This is an important step. Without this step the CTD will not record data. If you do not want to erase the data in the CTD memory, but just continue saving casts, then do not do the initialize logging step, just go into the quiescence mode.
9. Once in QS mode, quit TERM 19.

To take measurements in the field you have two options. You can allow the CTD to record data internally, and/or you can see the real time data by taking the computer out on the boat with you. If you don't have the computer, then be sure that the computer cable cap is secured in place prior to lowering the instrument in the water. Simply turning the magnetic switch on and off will make the

instrument record each cast separately. The data can then be downloaded at the end of the day. If you have the computer on board, then connect the long computer cable to the CTD and computer and proceed as follows:

10. At the C:\seasoft prompt type **Seasave**.
11. Hit **Acquire Real Time Data**
12. Check that the data is saved to the appropriate directory.
13. Hit **F10** to acquire real time data.
14. Fill in header information, then hit escape to quit.
15. The computer screen should read "Ready to turn CTD on".

To make a cast, be sure to do the following:

1. Confirm the instrument is properly secured with a line. For deep stations, you may want to have the end of the line tied to the boat. Do not use the computer cable as the support line for the CTD.
2. Remove the protective tubing from the conductivity cell, and don't lose this. Plug the tubing connecting the DO probe to the conductivity cell.
3. Turn the instrument ON by flipping the magnetic switch on the side of the instrument.
4. Lower the instrument to just below the surface of the water (approx. 0.5m). Keep the instrument there until all of the bubbles have escaped the instrument. This allows the instrument to equilibrate with the water temperature as well as compensates for the 45 sec pump delay.
5. After 45 sec, lower the instrument to the bottom of the station, then retrieve. The optimum speed of lowering is 1m per second, however, the pump on the instrument helps to compensate for differences in lowering speed.
6. Once the instrument is on the boat, turn it OFF.
7. Follow steps 10 through 15 above to set up the computer for the next station.

At the end of each day, the data must be downloaded from the instrument to the computer.

1. Rinse the entire instrument with fresh water. Rinse the conductivity cell well with deionized water. Put fresh DIW in the conductivity cell tubing and replace.
2. Plug the computer cable into the SEA-BIRD CTD and into the back of the computer.
3. Type **CD\SEASOFT**, then **TERM19.**
3. Type **DS** again to check the status of the instrument. This in an important step to establish the connection between the computer and the instrument.
4. Type **DH**. The SEA-BIRD will then begin to roll through each cast starting with 00.
5. Press the **F9** Key to upload the data. Each cast will be saved separately under a common name. Only six characters are allowed for a file name and the program automatically attaches two characters to the end of the name, therefore, you must limit you file names to four characters. Each cast will be saved in sequential order such as XXXX00, XXXX01, XXXX02, etc. For each cast, header information will be requested such as cast number, lat, long, notes, etc. Fill out this information for each cast.

6. Once all of the data is uploaded, backup up the data on a disk.

3.0 Laboratory Procedures

3.1 DIW and Glassware Cleaning

Two types of analyte-free water are produced in the laboratory: deionized-distilled water and double-deionized water. In general, the distilled water is used for washing equipment and glassware, while the deionized water is used as reagent water. Tap water is first deionized using a Culligan system containing activated carbon and 2-mixed bed ion exchange beds followed by filtering through a 0.45 µm polypropylene filter cartridge. The water is then either distilled through a Corning Mega-Pure 11 Liter Automatic Water Still to produce the deionized-distilled water or further deionized with a Barnsted model D8911 HN Ultrapure mixed bed deionization cartridge to produce the double-deionized water. Both types of water have proven to be analyte-free for the nutrients analyzed. The quality of this water is frequently checked with laboratory method and field equipment blanks.

To produce the distilled and deionized water be sure that the water level in the Mega-Pure water system is between the two yellow lines. Turn the water on by pressing the WATER switch, then the OPERATE switch. When the yellow indicator light goes out, Culligan needs to be called to change the cartridges. Dr. Jones needs to be notified whenever the cartridges need to be changed.

3.2 Inorganic Filtered Nutrients

Analysis for inorganic filtered nutrients (ammonium, nitrite, nitrate, and soluble reactive phosphorus) are simultaneously performed by wet chemical analysis using a four-channel Alpkem RFA-300 (Rapid Flow Analyzer) Nutrient Analyzer (Alpkem Corp., Clackamas, OR) following the procedure for each inorganic nutrient as suggested by the Alpkem Corporation, and modified for optimum conditions in our laboratory. Samples for inorganic nutrient determination are filtered in the field. These samples should be analyzed within 48 hours of sample collection. If they are going to be analyzed within 48 hours, then the samples can be stored in a refrigerator. If, by some chance, the samples cannot be analyzed within 48 hours, than they can be kept frozen in a freezer. Before freezing, approximately one-fourth of the water needs to be empty from the bottle to allow for expansion during freezing. Prior to running, the samples need to be brought to room temperature slowly with cold water and shaken well.

In the laboratory, sample bottles are opened, and 3.0 mL subsamples are transferred into sample-rinsed autoanalyzer cups. Samples are run in duplicate. A sampling time of 35 seconds, followed by a 45 second wash is used. Samples that are out-of-range of the standard curve are either automatically diluted and re-run by the autoanalyzer, or may be diluted by hand.

3.2.1 Autoanalyzer Methods

The indophenol blue method for ammonium is used. Ammonium reacts with alkaline phenol and hypochlorite to form indophenol. Sodium nitroferricyanide intensifies the blue color. Nitrite is determined as an azo dye formed by the reaction of nitrite with sulfanilamide and subsequent coupling with N-1-naphthylethylenediamine (NED). Nitrate is determined by the quantitative reduction of nitrate to nitrite using an activated cadmium column, and then determination of nitrite as described

above. The nitrite concentration before reduction is subtracted from the nitrite concentration after reduction to give nitrate concentration. Soluble reactive phosphate is determined by reacting phosphate with molybdenum (IV) and antimony (III) in an acid medium to form a phosphoantimonyl-molybdenum complex; this complex is reduced with ascorbic acid to form a colored dye.

3.2.2 Autoanalyzer Reagents

All reagents are made with the high reagent-grade chemicals dissolved in double-deionized water.

Blank and wash water: The salinity of the blank water and the samples effect the shape of the peaks from the autoanalyzer, therefore we match the salinity of the blank and wash water to the salinity of the samples. Samples with salinities of 15 ppt or less are run with nutrient-free DIW. Samples with salinities greater than 15 ppt are run with nutrient-free seawater acidified with sulfuric acid (250μL/1000mL). The nutrient-free seawater is obtained from the Sargasso Sea and stored in carboys fitted with ammonia traps. Blank and wash water are pumped directly from these carboys to the autoanalyzer.

Ammonium reagents: Four separate reagents are required by the autoanalyzer for ammonium analysis: complexing reagent, alkaline phenol, hypochlorite and, nitroferricyanide.

Complexing reagent: 25g sodium citrate is dissolved in 500 mL of DIW. 0.5 mL of 10 N sodium hydroxide is then added.

Alkaline phenol: 5 mL of 10 N sodium hydroxide and 10 mL 20% phenol in ethanol are added to 100 mL of DIW. Store this reagent in an amber bottle where it is stable at room temperature for a month.

Hypochlorite: 10 mL fresh hypochlorite is added to 100 mL DIW. This reagent is not stable and must be made daily.

Nitroferricyanide: 0.25g sodium nitroferricyanide is dissolved in 500 mL DIW. Store in an amber bottle where it is stable at room temperature for a month.

Nitrite reagents: Two reagents are required for nitrite analysis: sulfanilamide and N-1-naphthylethylenediamine.

Sulfanilamide: Cautiously add 100 mL conc. hydrochloric acid to 700 mL of DIW. Dissolve 10 g sulfanilamide. Dilute to 1 L total volume. Store the solution at room temperature or the refrigerator.

N-1-naphthylethylenediamine (NED): Dissolve 1 g of NED in 1000 mL DIW. Store the solution in a dark bottle in the refrigerator.

Nitrate reagents: Nitrate determination requires the reagents listed above for nitrite, plus an imidazole buffer and cupric sulfate.

Imidazole buffer: 6.81 g imidazole is dissolved in 1 L DIW. The pH of this solution is adjusted to 7.5 by the addition of hydrochloric acid. Store in a tightly sealed bottle at room temperature.

Cupric sulfate: 0.3125 g of cupric sulfate is dissolved in 125 mL DIW. This solution can be stored indefinitely at room temperature.

Soluble reactive phosphate reagents: This requires four reagents, three of which are mixed just prior to the analysis to make a working reagent. The components of the mixed reagent are antimony potassium tartrate, ammonium molybdate, and sulfuric acid. The fourth reagent is ascorbic acid.

Antimony potassium tartrate: 0.75g antimony potassium tartrate is dissolved in 250 mL DIW

Ammonium molybdate: 6.667g ammonium molybdate is dissolved in 500 mL DIW. Do not refrigerate.

Sulfuric acid solution: 140 mL conc. sulfuric acid is added to 900 mL DIW

Ascorbic acid: 6.0g ascorbic acid is dissolved in 200 mL acetone and 200 mL DIW

Working mixed reagent: Combine 50 mL sulfuric acid solution, 5 mL antimony potassium tartrate solution, and 45 mL ammonium molybdate.

3.2.3 Autoanalyzer Standards

Primary standards are made by dissolving anhydrous salts of the analyte of interest in DIW, and preserving the standard solutions with chloroform. All primary standards must be made using grade A flasks and stored in the glass bottles marked for these standards. All primary standards must be made up on at least a quarterly basis. Once made, the standard bottles need to be dated and initialed. Prior to discarding the old standards, both the new and old standards should be run on the RFA. The new standards should be within 5% of the old values. Once this is confirmed, then the old standards can be discarded.

A combined mixed standard is made by combining the primary standards of nitrate, ammonium, and phosphate in one solution. We are having a hard time maintaining the concentration of nitrite in the mixed standard, therefore, the nitrite primary standard is added to the mixed standard on a daily basis in a ratio of 4 mL of the mixed standard with 1 mL of the nitrite primary standard. This new mixed standard is then diluted to give working standards that bracket the range of most of the samples. Samples that are more concentrated than the highest standards are diluted.

Ammonium Primary Standard: Dissolve 0.33035 g ammonium sulfate in 1000 mL DIW. Add 2 mL chloroform. Final concentration: 5.0 μmoles/mL.

Nitrite Primary Standard: Dissolve 0.0690 g sodium nitrite in 1000 mL DIW. Add 2 mL

chloroform. Final concentration: 1.0 µmoles/mL.

Nitrate Primary Standard: Dissolve 1.020 g potassium nitrate in 1000 mL DIW. Add 2 mL chloroform. Final concentration: 10.0 µmoles/mL.

Phosphate Primary Standard: Dissolve 0.1360 g potassium dihydrogen phosphate in 1000 mL DIW. Add 2 mL chloroform. Final concentration: 1.0 µmoles/mL.

Mixed Standard: Combine 25 mL ammonium primary standard, 10 mL nitrate primary standard, 25 mL phosphate primary standard and 20 mL DIW.

Working Standards: Mix 4 mL of the mixed standard with 1 mL of the nitrite primary standard. Then dilute this new mixed standard according to Table 2 to produce the working standards.

Efficiency Standard: Mix 80 µL of the nitrate primary standard in 100 mL of DIW.

Table 2. Working standards for inorganic nutrient determination on the Alpkem RFA 300 autoanalyzer. Mixed standard volume refers to the amount of mixed standard added to 100 mL nutrient-free seawater (seawater samples) or DIW (freshwater samples). SRP is soluble reactive phosphate. N+N is nitrate (NO_3^-) + nitrite (NO_2^-).

Working Standard	Volume Mixed Standard (µL)	Working Standard Concentration (µmoles/L)				
		SRP	NH_4^+	NO_2^-	NO_3^-	N+N
BLANK	0	0	0	0	0	0
S1	100	0.25	1.25	0.20	1.00	1.20
S2	200	0.50	2.50	0.40	2.00	2.40
S3	400	1.00	5.00	0.80	4.00	4.80
S4	600	1.50	7.50	1.20	6.00	7.20
S5	800	2.00	10.00	1.60	8.00	9.60

3.2.4 Autoanalyzer Calibration

Prior to starting a run on the autoanalyzer, the injection needle is placed into the S5 standard and allowed to run through the instrument. The percent full scale of the high standard on each channel is recorded and compared to the previous day's run. The percent full scale should not change greater than 5% on a daily basis. Changes greater than 5% may indicate that either the standards were not prepared correctly, or that the instrument is in need of an overhaul.

Each run of the autoanalyzer begins with the running of a SYNC cup then a wash water blank. A complete set of blanks and the 5 working standards described in Table 5.2 are then run. Following the standard curve is a carryover (which is the low standard S1), then an S1 cup is run again. A check cal (S3) and an efficiency cup follow. Additionally, a blank sample and an S4 are run every 10 analyses in an autoanalyzer run to monitor baseline and intra-run calibration drift. At the end of the run, an S5 cup is run again. Any drift in the sensitivity of the autoanalyzer during the run is detected by this drift check, and corrections are made to account for any drift. A log book is kept to monitor the calibration curve parameters. Substantial changes in the curves indicate the need for either preventative maintenance on the autoanalyzer or re-making of the standards.

3.2.5 Autoanalyzer Preventative Maintenance

After every run of the autoanalyzer, the inside of the tubing is washed with a 10 min flow of detergent (Kem-Wash), followed by a 10 min flow of 10% (v/v) hydrochloric acid, followed by a 10 min flow of DIW. The system is then pumped dry. The peristaltic pump rollers are cleaned using ethanol-dipped cotton swabs. All spills are immediately cleaned up. Any worn tubing is replaced immediately. An hour meter attached to the instrument keeps track of the use time; after every 200 hr of use, the instrument receives a thorough overhaul. For each overhaul, all tubing is changed, the pump rollers are reconditioned, and the filters are inspected and changed if necessary.

3.3 Total Phosphorus

For the determination of total phosphorus in water, soil, sediment, and tissue samples, SERP does not use the typical ammonium persulfate digestion because of the explosive hazards and special handling requirements associated with the use of this chemical. Instead, SERP uses a modification of the sample preparation methods described by Solórzano and Sharp (1980. *Determination of total dissolved phosphorus and particulate phosphorus in natural waters.* Limnol. Oceanogr., 25(4), pp. 754-758 (see attached). Total phosphorus is determined in water, soil, sediment, and tissue samples by oxidizing and hydrolyzing all of the phosphorus-containing compounds in a sample to soluble reactive phosphate, and determining the soluble reactive phosphorus concentration by the same autoanalyzer method described above. SERP has a separate autoanalyzer used for the analysis of total phosphorus.

3.3.1 Total Phosphorus Sample Preparation

Sample preparation for total phosphorus determination is done as soon as possible. Water samples should be prepared within 24 hours of sample collection, and are often prepared immediately upon return to the laboratory.

3.3.1.1 Water Samples

1. Prepare a tray with two or three 8 ml scintillation vials (without the aluminum-lined caps) per sample bottle. Add 100 µl of 0.17 N $MgSO_4$ to each vial using the Eppendorf pipet setting of 1. Fill out a Total Phosphorus Preparation Log Sheet with the placement and contents of the vials on the tray.
2. Add 5 mL of water sample from each total bottle into each of the vials.
3. Place tray in an 80°C oven and evaporate to dryness (usually overnight).
4. Ash the samples at 550°C in a muffle furnace for 3 hours and allow to cool overnight.
5. Hydrolyze each sample with the addition of 5 mL of hydrochloric acid. The normality of the acid is dependent on the salinity of the sample according to the following table:

Salinity	HCl concentration
0.0 - 15 ppt	0.06 N
16 - 35 ppt	0.12 N
36 - 55 ppt	0.18 N
56 - 80 ppt	0.24 N

6. Cap each sample tightly with polylined caps, shake using a vortexer, and put into an 80°C oven overnight. After removing from the oven, allow to cool and shake again.

3.3.1.2 Soils, Sediment and Tissue Samples

1. Dry sample in an 80°C oven for 2 days, then grind.
2. Add 25 mg of sample into 10 mL glass scintillation vial (remove aluminum lined caps). Prepare two vials per sample. Fill out a Total Phosphorus Preparation Log Sheet with the

placement and contents of the vials on the tray.

3. Add 200 µl of 0.17N $MgSO_4$ and 1 mL DIW to each sample vial. Then dry and ash the sample as described for water samples.
4. Place tray in an 80°C oven and evaporate to dryness (usually overnight).
5. Ash the samples at 550°C in a muffle furnace for 3 hours and allow to cool overnight.
6. Hydrolyze each sample with the addition of 10 mL of 0.24 N hydrochloric acid.
7. Cap each vial tightly with polylined caps and put in 80°C oven overnight.
8. Cool and shake, allow to stand overnight.
9. Analyze at a 1:10 dilution (200 µl of sample with 1800 µl DIW).

3.3.2 Total Phosphorus Reagents

The reagents used for total phosphorus are the same as those used for soluble reactive phosphorus presented in Section 3.2.2.

3.3.3 Total Phosphorus Standards

The primary standard for total phosphorus is the same as that used for soluble reactive phosphate.

Phosphate Primary Standard: Dissolve 0.1360 g potassium dihydrogen phosphate in 1000 mL DIW. Add 2 mL chloroform. Final concentration: 1.0 µmoles/mL.

Total P Secondary Standard for water samples: Put 20 µL of phosphate primary standard into 10 mL of DIW.

Working standards for water samples are prepared from the primary standard in DIW or Sargasso Seawater depending upon the salinity of the samples. For water samples with salinities of 15 ppt or less, standards are prepared in DIW. Sargasso Seawater is used for water samples with salinities greater than 15 ppt. The working standards are made to bracket the expected concentration of the samples as shown in Table 3. A log book is kept by the total phosphorus autoanalyzer. In the log book record the slope and correlation coefficient of the calibration curve, the concentration of the high standard, the number of samples run, the water matrix (freshwater or seawater) and the technician's name.

Table 3. Working standards for Total Phosphorus determination.

Working Standard	Volume of Primary Standard in 100 ml of DIW or Seawater	Phosphorus Standard Concentration (µM)
Low Standard Curve		
S1	10 µl	0.10
S2	50 µl	0.50
S3	100 µl	1.00
S4	150 µl	1.50
S5	200 µl	2.00
High Standard Curve		
S1	125 µl	1.25
S2	250 µl	2.50
S3	500 µl	5.00
S4	750 µl	7.50
S5	1000 µl	10.00
Standard Curve for Solid Samples		
S1	125 µl	3.87 µg/g
S2	250 µl	7.74 µg/g
S3	500 µl	15.48 µg/g
S4	750 µl	23.22 µg/g
S5	1000 µl	30.97 µg/g

3.3.4 Total Phosphorus Autoanalyzer Preventive Maintenance

The cleaning and preventive maintenance schedule for the total phosphorus autoanalyzer is the same as for the filtered nutrient autoanalyzer.

3.4 Total Nitrogen

An ANTEK Instruments, Inc. Model 7000N Nitrogen Analyzer is used to determine total nitrogen of 5 µl of a preserved water sample. The instrument is run according to the Installation/Operation/Service Manual provided by ANTEK Instruments, Inc., except that oxygen gas is used as a carrier gas instead of argon to promote complete recovery of the nitrogen in the water samples.

3.4.1 Total Nitrogen Sample Preparation

Water samples for total nitrogen should be processed within 24 hours of sample collection and is often performed immediately upon return of the samples to the laboratory. Water sample preparation for total nitrogen includes transferring 1.5 mL of water sample from the unfiltered sample bottle to a small glass vial. The sample is acidified with 10 µl of 3 N HCl, using an Eppendorf pipet on a setting of 1. The glass vials are sealed with a teflon-lined crimp cap, labelled, and stored in a refrigerator at 2°C until analyzed.

3.4.2 Total Nitrogen Reagents

ACS reagent-grade 3 N HCl is made by adding 125 mL of concentrated 12 N HCl into a 500 mL flask and diluting to the mark with DIW.

3.4.3 Total Nitrogen Standard

A primary standard is made by dissolving 0.3612 g anhydrous potassium nitrate in 100 mL DIW. This standard has a concentration of 0.5 mgN/mL. A working standard of 2.0 mg/l is made by adding 400 µl primary standard in 100 mL DIW. Vials of the 2.0 mg/l standard are prepared identical to samples. Specifically, 1.5 mL of this standard is placed into a glass autoanalyzer sample vial and acidified with 10 µl of 3 N HCl. Duplicate vials of this standard are run in triplicate prior to each run, after every 20 samples, and at the end of the run to check instrument calibration.

A one-point calibration curve is obtained for each instrument run by beginning the run with duplicate 2.0 mgN/l standards. Zero total nitrogen has a signal of zero. Intra-run drift in the calibration curve is monitored by insertion of a blank and a 2.0 mgN/l standard after every 20 samples and at the end of each run.

3.4.4 Total Nitrogen Preventive Maintenance

The maintenance schedule for the ANTEK instrument includes replacing the septa on the autosampler every 80 samples, and replacing the column septa every 40 samples. On a daily basis, the vacuum pressure needs to be monitored and should be at 25 inches of mercury. The combustion column should be changed as needed.

3.5 Total Organic Carbon

Total organic carbon samples are analyzed by hot-platinum catalyst combustion of the non-purgeable organic carbon in the sample to CO_2 on a Shimadzu TOC-5000 Total Organic Carbon Analyzer.

3.5.1 Total Organic Carbon Sample Preparation

Sample preparation includes pipetting 4 mL from each unfiltered sample bottle into glass sample vials. Glass sample vials are cleaned by soaking in DIW with RBS 35 Concentrate cleaning solution and autoclaving for 15 minutes. The vials are rinsed three times with DIW then put upside-down in a test tube rack in the drying oven until dry (usually overnight). In order to remove inorganic carbon while in the automatic sampler, the samples are acidified and then purged for 8 minutes with CO_2-free air prior to analysis.

3.5.2 Total Organic Carbon Reagents

25% Phosphoric Acid. Add 500 mL of 85% phosphoric acid to 1,500 mL of DIW. This reagent is put in the reagent bottle in the instrument.

3N Hydrochloric Acid. ACS reagent-grade 3 N HCl is made by adding 125 mL of concentrated 12 N HCl into a 500 mL flask and diluting to the mark with DIW. This reagent is used to acidify the samples while in the autosampler.

3.5.3 Total Organic Carbon Standards

A primary standard is made by dissolving 2.125 g reagent-grade potassium hydrogen phthalate in 100 mL zero-grade DIW. The concentration of this primary standard is 10,000 mgC/L. Working standards are prepared of concentrations 0, 5, 10, 20 and 50 mgC/L by adding 0, 20, 40, 80 and 200 µL of the primary standard to 40 mL aliquots of zero-grade DIW. A standard curve, is run at the beginning of every sample run. To monitor for intra-run drift, a blank and a high 10 mg/l standard is run in the middle and end of each run. Each run and the instrument's performance needs to be recorded in the instrument log book.

3.5.4 Total Organic Carbon Preventative Maintenance

The TOC instrument maintenance schedule includes checking the reagent level, the DIW level, and the gas level on a daily basis. Replace the tubing and the needles on a daily basis and change the column every 2000 samples. A detailed schedule of replacement parts and regeneration of catalyst is contained in the operator's manual for the instrument. This schedule should be followed.

3.6 Silica

3.7 Chlorophyll-a

An extractive fluorometric technique is used to determine chlorophyll-a concentration. Acetone extracts of suspended material collected on filters are excited with 435 nm light, and the fluorescent emission of light at 667 nm is measured using a Fluoro IV Spectrofluorometer (Gilford Instruments, Oberlin, Ohio). The amount of fluorescence is directly proportional to chlorophyll concentration as determined by a standard curve of chlorophyll prepared in 90% acetone solution.

3.7.1 Chlorophyll Sample Preparation

Samples are brought to the laboratory on the same day of collection and stored in the dark in a freezer at a temperature of -15°C. Samples should be kept in the freezer for a minimum of 48 hours to allow for complete extraction of the suspended material from the filters to the acetone. Analysis should be performed within 7 days of sample collection.

3.7.2 Chlorophyll Reagents

90% Acetone. In a large 1000 mL cylinder add 900 mL of acetone and 100 mL of DIW. Store in a glass 1000 mL bottle and seal with a teflon lined cap.

3.7.3 Chlorophyll Standards

Put the contents of 1 vial of 1 mg/L chlorophyll-a standard from Sigma Chemical Co. (St. Louis, Mo) into 200 mL of 90% acetone. Determine the exact concentration of this high standard on the spectrophotometer at a wavelength of 664 nm and using the equation given in Section 3.6.5. Make 5 to 7 standards from the high standard in a range from 0 - 250 µg/L. All of these standards need to be in 90% acetone. Analyze these standards on the spectrofluorometer according to the following procedures:

a. Turn on spectrofluorometer and allow to warm up for at least one hour.
b. Set excitation to 435 nm and emission to 667 nm (Hit 2 ENTER, 435 ENTER, 667 RETURN).
c. Place an empty, glass cuvette in slot 2 and hit calibration key.
d. After calibration, set high voltage to 700 (Hit 3.2 ENTER, 700 RETURN).
e. Set the response to 4 seconds (Hit 3.5 ENTER, 4 RETURN).
f. Place 90% acetone in a clean, glass cuvette in slot 2, check to see that it reads 0.0. If it doesn't, hit the AUTO ZERO button. If it is still not zero, clean the glass cuvette to be sure it is free of fingerprints.
g. Place another clean, glass cuvette in slot 1 and check to see that it reads zero. If it doesn't, hit AUTO ZERO again or clean the cuvette.
h. Suction off the 90% acetone from slot 1. Rinse the cuvette with acetone and suction off.
i. Run each of calibration standards as you would a sample. Specifically, mix 750 µL of

each standard with 2250 µL of 90% acetone in the cuvette in Slot 1. Rinse the cuvett with acetone between each standard.

j. Determine the slope of the calibration curve of relative fluorescence to chlorophyl concentration. Check that the linear correlation coefficient is ≥0.95.

3.7.4 Chlorophyll Sample Analysis

a. Remove samples from the freezer.
b. Push the filters down to the bottom of the centrifuge vials with a spatula.
c. Place the vials in the centrifuge and spin for 1 - 3 minutes.
d. Place 750 µL of the acetone extract from the first vial into the fluorometer glass cuvette in slot 1.
e. Add 2250 µL of 90% acetone to the cuvette and mix twice with a disposable pipet.
f. Close the hood of the instrument and allow the reading to equilibrate. Hit READ PRINT to record relative fluorescence.
g. Suction off the sample from the cuvette, rinse with acetone, suction again and repeat procedures *d - g* for each sample.

3.7.5 Chlorophyll Calculations

a. Determine the chlorophyll concentration in the high standard spectrophotometrically using the extinction coefficient of 87.67 (L*gm-1*cm-1) following equation by Jeffrey and Humphrey (1975):

$$\text{Chl-a } (\mu g/L) = (\text{absorbance}/87.67) * 1 \times 10^{-6}$$

b. Determine the chlorophyll concentration using the following equation:

$$\text{Chl-a } (\mu g/L) = \text{R.F.} * \text{slope} * (1.5 \text{ mL}/0.75 \text{ mL}) / \text{L filtered}$$

where, R.F. is the sample relative fluorescence; slope is the slope of the standard curve; 1.5 mL is the amount of acetone in the microcentrifuge used for extraction; 0.75 mL is the amount of the acetone extract removed from the vial and placed in the cuvette; and the L filtered is the amount of water filtered in the field (e.g. 120 or 140 ml = 0.12 or 0.14 L).

c. Calculate the mean chlorophyll concentration at each station from duplicate samples.

3.8 Alkaline Phosphatase Activity

The alkaline phosphatase activity (APA) assay measures the activity of alkaline phosphatase, an enzyme used by bacteria to mineralize phosphate from organic compounds. The assay is performed by adding a known concentration of an organic phosphate compound (methylfluorescein phosphate, or MFP) to an unfiltered water sample. Alkaline phosphatase in the water sample cleaves the phosphate from the MFP, leaving methylfluorescein (MF), a highly fluorescent compound. The concentration of MF at the end of the assay is proportional to the APA of the sample.

3.8.1 APA Sample Preparation

APA measurements are made within 12 to 24 h of sample collection. Duplicate 3 mL subsamples from each sample bottle are pipetted into disposable plastic cuvettes, and 30 μL of a MFP solution are added to each.

The fluorescence of these subsamples are immediately measured using a Gilford Fluoro IV Spectrofluorometer (excitation = 430 nm, emission = 507 nm) and recorded. Samples are placed in an incubator at 25□C for 2 hr. Fluorescence of all the samples is then measured again using the same excitation and emission wavelengths. The amount of MF produced in 2 h is quantified by comparison to a standard curve.

3.8.2 APA Reagents

Methylfluorescein Phosphate Solution: 52.55 mg of anhydrous 3-o-methylfluorescein phosphate is dissolved in 100 mL of 100 mM Tris buffer, pH = 8.7. Concentration of this stock solution is 1 mM.

Trizma Buffer: Dissolve 12.8 g of Trizma crystals, pH=8.7 in 1 L of DIW.

3.8.3 APA Standards and Calibration

A stock standard solution of methylfluorescein is diluted to make working standards that bracket the concentration of MF in the APA assays after 2 hr. Working standards are made up from standard stock solution and the fluorescence of the working standards is measured each day that analyses are performed.

Methylfluorescein Standard Stock Solution: Dissolve 0.0346 g methylfluorescein in 100 mL methanol. Concentration of this standard stock solution is 1 mM. This solution is kept in 1.8 mL centrifuge vials in the freezer. One vial is thawed each day prior to use.

Working Standards: 0, 3, 7.5, 15 and 30 μL of standard stock solution are diluted into 3.0 mL of Trizma buffer, to give standards of 0, 1, 2.5, 5 and 10 μM methylfluorescein.

3.8.4 APA Sample Analysis

a. Turn the machine on to warm up, preferably an hour.
b. Set the excitation and emission wavelengths (Hit 2 ENTER, 430 ENTER, 507 RETURN).
c. Set the response time to 4 seconds (Hit 3.5 ENTER, 4 RETURN).
d. Calibrate the machine with an existing cuvette in slot 2. Hit calibrate.
e. After calibration, set high voltage to 425 (Hit 3.2 ENTER, 425 RETURN).
f. Put in blank (Trizma buffer) and see if it reads zero. If not, zero the instrument by hitting AUTO BLANK.
g. Analyze each standard, pressing READ PRINT after each to record the relative fluorescence.

h. Prepare the samples by placing 3 mL of sample into each of two disposable plastic cuvettes. Put 30 µL of stock MFP into each sample cuvette and mix with a disposable pipet.

i. Insert each sample cuvette into slot 1, close the hood, and hit READ PRINT to record the relative fluroescence when the number stabilizes.

j. Place the sample cuvettes into an incubator at 25□C for 2 hr, then repeat step *i*.

3.9 Turbidity

Turbidity is often determined for unfiltered (total) samples immediately upon return to the laboratory. An HF Scientific Inc. model DRT-15 C turbidimeter is used. This instrument is portable and can be taken into the field for field determinations of turbidity. Turbidity needs to be done on the samples within 24 hours of samples collection. Therefore, if the samples can not be brought back to the lab within one day of sample collection, then the turbidimeter needs to be taken out into the field.

3.9.1 Turbidity Standards and Calibration

The instrument reads directly in Nephelometric Turbidity Units (NTUs). Calibration is done quarterly with a 3-point calibration by diluting a 4000 NTU stock Formazin solution. The following table summarizes the preparation of the calibration solutions.

Table 2. Turbidity Standards

Value	Pipet into a 200 mL volumetric flask
198 NTU	9.9 mL of 4000 NTU stock. Add DIW to mark.
19.8 NTU	20 mL of the 198 NTU dilution made above. Add DIW to mark.
2.0 NTU	2.0 mL of the 198 NTU dilution made above. Add DIW to mark.

For proper calibration of the unit, access to the trimpots on the right side of the instrument needs to be made. See Figure 2 in the instrument manual for location of the trimpots. Proceed with the instrument calibration as dictated in the instrument manual.

3.9.2 Turbidity Sample Analysis

Sample readings are made according to the following procedures:

a. Turn the instrument range knob to a scale of 20.

b. Place the 0.02 NTU standard in the sample well. Turn the cuvette to the lowest reading on the instrument.

c. Turn the reference adjust knob until a reading of 0.02 NTU is obtained.

d. Put the control standard in the sample well.

e. A reading of about 18 NTU should be obtained for this sample on both the 20 and 200 scales. Record this reading on turbidity data sheet.

f. Turbidity is done on sample water collected in the unfiltered (total) bottle.

g. Rinse the sample cuvette with a little of the sample water. Be sure to shake the sample bottle well, then pour the sample into the sample cuvette to about 75% full.

h. Wipe the outside of the cuvette well to be free of water and fingerprints.

i. Place the sample cuvette in the sample well, being sure that the reference point on the cuvette is lined up with the reference point on the instrument.

j. Record the sample reading on the 200 scale.

k. Discard the sample, rinse the cuvette with DIW, then repeat steps *g* through *k* for all of the samples.

4.0 QA/QC Samples

4.1 Equipment Blanks

These are labelled as C1, C2, and are often called control samples. Equipment blanks need to be prepared for all SFWMD funded projects including Florida Bay, Whitewater Bay, Biscayne Bay, Ten Thousand Islands, and Southwest Florida Shelf. One equipment blank needs to be prepared for every 20 samples. This blank is prepared in the field prior to sampling by pouring or rinsing deionized water on each piece of precleaned field sampling equipment. Equipment blanks for surface water samples are collected by pouring DIW into a syringe, through a filter if required for the analysis, then into the sample bottle. Since SERP generally does not collect samples from over 20 locations in a day, then an equipment blank should be prepared at the beginning of each sampling day.

4.2 Matrix Spike Samples (Labelled MS)

These samples are prepared for every sampling event, including Marsh, Miccosukee, Miller and Loxahatchee samples. One matrix spike is prepared for every 25 samples, usually by the QA Officer or by Pete Lorenzo. These samples are prepared by randomLy selecting one sample from the batch, splitting the sample into two duplicate samples, and spiking one of the duplicate samples with the RFA mixed standard at a ratio of 400 µL per every 100 mL of sample. For most of our sampling program, one totals bottle is collected at random from each sampling day. 50 mL of this sample is put into another totals bottle and spiked with 200 µl of the RFA mixed standard.

4.3 Continuing Calibration Check Samples (Labelled at B and S5)

A method blank and a high standard are run after the standard curve and after every 20 samples. These need to be run on every instrument during every run.

4.4 Quality Control Check Samples (Labelled as QCSW and QCFW)

These samples are the freshwater and seawater vials made up by
Dr. Jones and Pete. A seawater vial needs to be run each month with the Florida Bay samples. A freshwater vial needs to be run every time freshwater samples are run, including Miccosukee, Marsh and Loxahatchee samples. The vials should be broken with the contents (10 mL) used for as many parameters as possible. Try to use only one vial for all analyses (TP, TN, TOC, and nutrients). These do not need to be run in duplicate to spare the volume. If a vial is not used all in one day, the vial can be capped with parafilm and stored in the refrigerator until used. In addition to these vials, the QA officer may make up QC check samples periodically and submit them blind to the analysts.

4.5 Sample Shipping and Sample Chain of Custody

All samples that are either collected by SERP and sent to another laboratory, or samples that are collected by outside researchers and sent to SERP for analysis must be accompanied by a sample chain-of-custody form (see attached). Try and use the sample chain-of-custody form from the other laboratory or researcher as SERP as yet does not have its own chain-of-custody form. This form must include a list

of all samples sent or received and the analyses requested. A copy of the chain-of-custody form must be included in the project specific files, or will be kept by the QA officer.

4.6 Standard Receipt

When any chemicals or reagents are first received, they will be dated and initialed by the person unpacking them. In addition, when the chemical or reagent container is opened for the first time it is dated again and initialed by the opener. All primary standards need to be dated and initialed when made. Secondary standards that are made on a daily basis do not need to be dated or initialed. All primary standards need to be made up on a quarterly basis.

5.0 Forms

The following forms are attached:

a. Field Instrument Calibration Form

b. Field Data Sheet without light

c. Field Data Sheet with light

d. Turbidity Form

a) Field Instrument Calibration Form

Sampling Event	Date	Names	Comments

Instrument Name	Instrument Number	Probe Number	Time	Calibration Check

ata She. ...out light

Field Data Sheet

Event	Date	Names	Weather Conditions

	Station Name	Time	Depth	Sal.	Temp.	D.O.	Total Depth	Volume Filtered	Comments

ata Shee [redacted]ight

Field Data Sheet

Event	Date		Names				Weather Conditicns		
Station Name	Time	Depth	Sal.	Temp.	D.O.	Z(1;0.5)	Iz/Io	Volume Filtered	Comments

urbidity .orm

TURBIDITY

Date

Project

Sample nos.

Technician

ple #	Reading 1	Reading 2
ndard		

6.0 References

Jeffrey, S.W. and G.F. Humphrey. 1975. New Spectrophotometric Equations for Determining Chlorophylls a, b, c_1 and c_2 in Higher Plants, Algae and natural Phytoplankton. Biochem. Physiol. Pflanzen (BPP), Bd. 167, S. 191-194.

Solorzano L. and J.H. Sharp. 1980. Determination of Total dissolved Phosphorus and Particulate Phosphorus in Natural Waters. 25(4). pp. 754-758.

Hashimoto, S., K. Fujiwara, and Keiichiro F. 1985. Relationship Between Alkaline Phosphatase Activity and Orthophosphate in the Present Tokyo Bay. Environ. Sci. Health, A20(7), 781-809.

COMPREHENSIVE QUALITY ASSURANCE PLAN
Mercury Laboratory

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Ronald D. Jones, Ph.D. Date
SERP Director and Professor

_____ _____
Ruth B. Justiniano Date
SERP Quality Assurance Officer

_____ _____
Sylvia S. Labie Date
FDEP QA Officer

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3.0 Statement of Policy

The Southeast Environmental Research Program (SERP) is made up of university research professors and their staff from Florida International University (FIU). FIU is one of the nine State University System (SUS) universities and all SERP personnel are employees of the State of Florida. The goals of SERP are to advance scientific research, the understanding of biogeochemical processes, and to publish results in high quality refereed scientific publications. Pertinent to these goals, is the need to collect accurate, high quality, and reproducible data, which can only be obtained through strict internal and external quality assurance practices. SERP is committed to follow sound quality assurance/quality control (QA/QC) practices for the purposes of producing verifiable quality data.

The professors associated with SERP have been involved in monitoring surface water quality in Florida Bay, Biscayne Bay, the Everglades, other areas of South Florida, and the world's oceans for over 15 years. The SERP mercury laboratory is currently the EPA contract laboratory for the Ecological Risk Assessment of Mercury Contamination in the Everglades Ecosystem (R-EMAP Project).

This Comprehensive Quality Assurance Plan (CompQAP) describes the sampling and analytical methods used by SERP personnel for mercury. These procedures are used to ensure the integrity and accuracy of field and laboratory data collection and analysis. The CompQAP has been prepared in accordance with the Florida Department of Environmental Protection (FDEP) guidelines. Project-specific objectives and sampling protocols will be described in more detail in Quality Assurance Project Plans (QAPPs).

4.0 Organization and Responsibility

4.1 Capabilities

The mercury research group at SERP conducts both field sampling and laboratory analysis of mercury. SERP performs field sampling of surface water, pore water (water in soils and sediments), soils, sediments, and animal (fish) tissue. Low level mercury concentrations (parts per trillion) in water samples (surface water, pore water, and groundwater), solid samples (soils, sediments), and tissue samples (fish) are determined in the laboratory.

4.2 Key Personnel

Dr. Ronald D. Jones is the director of the Southeast Environmental Research Program (SERP) at Florida International University (Figure 4-1). As director, Dr. Jones supervises all laboratory and field operations and personnel. He provides a final review of all data and documents produced.

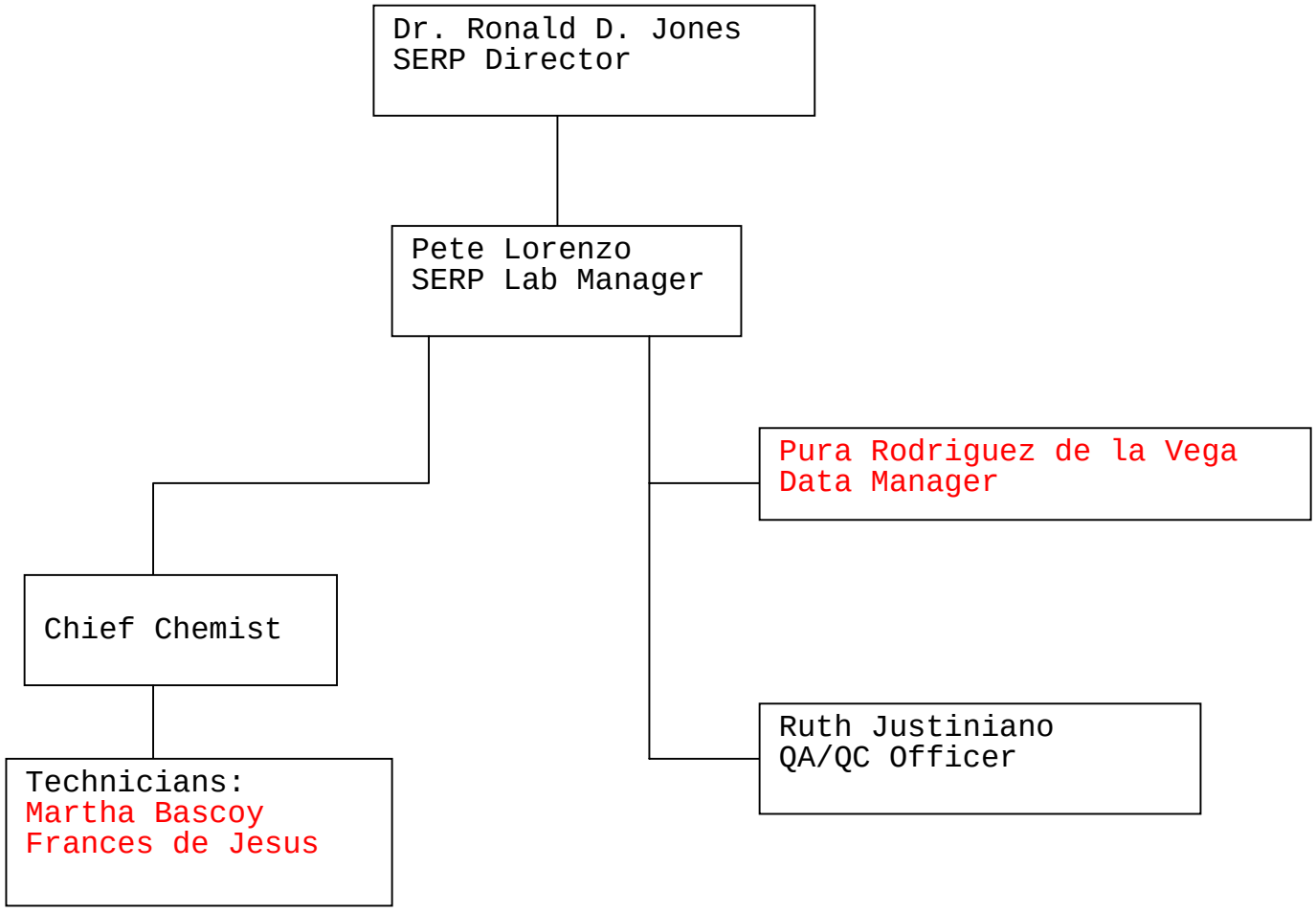
Mr. Pete Lorenzo is the SERP laboratory manager. In this role, he is responsible for the proper execution of the daily field and laboratory operations. He provides scheduling of field and laboratory personnel, and is responsible for the collection, custody, storage, and analysis of all samples.

Ms. Pura Rodriguez de la Vega is the SERP Data manager. She is responsible for checking all the data produced in the lab according with QC criteria and for the preparation of the final data reports.

Ms. Ruth Justiniano is the SERP QA officer. She is responsible for preparing all QAPs, and overseeing that the field and laboratory operations are performed according to the QAPs. She is also responsible for a final check of all data produced with respect to QC criteria, initiating and conducting audits, and preparing QA reports.

Mr. Julio Lopez is the chief mercury chemist. In this role, he is responsible for the proper execution of laboratory operations. He is responsible for the custody, storage, and analysis of all samples. Additional mercury laboratory technicians include Ms. Martha Bascoy and Frances de Jesus.

Figure 4.1- SERP Mercury Laboratory Organization Chart



5.0 Quality Assurance Objectives (Precision, Accuracy, and Method Detection Limits)

Field determined parameters include temperature, conductivity, and pH of surface water and porewater (Table 5.1). Mercury is determined in surface waters, pore waters, ground waters, soils, sediments, and tissue (fish) samples. Dry weight is determined on soil/sediment samples, while wet weight is determined on fish samples. Laboratory precision, accuracy, and method detection limits (MDLs) for these parameters (Table 5.2) are determined using in-house, historically generated data.

Sample preparation methods for analysis of total mercury and organomercury compounds are listed in Table 5.3. Details of the sample preparation methods are given in Appendices A, B, C, and E.

TABLE 5.1
Quality Assurance Objectives
Field Measurements

Method No.	Matrix	Parameter
EPA 170.1	Surface Water, Pore Water	Temperature
EPA 120.1	Surface Water, Pore Water	Conductivity
EPA 150.1	Surface Water, Pore Water	pH
SM 2520 (B)	Surface Water,Pore Water	Salinity

EPA = U.S. Environmental Protection Agency. Methods for Chemical Analysis of Water and Wastes, Revised March 1983.
SM = Standard Methods for Examination of Water and Wastewater, 1989, 18th Edition.

TABLE 5.2
Quality Assurance Objectives
Laboratory Measurements

Analyte	Matrix	Analytical Method	Precision (a)	Conc. Range (b)	Accuracy (%R) (a)	Conc. Range (b)	MDL (c) (ppt)
Inorganic Mercury	Water	(d)	< 20 %*	L, M, H	90 - 110	L	0.3
Methyl & Ethyl Mercury	Water	(e)	< 30 %**	L, M, H	75 - 125	H	0.02
Total Mercury	Water	(d)	< 20 %*	L, M, H	80 - 120	L	0.3
Methyl & Ethyl Mercury	Soils, Sediments	(f)	< 30 %**	L, M, H	N.A.	N.A.	0.02 ppb
	Tissue	(f)	< 30 %**	L, M, H	70 - 130	M	0.02 ppb
Total Mercury	Soils, Sediments	(d)	< 20 %**	L, M, H	80 - 120	L	4.3 ppb
	Tissue	(d)	< 20 %**	L, M, H	70 - 130	L	3.2 ppb
Dry Weight	Soil, Sediments	ASTM D2216-80	< 20 %**	L, M, H	N.A.	N.A.	N.A.
Wet Weight	Tissue	ASTM D4638	< 20 %**	L, M, H	N.A.	N.A.	N.A.

*: Relative Standard Deviation.
**: Relative Percent Difference of Duplicates.
(a) Temporary QA targets for precision and accuracy (valid until enough in-house, historical data is available).
(b) Concentration Range of the linear calibration used to determine precision and accuracy values. Calibration range to 1000 ppt.
L = lower 20% of linear calibration range
M = from 20% to 80% of the linear calibration range
H = the upper 80% of the linear calibration range
(c) Method Detection Limits (MDLs) determined by EPA procedure described in 40 CFR Part 136, Appendix B, revision 1.11.
(d) Inorganic and total mercury determined using a PSA Merlin Plus Fluorescence Detector. Method validation package included in Appendix A.

(e) Organic mercury determined by capillary gas chromatography coupled with atomic fluorescence detection as described by Cai et al. (1996).
Y. Cai., R. Jaffé, A. Alli, and R. Jones. 1996. Determination of organomercury compounds in aqueous samples by capillary GC - atomic fluorescence spectrometry following solid-phase extraction.. Analytica Chimica Acta 334 (251-259)

(f) Organic mercury determined by capillary gas chromatography coupled with atomic fluorescence detection as described by Cai et al (1997).
Y. Cai, G. Tang, R. Jaffe, and R. Jones. 1997. Evaluation of some isolation methods for organomercury determination in soil and fish samples by capillary gas chromatography-atomic spectrometry.

TABLE 5.3
Sample Preparation Methods

Description	Matrix	Sample Prep. for these Methods
Bromination	Water	Total Mercury
Preconcentrated with sulfhydryl cotton fiber, eluted with KBr and $CuSO_4$, extracted with dichloromethane	Water	Methyl & Ethyl Mercury
Sodium thiosulfate clean-up, isolation with cupric chloride, extraction with dichloromethane	Sediment/Tissue	Methyl & Ethyl Mercury
Slurried, Acidification, Autoclave Digestion	Sediment	Total Mercury
Slurried, Autoclave Digestion	Tissue	Total Mercury

Jones, R.D., M.E. Jacobson, R. Jaffe, J. West-Thomas, C. Arfstrom, and A. Alli. 1995. Method **Development and Sample Processing of Water, Soil, and Tissue for the Analysis of Total and Organic Mercury by Cold Vapor Atomic Fluorescence Spectrometry.** Water, Air and Soil Pollution. 80: 1285-1294. (See Appendix B.)

6.0 Sampling Procedures

6.1 Sampling Capabilities

SERP performs sampling of surface water, pore water, soil and sediments and animal tissue (fish) for determination of total, inorganic, and organic mercury (Table 6.1)

6.2 Sampling Equipment and Cleaning Procedures

6.2.1 Sampling Equipment

Preceding a trip to the field, the personnel responsible for collection of the samples are required to ensure that everything is prepared for the expedition. This entails making sure that all sample containers are clean, properly labeled, and stored in plastic bags for transport to the field. Table 6.2 lists the field sampling equipment used for each matrix, while Table 6.3 list the miscellaneous sampling equipment.

Surface water samples are collected using a vacuum system. Surface water typically has extremely low levels of total mercury (less than 10 ppt), and the incorporation of sediment within the sample bottle may lead to misleadingly elevated levels of mercury. The vacuum system illustrated in Figure 6.1 is designed to reduce the collection of sediment and other large particles in the water samples. A 2L Teflon sample bottle is housed within a plastic vacuum chamber. A screen holder is attached to the end of either a 1.5 or 3 ft-long Teflon sampling pole. The screen holder is constructed of polyethylene and polysulfone and houses 105 μm Nytex netting. A vacuum pump is used to create a vacuum within the chamber. All tubing and fittings associated with the system are constructed of Teflon.

Pore water samples are collected using plastic syringes equipped with Teflon tubing. Soil and sediment samples are collected using either polyethylene specimen cups, polycarbonate core tubes, a Wildco Eggshell Core or and Eckman Dredge. Fish are collected using a dip net. All samples are collected with the sampler wearing at least one pair of gloves.

6.2.2 Sampling Equipment Cleaning Procedures

The vacuum system is rinsed with sample water three times prior to sample collection by placing a 2L polyethylene bottle in the system and filling. The Nytex netting is replaced prior to each sample collection. Additional cleaning measures attempted in the field, such as detergent washes or acid rinsing is not conducted due to potential mercury contamination from these solutions. Surface waters contain such low levels of mercury that the potential of mercury contamination of the samples in the field is

TABLE 6.1
SERP Sampling Capabilities

Parameter Group	Sample Source
Inorganic Mercury	Surface Water, Pore Water
Methyl & Ethyl Mercury	Surface Water, Pore Water, Soils, Sediments, Tissue
Total Mercury	Surface Water, Pore Water, Soils, Sediments, Tissue

TABLE 6.2
Field Sampling Equipment

Equipment	Construction	Use	Parameter Groups	Restriction, Precautions, Notes
Water Samples				
Vacuum Apparatus	Housing: Plastic Tubing: Teflon Screen Holder: Polyethylene with polysulfone Screen: 105 μM Nytex netting	Collection	Total, inorganic, and methyl & ethyl mercury in Surface Water	Wear 2 pairs of gloves
Syringe	120 ml plastic (HDPP) with Teflon tubing	Purging, Collection	Total, inorganic, and methyl & ethyl mercury in Pore Water	Use new tubing prior to each sampling event
Soils, Sediments				
Specimen Cups	Polyethylene	Sample Collection	Total and methyl & ethyl mercury	Wear single pair of gloves
Core Tubes	Polycarbonate	Sample Collection	Total and methyl & ethyl mercury	Wear single pair of gloves
Wildco Eggshell Core	Stainless Steel	Corer	Total and methyl & ethyl mercury	Wear single pair of gloves
Eckman Dredge	Stainless Steel	Sample Collection	Total and methyl & ethyl mercury	Wear single pair of gloves
Fish Tissue				
Dip Net	Nylon	Sample Collection	Total and methyl & ethyl mercury	Wear single pair of gloves

TABLE 6.3
Miscellaneous Sampling Equipment

Surface Water Sampling Equipment

1. Labeled sample bottles
2. Ziplock Bags
3. Sounding Line

Pore Water Sampling Equipment

1. Water Level Indicator
2. Ph meter
3. S/C/T meter
4. pH Buffers (7.00 and 10.00)
5. Conductivity check Standard
6. Plastic Beaker
7. Labeled Sample Bottles

Soil, Sediment & Sampling Equipment

1. Labeled Sample Containers
2. Measuring Rule

Tissue Sample Collection

1. Ziplock bags

Sample Preservation and Transportation Supplies

1. Ice
2. Coolers
3. Shipping labels and forms
4. Sample container labels
5. Sealing tape

Protective Clothing

1. Vinyl Gloves (inner gloves)
2. Shoulder length polyethylene gloves (outer gloves)

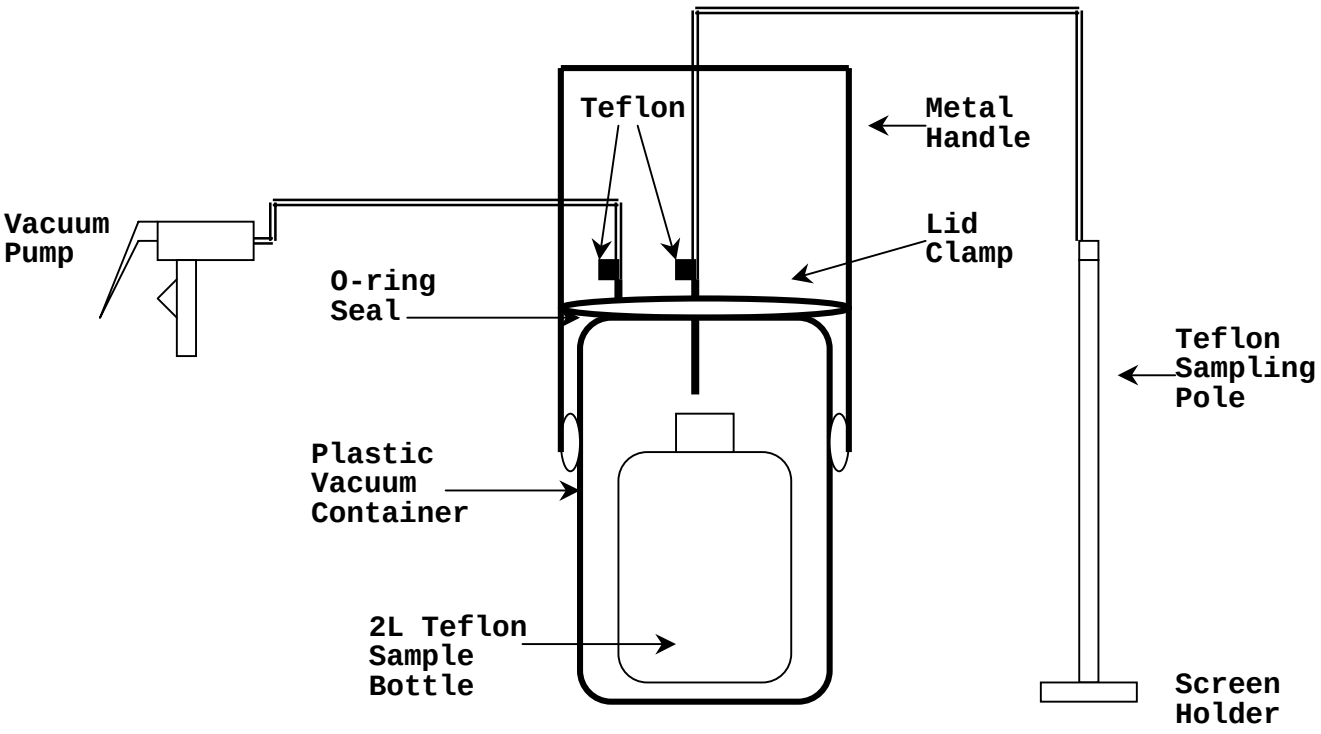
Documentation Supplies

1. Notebooks/logs/field sheets
2. Pens, Markers
3. Sample container labels
4. Custody transmittal forms

Reference Materials

1. COMPQAP/SOP
2. Site map

Figure 6.1
Vacuum System for the Collection of Surface Water Samples



high. The simple rinsing of the vacuum system with sample water three times prior to sample collection has proved acceptable in measuring parts per trillion levels of mercury in surface water samples collected as part of the USEPA Ecological Risk Assessment of Mercury Contamination in the Everglades Ecosystem: R-EMAP Study (USEPA 1993).

The Eckman dredge and the core tubes used in soil and sediment sample collection are subject to precleaning in the laboratory prior to transportation to a field site. All surfaces are washed thoroughly with hot, tap water, using a brush to remove large or stubborn particles. Liquid lab detergents are not used due to possible contamination with mercury. The equipment is then rinsed with acid (0.5N HCl and 0.05N HNO_3), then with "mercury-free" water. The equipment is allowed to air dry completely, then it is wrapped in plastic bags for storage and transportation to the field. The Eckman dredge is cleaned in the field between samples by rinsing with the water overlying the sediment to be collected. This is appropriate since surface water has significantly lower concentrations of mercury then the sediment. Core tubes are not reused between samples, and therefore, do not require additional cleaning in the field. The plastic syringes and Teflon tubing used to collect pore water samples are rinsed three times with the pore water prior to sample collection. Following sample collection, the plastic syringes and Teflon tubing is rinsed with acid (0.5N HCl and 0.05N HNO_3), then with "mercury-free" water. The dip net is rinsed with the surface water at each fish sampling station.

All field cleaning procedures are documented in the field note book. Any sampling equipment used once in the field and not cleaned in the field are tagged with the sample location and cleaned under controlled conditions in the laboratory. All field equipment is cleaned upon return to the laboratory by rinsing with acid (0.5N HCl and 0.05N HNO_3), then with "mercury-free" water. The equipment is allowed to air dry completely, then it is wrapped in plastic bags for storage and transportation to the field. Any field equipment suspected of being contaminated and can not be cleaned is discarded.

6.3 Sample Containers and Cleaning Procedures

Sample containers, preservation methods, and appropriate holding times for each matrix are listed in Table 6.4. New Teflon sample bottles are etched with a unique number and decontaminated with the method outlined below. Once cleaned, one bottle from the batch is filled with mercury-free water (DIW) and analyzed as a quality control check for batch contamination.

Teflon sample bottles are cleaned and reused indefinitely. After old samples are discarded, the bottles are rinsed three times with mercury-free DIW and filled with 1% HCl. After filling, 1 ml of brominating agent is added for every 50 ml and the bottle

TABLE 6.4
Sampling Containers, Sizes, Preservations and Holding Times

Sample Type/ Parameter	**Container/ Size**	**Preservative**	**Holding Time**
Water/ Total, Inorganic and Methyl & Ethyl Mercury	Teflon, 2-liter	10 ml HCl per 2 liters of sample	28 days in a Hg-free room
Soils, Sediments/ Total and Methyl & Ethyl Mercury	Polyethylene Specimen cups	Frozen	Indefinitely, but preferably within 28 days
Tissue/ Total and Methyl & Ethyl Mercury	Plastic Bags	Frozen	Indefinitely, but preferably within 28 days

is shaken and stored in a mercury-free clean room until used. When ready to be used, 500 µl of hydroxylamine hydrochloride is added to each bottle to remove all of the free bromine. The bottle is then shaken, emptied, and the bottle and cap are rinsed three times with mercury-free water. All sample containers are placed in new plastic bags for transport to the field.

Mercury-free water is produced by filtering tap water through a Culligan system consisting of activated charcoal and two mixed bed ion exchange cartridges. The filtered water is piped to a mercury-free clean room, where it is passed through a Barnstead Mega-ohm B Pure system. This system is fitted with two filters (Thermolyne: colloid/organic-D0835, and ultrapure-D0809) in line with a 0.22 micron pleated particle filter. Mercury levels are not detectable in this water in both our laboratory and in an independent laboratory analysis (<0.1 ppt). The only water used in the mercury laboratory is this mercury-free water and it is used for blank preparation and final decontamination rinse. Documentation is maintained within the laboratory demonstrating the reliability and "purity" of the analyte free water from analysis of method reagent blanks. Mercury-free water containers are cleaned by rinsing with 0.5N HCl and 0.05N HNO_3, then three times with the mercury-free water prior to filling.

6.4 Sampling Protocols

For the determination of ultra-low levels of mercury (parts per trillion) clean sampling protocols must be employed throughout the field sampling effort. The field methods described herein were developed for the USEPA Ecological Risk Assessment of Mercury Contamination in the Everglades Ecosystem: R-EMAP study (USEPA, 1993). These sampling protocols are described in SERP's internal standard operation procedures (Appendix D). A copy of the SERP S.O.P. is carried to the field during each sampling event.

For each sampling event, one of the field crew members is designated as the "clean person". This person is responsible for all sample handling, including sample collection, securing the sample container in a plastic bag, and placing the sample in the cooler. All samples are collected with the sampler wearing vinyl gloves. For collecting water samples, the sampler dons shoulder length polyethylene gloves over the vinyl gloves. All samples are secured in a plastic bag prior to placing in the cooler. Water samples are most susceptible to mercury contamination, therefore, they are enclosed in two plastic bags. All samples are stored in coolers for transport to the laboratory. These coolers are used exclusively for low level mercury samples.

Specific sampling locations are chosen based on criteria described in the appropriate Quality Assurance Project Plans. In general, surface water, sediment, and tissue samples are collected from a boat, helicopter, or airboat. Airboat, van, and helicopter exhaust are a potential source of mercury. To ensure collection of undisturbed and uncontaminated samples, the boat or airboat is advanced toward a sampling station from the downstream direction, while the helicopter is advanced toward a station from the downwind direction. Samples are collected from the bow of the boat away from the engine. If wading or walking is possible, samples should be collected approximately 10 meters upgradient from the boat, airboat, or helicopter. If surface water samples and sediment and/or tissue samples are to be collected at one location, then the sequence for sampling is surface water, sediment, then tissue samples. In areas of suspected high concentrations,

samples are collected in order of suspected low concentration to higher concentration.

6.4.1 Surface Water Sampling

All surface water samples, whether from an ocean, bay, canal, or marsh are collected according to the following protocols.

1. Advance to the sampling station from the downgradient direction.
2. The "clean person" will put on a pair of vinyl gloves, then a pair of shoulder length polyethylene gloves and handle the vacuum system sampling pole and sampling container. Other members of the sampling team, wearing a pair of vinyl gloves, can provide assistance with creating and releasing the vacuum in the vacuum chamber. The "clean person" will also don a pair of waders if necessary.
3. Place a 2-liter plastic, rinse, bottle in the vacuum chamber. Submerge the sampling pole and screen holder beneath the surface of the water. Create a vacuum in the chamber and fill the bottle to rinse the screen and tubing with the sample water. Release the vacuum and discard the rinse water in the bottle downstream of the sampling location.
4. Remove a 2-liter Teflon bottle from its protective plastic bag, remove the cap and place the bottle within the vacuum system.
5. Submerge the sampling pole and screen holder to a depth applicable to the project objectives, create a vacuum within the chamber, and fill the bottle.
6. Release the vacuum in the chamber and remove the bottle.
7. Tightly cap the bottle, and place in a ziplock bag and seal it. Place the sample in a second bag and seal it. Place the double-bagged sample in a cooler.

Duplicate water samples are collected by placing another 2-liter Teflon bottle within the vacuum chamber immediately following collection of the first sample. Split samples for inter-laboratory comparison are made in the "clean-room" in the laboratory, where the first sample is preserved with acid, then split into two equal sample volumes.

6.4.2 Surface Soil Sample Collection

1. Advance to the sampling station from the downwind direction.
2. The "clean person" will put on a pair of vinyl gloves and a pair of shoulder length polyethylene gloves and handle the sample container.
3. Remove a 4-ounce specimen cup from its protective plastic bag.
4. Remove surface detritus prior to sample collection.
5. Using a stainless steel trowel, spade or spatula collect a grab sample of surface soil (upper 10 cm) and place into the 4-ounce specimen cup.

6. Place the lid on the cup, label the cup, secure in a ziplock bag and place in a cooler.

Duplicate surface soil samples are collected following the above procedures using the same sampling equipment used to collect the first sample. Soil samples are homogenized either in the field or in the laboratory, depending upon the project objectives. If homogenized in the field, the soil sample is placed into a polypropylene mixing tray and homogenized by slicing, mixing, and remixing of the sample. The homogenized soil sample is then placed into a wide-mouth specimen cup and stored in a cooler in the dark for transport to the laboratory. In the laboratory, soil samples are homogenized by mixing the entire sample in a blender.

6.4.3 Subsurface Soil Sample Collection

1. Advance to the sampling station from the downwind direction.
2. The "clean person" will put on a pair of vinyl gloves and a pair of shoulder length polyethylene gloves and handle the sample container.
3. Remove a plastic core tube from its protective plastic bag.
4. Push the core tube into the soil by hand while rotating the tube in a circular motion.
5. Cap the top of the core tube with either a plastic or rubber stopper, then extract the core tube.
6. Cap the bottom end of the core tube, label the core indicating the top direction.
7. Seal the core in a plastic bag and place in a cooler.

A duplicate subsurface soil sample is collected according to the procedure described above immediately following the collection of the first sample. Soil samples are homogenized either in the field or in the laboratory, depending upon the project objectives. If homogenized in the field, the soil sample is placed into a polypropylene mixing tray and homogenized by slicing, mixing, and remixing of the sample. The homogenized soil sample is then placed into a wide-mouth specimen cup and stored in a cooler in the dark for transport to the laboratory. In the laboratory, soil samples are homogenized by mixing the entire sample in a blender.

6.4.4 Sediment Sample Collection

Sediment samples are collected either with polycarbonate core tubes or with an Ekman Dredge. The Ekman Dredge is used during sample of quiescent waters such as impoundments or lakes, while the core tubes are used in moving waters such as rivers or streams to minimize the loss of fine particles. Samples collected with core tubes are collected following the procedures outlined in Section 6.4.3. Sediment samples collected with an Ekman Dredge are collected according to the following protocols.

1. Advance to the sampling station from the downgradient direction.
2. The "clean person" will put on a pair of vinyl gloves and a pair of

shoulder length polyethylene gloves and handle the sample container.

3. Other members of the sampling team can assist in securing the line of the dredge to the boat or helicopter.
4. Slowly lower the dredge through the water column to the bottom.
5. Close the dredge to collect the sediment, then pull up the dredge.
6. The "clean person" will use a stainless steel knife or spatula to collect a section of the sediment from the center of the dredge and place into a clean specimen cup.
7. Cap and label the specimen cup. Place the cup in a ziplock bag and into a cooler.

A duplicate sediment sample is collected immediately following the collection of the first sample using the same sampling equipment used to collect the first sample. Soil samples are homogenized either in the field or in the laboratory, depending upon the project objectives. If homogenized in the field, the soil sample is placed into a polypropylene mixing tray and homogenized by slicing, mixing, and remixing of the sample. The homogenized soil sample is then placed into a wide-mouth specimen cup and stored in a cooler in the dark for transport to the laboratory. In the laboratory, soil samples are homogenized by mixing the entire sample in a blender.

6.4.5 Pore Water Sample Collection

SERP collects pore water from either temporary or permanently placed lysimeters. Prior to sampling, the water level and bottom of the lysimeter are measured to determine the standing volume of water. Using either a peristaltic pump or a syringe equipped with Teflon tubing, the lysimeter is purged of three volumes of standing water or pumped dry. The volume of water removed is recorded in the field note book. Temperature, specific conductance and pH are monitored while purging. If the lysimeter does not produce enough water, then it is pumped dry and sampled immediately following recovery. Since SERP does not sample hazardous water, the purge water is allowed to drain on the ground away from the lysimeter. Pore water is collected in 2-liter sample bottles (Table 6.4), after the bottle is rinsed three times with sample water. Duplicate samples are collected immediately following the collection of the first sample using the same sampling equipment and tubing. Split samples for interlaboratory comparison are prepared in the laboratory "clean-room" where the first sample is preserved with acid, then split into two equal volumes.

6.4.6 Fish Sample Collection

Fish are collected from a helicopter, boat, or airboat using a dip net. The fish are identified and placed in ziplock bags. The bags containing the fish are labeled and stored in a cooler with ice.

6.5 Sample Documentation and Identification

All sample bottles or containers are pre-labeled in the laboratory prior to transport to the field site. Labels consisting of colored tape are attached to the side of the bottle. Water-proof ink pens are

used to mark the labels. The collection of all samples is recorded in the field notebook.

Documentation brought into the field include:

1. Equipment checklist
2. Field Notebook
3. SERP Internal S.O.P.
4. Field equipment manuals (if appropriate)
5. Chain of custody form

6.6 Sample Preservation, Holding Times, and Sample Volume

Sample containers, sizes, preservatives, and maximum holding times, by matrix are included in Table 6.4. Samples are preserved in the field by putting in a cooler with ice for transport to the laboratory. These coolers are used exclusively for low level mercury samples.

In the laboratory, the 2-liter bottles with water sample are preserved with 10 ml of trace metal grade HCl to a pH less than 2. The HCl is added to the water bottles in a "mercury-free" room. Hydrochloric acid is preferred over nitric acid as it results in higher recoveries of mercury (Ahmed et al. 1987). The pH of the water sample is checked by shaking the bottle and then pouring a small amount of the sample onto a pH test strip. The water samples are stored in the "mercury-free" room and analyzed within 28 days.

Acid addition is completed within the mercury-free room following collection of the samples, because addition of acid to sample bottles has resulted in a measurable uptake of mercury from the atmosphere. This uptake is measurable if the acid is added to the sample bottle either in the laboratory prior to sample collection or in the field following sample collection. The addition of HCl to the sample bottles in a mercury-free "clean room" within the same day of sample collection results in no measurable loss or addition of mercury. This method of preservation is considered standard protocol by EPA Region IV in Athens, GA, EPA ORD-EMSL in Cincinnati and Battell Marine Science Laboratory in Sequim, Washington (USEPA, 1993).

The fish and sediment samples are stored in a freezer in the mercury laboratory. These samples can remain in the freezer for an indefinite time; however, SERP prefers to process the sediment and the fish samples within 28 days of receipt.

6.7 Sample Dispatch

Samples are stored in coolers (on wet ice for sediment and tissue samples) and delivered to the SERP laboratory by the field personnel on the same day of sample collection. Samples to be sent to an out-side laboratory are shipped no later then one day following sample collection using a common carrier and overnight delivery. These samples are placed in individual plastic, sealable bags and packaged with bubble wrap or styrofoam. Non-insulated cardboard boxes are used for shipment of water samples, while insulated coolers with ice are used for sediment and fish samples.

6.8 Reagent Storage and Waste Disposal

Field reagents are not required for mercury sampling. Acid preservative is added to the water samples in a "mercury-free" room within the laboratory. SERP does not perform sampling of hazardous waste sites, and there is no generation of field wastes.

7.0 Sample Custody

Sample custody is controlled by SERP, mainly since the employees responsible for sample collection are also the same employees responsible for sample analysis. Should the need arise to send samples via courier to another laboratory, then the sample chain-of-custody (Figure 7.1) and/or one supplied by the contract laboratory will be used. This form will be included within the sample cooler and protected within a plastic, sealable bag. A copy of the form will be retained by SERP in project specific files. Upon receipt of the samples, the receiving laboratory will be requested to sign the sample chain-of-custody form and send a copy of the signed form via mail or facsimile to SERP. The QA officer will be in charge of ensuring that a copy of the signed chain-of-custody form is obtained from the receiving laboratory.

Sample bottles sent by SERP to another laboratory are sent differently according to their matrix. Water samples are sent sealed in individual plastic bags and then in a larger plastic bags. The samples are packaged in cardboard boxes filled with packing material (peanuts) to prevent breakage. Fish and sediment samples are shipped in coolers with ice. The lids and drain ports of the coolers are secured with shipping tape to avoid opening. These samples are sent overnight delivery, and all shipping receipts are kept and maintained in a central file location. Sample personnel responsible for sample delivery are identified in the chain of custody form as well as common carriers that might have been used in the process.

SERP also often receives samples (surface water, ground water, soils, sediments, and tissue) collected by other researchers for analysis. A completed SERP chain-of-custody form will be required to accompany the samples and will be signed by the QA officer or SERP technician receiving the samples. These samples will be inspected by the SERP QA officer or SERP technician for integrity and completeness according to the chain-of-custody form. Any discrepancies between the samples received and the chain-of-custody form will be reported immediately to the researcher that collected the samples. A copy of the chain-of-custody form will be kept in the project specific files.

All sample bottles are pre-labeled in the laboratory prior to transport to the field site. Labels consisting of colored tape are attached to the side of the bottle. Water-proof ink pens are used to mark the labels. Since sample containers used for the suspended matter samples, sediment samples and tissue samples are used only once, they are marked with water-proof ink pens directly on the outside of the sample container.

Each sample container is labeled with a unique sample identification number as indicated below:

AAA###YYMMDDB.

FIGURE 7.1 Sample Chain-of Custody

SOUTHEAST ENVIRONMENTAL RESEARCH PROGRAM

OE 148 (office)/VH 321 (lab), University Park, Miami, FL 33199, 305-348-3095

Page of

Chain of Custody Record/Sample Log - Mercury

CLIENT/PROJECT NAME:	ACCOUNT NO.:	AUTHORIZATION:
DELIVERED BY:	RECEIVED BY:	DATE AND TIME:
RECEIPT		ICE IN COOLERS?:

BOTTLE ID	SAMPLE ID	MATRIX *	COLLECTION DATE	COLLECTION TIME	# OF REPLICATES	PRESERVATIVE	ANALYSES THG	OHG	AFDW	BULKD	TP	TN	TOC	SAMPLE COMMENTS

** Matrix: FW = fresh wate, SW = seawater, S = soil/sediment, P = particulates, T = tissue (fish)*

***: Analyses and methodsTHG (Total mercury, see CompQAP); OHG (Organic Mercury, see CompQAP); AFDW (Ash-Free Dry Weight, ASTM D2974-87); BULKD (Bulk Density, ASTM D4531-86);*

TP (Total Phosphorus, EPA 365.1), TOC (Total Organic Carbon, EPA 415.1); TN (Total Nitrogen, Antek);

The first letters in the sample identification number (AAA) refer to the program name. For example, the three letters ECS would be used to designate the EPA Canal Sampling program. The first set of numbers (###) can vary from 1 to 999 and refers to the sample location number. The date of collection follows in a year, month, day format. The second letter designation (B) refers to the type of media collected and will be a W, S, or T to represent water, soil/sediment, or tissue, respectively. The reusable water sample containers are etched with permanent numbers. These numbers will be identifying the sample as an additional character after the W. An example of the sample label is included below:

ECS101950115W34

Sample numbers are recorded on sample containers, in field notebook, and sample log list.

SERP often receives samples collected by other researchers. When this is the case, SERP uses the unique sample numbers supplied by the other researcher.

7.1 Field Custody

Loose-leaf field notebooks are used for all field documentation. Once QA checked, the sheets are removed from the notebooks and kept in project-specific files. All field notebook entries are made in waterproof ink. If an error is made, corrections are made by drawing a single line through the mistake and initialed by the person making the correction, entering the corrected information next to the error.

General field notebook entries include the following: name and number of sampling trip; date of sampling trip; general weather and water conditions (waves and tides); name of individuals in sampling team; sample identification number; time of sample collection; a description of the sampling location; and latitude and longitude as determined with a GPS unit, and sample preservation method. Additional information includes sampling equipment used, decontamination procedures used, types of QC samples collected, the use of fuel powered units if any, and the depth that the samples were collected. For the collection of pore water samples, additional information recorded in the field notebook includes the date and time of purging, the equipment used, the water level in the lysimeter, the bottom depth of the lysimeter, the volume of water removed during purging, and the specific conductance, temperature, and pH during purging. For soil and sediment samples, additional information recorded in the field notebook include the depth of sample collection, physical characteristics of the soil, and method of homogenization. For tissue samples, identifying characteristics of the fish are included.

In addition to the field notebook, the field sampling team keeps a field instrument sheet. On this sheet is recorded the number of each field instrument and probe, as well as instrument calibration check information.

7.2 Laboratory Custody

Upon transport of the samples to the SERP laboratory at FIU, the Chief Chemist checks that the

number and identity of the samples matches those on the field notebook (if sampled by FIU) and on the chain-of-custody form. The samples and coolers are checked for presence of ice, odors and/or contamination. In addition, the integrity of the samples is checked and any bottles found broken or leaking are noted in the field notebook and chain-of-custody form. Samples are logged-in by the Chief Chemist on the Mercury Sample Log List (Figure 7.2). The temperature of the samples are checked. Sample bottles found broken, leaking, or not properly preserved are rejected from analysis, and noted on the chain-of-custody form. The Mercury Sample Log List is kept into the corresponding sample set folder and serves to track the samples collected from a sampling event through sample storage, analysis, data validation, and sample disposition with dates and authorized initials.

Samples will not be rejected based on incomplete documentation. Analysis will continue, but efforts will be made to get the missing information. If there is still missing information by the time the results are complete, it will be noted in the data report.

Samples are stored in the appropriate conditions, "mercury-free" room or freezer, in a locked room, with access to the room limited to SERP employees. Standards are stored separately from all samples. Sample preparation methods are described in the Standard Operating Procedures for the laboratory (Appendix C). Primary and secondary standards for analysis of water samples are prepared on a daily basis. The standard concentration and resulting peak height are recorded in the instrument log book (Figure 7.3). Preparation of all standards (primary and secondary) are recorded on a separate log (Figure 7.4).

Sample analysis is tracked through sample preparation sheets, instrument printouts, and analytical calculation sheets. Examples of each of these sheets for each matrix (water, sediment, tissue) are included in Appendix D. Sample analysis is also recorded on the instrument log book (Figure 7.3). All laboratory documentation, Sample Log Lists, chain-of-custody forms, standard prep logs, sample prep logs, instrument printouts, and calculation sheets are kept indefinitely by the Chief Chemist in a locked cabinet. All internal memos, phone logs, and sample receipt/log-in forms are saved as well.

7.3 Electronic Data Records

Data from field measurements and laboratory analyses are compiled and summarized in computer spreadsheet format (Microsoft Excel). Separate spreadsheets for each sampling day are kept, and a compilation of all data to date is made. Spreadsheets are stored both on the hard drive of the computer, as well as onto write-protected floppy disks. In the event of computer equipment failure, the data files on the floppy disks are used as backup. All spreadsheet calculations are checked by the Chief Chemist or by the QA Officer on a calculator.

The access to these electronic records is password protected. A hard copy of the spreadsheets are stored in the project files indefinitely.

All deletions or corrections will be documented on a hard copy of the spreadsheet and the person making the corrections will initial any changes.

Records of all aspects relating to changes, updates, problems and maintenance of the instrument and database software will be maintained in the instrument logbooks.

FIGURE 7.2. Mercury Sample Log Checklist

Sample Checklist

Date Received: _____

Date Collected: _____

Collected From (Area): _____ Technician: _____

Lab Receipt Date: _____ Received From: _____

Sample Type: ____ Freshwater ____ Soil ____ Particulates

____ Seawater ____ Sediments ____ Tissue (Fish)

Description of Sample Containers Number of Containers: _____

Type of Containers: _____

Containers Labelled as : _____

Analyses Required		Date Required:	Date Performed:	Data Discarded:	Date / Data Entry:
THg		_____	_____	_____	_____
OrgHg		_____	_____	_____	_____
TP		_____	_____	_____	_____
Bulk Density		_____	_____	_____	_____
Ash Free Dry Wt		_____	_____	_____	_____
NPOC		_____	_____	_____	_____
Turbidity		_____	_____	_____	_____

FIGURE 7.3. Instrument Log Book

FIGURE 7.4. Reagent and Standard Prep Log

8.0 Analytical Procedures

SERP determines total and inorganic mercury concentrations by Cold Vapor Atomic Fluorescent Spectrometry (CVAFS). A PS Analytical (PSA) 10.025 Millennium Merlin is used to detect total and inorganic mercury in water samples while a PS Analytical (PSA) Merlin Plus CVAFS mercury analysis system equipped with a PSA autosampler, a PSA vapor generator, and a mercury fluorescence detector Model PSA 10.023 is used for determination of total and inorganic mercury in soil and sediment samples.

The method validation for part per trillion (ppt) concentrations of inorganic and total mercury in water, solid, and tissue samples (April, 1996) is included in Appendix A. A detection limit of 0.3 parts per trillion (ppt) Hg in water samples is obtained at a precision of better than 5% relative standard deviation and an accuracy between 90 and 110%. Higher levels of precision and accuracy are obtained for sediment and tissue samples due to their inherent higher mercury concentrations.

Organomercury concentrations are determined by capillary gas chromatography coupled with atomic fluorescence detection (GC-AFS) as described by Cai et al. (1996) and Cai et al (1997). Chromatography is performed with a Hewlett-Packard (Model 5890 Series II) gas chromatograph coupled with an HP (Model 7673) automatic sampler. A Merlin Mercury Fluorescence Detector System (AFS), Model 10.023, (P.S. Analytical) is used. Initial extracts of sediment and tissue samples are subjected to sodium thiosulfate clean-up and the organomercury species are isolated as their chloride derivatives by cupric chloride and subsequent extraction into a small volume of dichloromehtane. For water samples, the organomercury compounds are pre-concentrated using a sulfhydryl cotton fiber adsorbent, followed by elution with Kbr and $CuSO_4$ and extraction in dichloromethane. Detection limits of 0.02 ppt and 0.02 ppb are obtained for water and sediment/tissue samples, respectively. The method validation for organomercury compounds in water, sediment, and tissue samples (November, 1997) is also included in Appendix A.

See Appendix C for a detailed description of the currently used methods in the corresponding Standard Operating Procedures.

8.1 Laboratory Operations

Mercury contamination at levels near the method detection limit is a consistent problem as water samples easily absorb mercury from the air and improperly cleaned glassware. To minimize contamination, all technicians are required to wear vinyl gloves. In addition, all glassware, acids, reagents, pipettes etc. are kept dedicated to mercury analysis and stored in a mercury-free clean room. The clean room contains a bank of laminar flow hoods equipped with gold and charcoal filters. The floor is covered with flypaper to trap particulates. The clean room also contains a separate water supply, a refrigeration unit, a drying oven, and an analytical balance used exclusively for mercury determinations. Potential contamination in the clean room is checked weekly by monitoring acidified (1% HCl) water samples, which are stored open inside the clean room. If significant levels of mercury are detected in these samples (>20 ppt), then the source of the mercury contamination is located and eliminated. If necessary, the gold and charcoal filters within the flow hoods are reconditioned.

8.2 Laboratory Glassware Cleaning

Laboratory glassware is kept to a minimum, with Teflon bottles and beakers used when possible. All reusable laboratory glass bottles, volumetric flasks, and graduated cylinders, and teflon beakers are dedicated to the preparation and storage of a specific reagent, and are rinsed between usage with acid (0.5N HCl and 0.05N HNO_3), three times with DIW, and stored in the mercury-free room. The volumetric flask used for making the primary standard is dedicated for that standard and rinsed only with the standard. Glassware or plastic containers that have come in contact with samples, such as ampoules and scintillation vials are used once then discarded.

New containers are decontaminated. A log book kept in the Hg-clean lab (Figure 8.1) is used to document new containers, dates of decontamination with initials of involved personnel. Results of quality control tests are also documented in this log book.

8.3 Reagent and Chemical Storage

Reagents and chemicals used in the mercury laboratory include acids, dry chemicals, solvents, and compressed gases (Table 8.2). All acids and dry chemicals are stored in the mercury-free clean room. As each reagent or chemical is received it is dated and initialed by the person unpacking it. When the container is opened for the first time it is dated again and initialed by the opener. While being used in the laboratory, compressed gas cylinders are secured upright with straps or chains.

8.4 Waste Disposal

Wastes produced in the laboratory include liquid acids, solvents and salt mixtures. Many of these reagents are spent during sample prep and analysis. Any remaining waste acids or salt mixtures are neutralized or diluted, respectively, then washed down the sink to the sanitary sewer. According to Dade County Code of Regulations Chapter 24-11(d), effluents containing 0.01 mg/l or less of mercury may be discharged to a sanitary sewer. The source standard with a concentration of 1 mg/l Hg, is never emptied down the sink. This standard is used completely to make the primary standard. The primary standard has a mercury concentration of 0.1 mg/l, and is diluted 1:10 before discharged to the sink. Secondary standards have a high concentration of 0.0005 mg/l and do not need to be diluted before discharged to the sink. Empty reagent bottles are rinsed with hot tap water and disposed in trash receptacles. Waste solvents, such as dichloromethane, are stored in a clearly marked, capped, glass container within a fume hood. When the container is full it is picked up from the laboratory by FIU's Environmental Health and Safety Department who in turn ensure that the solvent waste is disposed of by an licensed and approved hazardous waste disposal facility.

FIGURE 8.1 Hg Lab Decontamination Log Book

TABLE 8.1
Reagent and Chemical Storage

Chemical	**Method of storage**
Laboratory Chemicals	
Mineral Acids	Stored in original glass containers in the mercury-free room.
Dry Chemicals	Stored in original containers in the mercury-free room.
Solvents	Stored in original containers in a locked cabinet marked flammable.
Compressed Gases	Secured upright in laboratory.

9.0 Calibration Procedures and Frequency

9.1 Instrument

SERP determines total and inorganic mercury concentrations by Cold Vapor Atomic Fluorescent Spectrometry (CVAFS) and organomercury concentrations by capillary gas chromatography coupled with atomic fluorescence detection (GC-AFS). Instrument lists for both field and laboratory are included in Tables 9.1 and 9.2, respectively.

9.2 Standard and Reagent Receipt and Traceability

Primary standards traceable to NIST reference standards are purchased from reliable scientific supply firms. The manufacturer's certificates for each standard received are kept on file in a central location. The standards and reagents are received by the Chief Chemist, inspected, dated, and initialed directly on all chemical bottles. Once opened, the standard/reagent bottles are dated and initialed again.

Primary and secondary standards are prepared daily by diluting the source and primary standards, respectively, with mercury-free water. Records of the standard and reagent preparation (including calibration, QC, and MDL standards) are kept in the reagent and standard prep log book (Figure 7.4). Once prepared, the standard/reagent solutions are dated, initialed, marked with the concentration, and referenced to the stock solutions with the date the stocks were opened. The expiration date and storage instructions are recorded as well on the standard/reagent bottles. As no new standard or reagent is prepared until the previous one has been either completely used or expired and discarded, the logbook records link the preparation with every specific analysis.

9.3 Standard Sources and Preparation

The source, preparation, and storage of standards for each sample matrix are included on Table 9.3. Standard preparation methods are available in the laboratory SOPs (Appendix C). The calibration secondary standard for total and inorganic mercury is prepared on a daily basis from a primary NBS certified mercury source standard of 1000 μg Hg/ml (Fisher Scientific) and the second source secondary standard is prepared from a primary SPEX 1000 μg Hg/ml solution. Due to their high mercury concentrations, the primary and secondary standards are stored outside the mercury-free room. The working calibration and second source standards are prepared daily from the corresponding secondary standard solution.

The methyl and ethylmercury chloride standards are purchased from Ultra Scientific. The primary standards are prepared from the source by dissolving appropriate amounts of the standards in Optima grade methanol (Fisher Scientific). A mixed methyl & ethylmercury chloride secondary standard is prepared in methanol from the primary solutions and stored in dark. The working, spiking , and calibration standards are prepared consecutively by dilutions in DIW.

A detailed description of the standard preparation procedure for every matrix/analyte combination is included in the specific SOPs in Appendix C.

TABLE 9.1
Field Instrument List

Manufacturer	Model	Parameters	Matrix
Field Equipment			
Orion	140 Conductivity/ Salinity/ Temperature Meter	Conductivity, Temperature	PW
Orion	SA 250 Meter and Ross Combination Electrode	pH	PW

TABLE 9.2
Laboratory Instrument List

Manufacturer	Model	Parameter	Matrix
P.S. Analytical Ltd.	Merlin Plus Fluorescence Detector (detector PSA 10.023)	Inorganic and Total Mercury	S, SED, T
P.S. Analytical Ltd.	10.025 Millennium Merlin System	Inorganic and Total Mercury	SW, PW, GW
Hewlett-Packard	Model 5890 Series II Gas Chromatograph	Methyl & Ethyl Mercury	SW, PW, GS, S, SED, T
Wilmont Castle Co.	Thermatic 60 Autoclave	Methyl & Ethyl Mercury and Total Mercury	S, SED, T
Fisher Scientific	738F Isotemp Oven	Dry Weight	S, SED
Allied	Model 7303DA Balance	Wet and Dry Weight	S, SED, T
Osterizer	10 Speed Blender	Methyl & Ethyl Mercury and Total Mercury	S, SED, T

TABLE 9.3
Standard, Source, Preparation, and Storage

Instrument/ Parameter	Standard Sources	How Received	Source Storage	Preparation from Source	Lab Stock Storage	Preparation Frequency
Merlin Plus Fluorescence Detector	FISHER SM114-100	1000 ppm Solution (100 ml)	Room Temperature outside the Hg-free room	. Secondary from source: 100 ppb . Working from secondary: See SOPs 002-99 and 003-99 in Appendix C	Room Temp. outside the Hg-free room	Daily
	……	……..	……..	……	……..	.....
	SPEX PLHG4-2Y	1000 ppm Solution (100 ml)	Room Temperature outside the Hg-free room	. Secondary from source: 100 ppb . Working (QC-check)from secondary: See SOPs 002-99 and 003-99 in Appendix C.	Room Temp. inside the Hg-free room	Daily
	.....	.....SRMs.....	.....	…….	.....……	……
	NIST Sediment 8407 and 8406	Dry Soil Standards	Desiccator in the Hg-free room	Digested as if a solid sample	Not Applicable	Daily
	NBS Oyster Tissue 566a	Dry Tissue Standard	Desiccator in the Hg-free room	Digested as if a tissue sample	Not Applicable	Daily
	NRCC DORM-2	Dry Tissue Standard	Desiccator in the Hg-free room	Digested as if a tissue sample	Not Applicable	Daily

TABLE 9.3
Standard, Source, Preparation, and Storage

Instrument/ Parameter	Standard Sources	How Received	Source Storage	Preparation from Source	Lab Stock Storage	Preparation Frequency
10.025 Millennium Merlin System	FISHER SM114-100 …… SPEX PLHG4-2Y	1000 ppm Solution (100 ml) …….. 1000 ppm Solution (100 ml)	Room Temperature outside the Hg-free room …….. Room Temperature outside the Hg-free room	. Secondary from source: 100 ppb . Working from secondary: See SOP 001-99 in Appendix C …… . Secondary from source: 100 ppb . Working (QC-check)from secondary: See SOP 001-99 in Appendix C	Room Temp. outside the Hg-free room Room Temp. inside the Hg-free room …….. Room Temp. outside the Hg-free room Room Temp. inside the Hg-free room	Daily ..... Daily
HP Gas Chromato-graph	Ultra Scientific	Dry methyl- and ethylmercury chloride crystals	Desiccator outside the Hg-free room	. Primary from source in methanol . Secondary from primary in methanol .Working from secondary in DIW	Stored in a dark bottle in desiccator outside the Hg-free room. Stored in a dark bottle in desiccator outside the Hg-free room. Not Applicable	Yearly Monthly Weekly

TABLE 9.3
Standard, Source, Preparation, and Storage

Instrument/ Parameter	Standard Sources	How Received	Source Storage	Preparation from Source	Lab Stock Storage	Preparation Frequency
HP Gas Chromato-Graph (cont.)	Ultra Scientific	Dry methyl- and ethylmercury chloride crystals	Desiccator outside the Hg-free room	. Spiking from Working in DIW . Calibration from Spiking in DIW See SOPs 004-99, 005-99, and 006-99 in Appendix C	Not Applicable Not Applicable	Daily Daily
	Canada National Research Council	.....SRM..... Dry Tissue Standard	Desiccator in Hg-free room	Digested as if tissue sample	Not applicable	Daily
Orion S/C/T Meter	Orion	1000 ml Conductivity Solutions (98.6, 993, and 102822 µmhos/cm) and Salinity standard (Gulf Stream Water, 36 ppt)	Room Temperature	Used Directly	Room Temperature	Replace on expiration
Orion pH Meter	Fisher Scientific, Inc.	pH 4.0, 7.0 and 10.0 solutions	Room Temperature	Not Applicable	Not Applicable	Replace on expiration
Analytical balances	Troemner	Stainless Steel (class S weights)	Room Temperature	Not Applicable	Not Applicable	Daily, Semiannual Service Calibration

An NIST certified Tennessee River sediment (8406, 60 ppb Hg or 8407, 50 ppm Hg) is used as a Standard Reference Material (SRM) for total mercury determination in soils and sediments . Either an NBS certified oyster tissue (566a , 60 ppb Hg) or a NRCC (National Research Council of Canada) certified dogfish muscle (DORM-2, 4600 ppb Hg) is used as the SRM for total mercury analysis in fish tissue samples. Both the sediment and tissue SRMs are digested and analyzed in the same manner as the unknown samples.

The NRCC certified dogfish muscle (DORM-2, 4600 ppb Hg) is also used as standard reference material for organic mercury determination in fish tissue samples.

9.4 Instrument Calibration

All field and laboratory instruments are calibrated, and checked for proper function in the field prior to analysis. Table 9.4 summarizes the calibration procedures for both field and laboratory instruments. Calibration procedures for all instruments are described below.

9.4.1 Field Instruments

Field instrument calibration checks are recorded on the Field Instrument Calibration Sheet, which are kept in individual project files.

9.4.1.1. Salinity/Conductivity/Temperature

The Orion model 140 Salinity/Conductivity/Temperature meter, with a 014010 4-electrode probe, is factory calibrated and compensated for temperature. Salinity and/or conductance is checked daily with a solution of known salinity or conductance, while temperature is checked daily against an NIST thermometer. The S/C/T meter probe and the NIST thermometer is inserted into 25 ml of the salinity and/or conductance standard (See Table 9.3). A conductivity and/or salinity reading within 5% of the standard value, and a temperature within 0.1 degrees are considered acceptable. Values outside these acceptance criteria will require the unit to be factory calibrated.

9.4.1.2. pH

The pH meter/probe is calibrated before each field day. We use an automatic temperature compensation (ATC) probe to adjust for differences in temperature between standards and samples. Standard pH buffers (pH 7.00, cat. no. SB108-500; and 10.00, cat. no. SB 116-500)are purchased from Fisher Scientific. The two-point calibration procedure is as follows:

1. Choose pH 0.01 mode.
2. Rinse probes (pH combination and ATC) in DIW. Blot dry. Rinse with ca. 2 ml of pH 7.00 buffer. Immerse probes in pH 7.00 buffer.

TABLE 9.4
Instrument Calibration

Instrument	Calibration Type	No. of Standards	Type of Curve	Acceptance/ Rejection Criteria	Frequency
Orion S/C/T Meter					
Conductivity-	Continuing Check	3	Linear	Conductivity within 5% of Standard Value	Daily, prior to use, every 4 hours, and end of each use.
Temperature	Continuing Check	1	Linear	Temperature within 0.1 degrees of NIST thermometer value	Daily
Orion pH Meter	Initial	2	Linear	Reading must be with 0.05 pH units.	Daily, prior to use.
	Continuing	1	Linear	Reading must be within 0.05 pH units.	Every 4 hours, and end of each use.
PSA Merlin Plus Mercury System	Initial	4	Linear	R^2>0.995	Daily, prior to each run
	Continuing	1 Blank 1 Intermed.		Value of Zero 85-115% of initial calibration	Every 10 samples
GC-AFS	Initial	5	Linear	R^2>0.995	Daily, prior to each run
	Continuing	1 Blank 1 Intermed.		Value of Zero 85-115% of initial calibration	Every 10 samples

TABLE 9.4 (cont.)
Instrument Calibration

Instrument	Calibration Type	No. of Standards	Type of Curve	Acceptance/ Rejection Criteria	Frequency
10.025 Millennium Merlin System	Initial	4	Linear	R^2>0.995	Daily, prior to each run
	Continuing	1 Blank 1 Intermed.		Value of Zero 85-115% of initial calibration	Every 10 samples

3. Press Cal button. The meter will display ".1." and the pH value of the buffer; the meter automatically recognizes the pH of the buffer solution. When pH stabilizes, press Enter. The display will freeze for 3 seconds, and then display ".2.".
4. Rinse probes in DIW. Blot dry. Rinse with ca. 2 ml of pH 10.01 buffer. Immerse probes in pH 10.00 buffer.
5. Wait for pH display to stabilize, and press Enter. Display now will say "PH" and be ready for sample measurement.
6. Rinse probe in DIW, place probe in pH 7.00 buffer, and check that pH meter reading is within 0.05 pH units.

In case of low pH level samples, the pH 4.00 and pH 7.00 standards will be used in the calibration procedure.

The response of the pH meter is checked with the pH 7.00 buffer after 4 hours of use and at the end of each use. If the response is outside 0.05 pH units, the two-point calibration is repeated.

9.4.2 Laboratory Instruments

The PS Analytical Merlin Plus and Millennium Merlin mercury instruments are calibrated according to the following procedures:

1. Initial calibration is a four-point standard curve. The range of standards reflect the expected range of sample concentrations. For low level water samples, standards include 0, 2.5, 5 and 10 ppt. Standards run for tissue and sediment samples usually include 0, 25, 50, and 100 ppt.
2. All standards are run in duplicate and plotted on a linear calibration graph using the software inherent to the instrument or a specific Microsoft Excel calculation template.
3. The linear correlation coefficient is checked by the analyst to ensure that it is 0.995 or better. Standard curves outside the acceptable limits are run again, and new standards are prepared if necessary.
4. A second source standard (calibration check) is analyzed at the end of the standard curve with a concentration of 1-2 times the PQL to monitor instrument sensitivity and accuracy.
5. A method blank and a middle level standard are analyzed in duplicate following the standard curve and at a frequency of 5%, thereafter. The blanks should be less than the corresponding MDL and the middle level standard should fall within the 85-115 % of the initial calibration range. If outside the acceptable levels, the run is stopped, and initial calibration is begun again.
6. For solid and tissue samples analyze one QC check standard, in duplicate, per sample set.

Multiple dilutions of the primary standard, as required to make the low calibration standard of 10 ppt, often results in error, thereby producing a standard curve that does not intercept the origin. The

sensitivity of the Merlin Plus System is such that a zero mercury concentration results in a zero result. Therefore, once the standard curve is checked for linearity within the acceptable limits, the curve is dropped parallel through the origin by subtracting the intercept value from all the points. Mercury concentrations in samples are then determined by comparing sample peak heights to the new standard curve.

The results of the standard curve generated, the resultant correlation coefficient, and results of QC check samples for each run are kept with the analytical results in project specific files. Examples of standard curves generated for each matrix (water, sediment, tissue) are included in Appendix D.

The GC-AFS system is calibrated for organic mercury analysis according to the following:

1. A standard curve is created at the beginning of each run by running the following concentrations: 0.0, 0.833, 1.667, 2.500, and 3.333 pg/µL for water samples; 0.0, 1.25, 2.50, 3.75, and 5.00 pg/µL for solid samples.
2. All standards are run in duplicate and plotted on a linear calibration graph using the software inherent to the instrument or a specific Microsoft Excel calculation template.
3. The linear correlation coefficient is checked by the analyst to ensure that it is 0.995 or better. Standard curves outside the acceptable limits are run again, and new standards are prepared if necessary.
4. A blank and a middle level standard are analyzed in duplicate following the standard curve and at a frequency of 5%, thereafter. The blanks should be less than the corresponding MDL and the middle level standard should fall within the 85-115 % of the initial calibration range. If outside the acceptable levels, the run is stopped, and initial calibration is begun again.
5. All samples are run in duplicate.
6. For sediment and tissue samples two duplicate samples and one matrix spike sample are run for every sample.

10.0 Preventive Maintenance

10.1 Routine Maintenance

Preventive maintenance is an essential part of a properly functioning laboratory. Maintenance of field and laboratory instruments are summarized on Tables 10.1 and 10.2. For the Merlin Plus System, maintenance includes running DIW through the instrument for 15 minutes at the beginning and end of each day. The gas separator is cleaned as needed.

10.2 Maintenance Documentation

A use log book is kept on each mercury instrument. Instrument response to calibration standards, the number of samples run, and the hours of instrument use are recorded in each log book. In addition, all maintenance activities for each instrument are recorded in the log book. A record of service performed by the manufacturer or other service contractor is kept in the instrument files.

10.3 Contingency Plans

SERP maintains a stock of spare parts for its analytical instruments. Instruments which can not be fixed by SERP personnel are sent to the manufacturer or other service contractor.

TABLE 10.1 Field Equipment Preventive Maintenance

Instrument	Activity	Frequency
pH meter	Check batteries - recharge Check liquid in probe Replace probes Rinse with analyte-free water	Daily Daily Every 6 to 9 months Before and after each use
Dissolved oxygen meter and S/C/T meter	Check batteries - recharge Check probes	Daily Daily

TABLE 10.2 Laboratory Equipment Preventive Maintenance

Instrument	Activity	Frequency
PSA Merlin Plus	Rinse with DIW for 15 minutes Replace tubing Replace wash water Clean gas separator	Beginning and end of day As needed As needed As needed
PSA Millennium Merlin System	Rinse with DIW for 15 minutes Replace tubing Replace wash water Clean gas separator	Beginning and end of day As needed As needed As needed
GC-AFS	Replace Septum Replace Column Replace Pyrolyzer Replace silanized glass wool in glass linear	Every 100 injections As needed As needed As needed
Analytical Balances	Clean weighing compartment Clean interior/exterior Check calibration Factory service calibration	Daily Monthly Daily Semiannually
Ovens and Refrigerators	Check temperature Calibrate with NIST thermometer	Daily Annually

11.0 Quality Control Checks and Routines to Assess Precision, Accuracy and Calculation of MDLs

SERP uses both field and laboratory QC check samples. Each of these QC check samples are included on Table 11.1.

11.1 Field QC Checks

SERP's field quality control includes the collection of one duplicate sample for every 10 field samples collected. In addition, according to FDEP-QA-001/90, one pre-cleaned equipment blank is prepared for every 10 samples. For sampling events involving 1 to 10 samples, one equipment blank is prepared. This blank is prepared in the field prior to sampling by pouring or rinsing using analyte-free water on each piece of precleaned field sampling equipment. Equipment blanks for surface water samples are collected by running analyte-free, water through the vacuum system then into the sample bottle. For pore water samples, the equipment blank is prepared by running DIW through the peristaltic pump then into sample bottles. For soil and sediment samples, the equipment blank is prepared by pouring DIW over the sampling equipment and into the appropriate sample bottles. All duplicate and blank samples are placed in appropriate bottles with the corresponding preservatives for each analysis. The collection of blank samples are recorded in the field note book. For field equipment cleaned in the field, an additional equipment blank is prepared following the field cleaning procedures at a frequency of one per sampling event or one every 10 samples, whichever is greater. The time and number of all equipment blanks are recorded in the field note book.

Field instrument checks are completed prior to each sampling event, once every four hours of operation and at the end of the field sampling event. The results of the field QC checks are recorded on the Field Instrument Calibration Sheet. Field equipment not functioning properly are not used to collect data until they are brought back to the laboratory for maintenance and recalibration. Duplicate field equipment and probes are kept on hand in the laboratory if needed.

11.2 Laboratory QC Checks

SERP's standard laboratory QC checks includes blanks, replicates, matrix spikes and QC standards and QC check samples. Method reagent blanks consisting of analyte-free water are prepared exactly like a sample and run prior to each instrument calibration. As standard practice SERP usually analyzes all samples in replicate. In the laboratory, water samples are split into two bottles, and each bottle is analyzed three times for a total of six data points for one sample location. One of every ten water samples has two additional bottles prepared so that two bottles are analyzed asreplicates and two bottles are spiked to serve as matrix spikes. For total mercury determinations, two ampoules are prepared for each sediment and fish sample, and each ampoule is analyzed two times. For organic mercury determinations, four replicate samples are prepared for each fish and sediment sample, with two run as replicate samples and the remaining two are spiked to serve as matrix spikes.

Table 11.1
Quality Control Checks

Type	Description	No. of samples per event	Frequency (all parameter groups unless specified)
Field			
Equipment Blank (non-field cleaned equipment)	Fill or rinse all pre-cleaned sampling equipment (tubing, syringes, filter holders, etc.) with analyte-free water, fill appropriate sample containers and preserve according to each analysis.	< 10 > 10	1 prepared on-site at the beginning of the sampling event 1 prepared on-site at the beginning of the sampling event, and after every 20 samples
Equipment Blank (field cleaned equipment)	If equipment is cleaned on-site, then prepare additional equipment blank sample by filling or rinsing the field-cleaned equipment with analyte-free water, filling the appropriate sample containers and preserve according to each analysis.	< 10 > 10	1 at the beginning and end of the sampling event 1 after every 20 samples or 5 % (whatever is greater)
Field Duplicate	A duplicate sample collected and analyzed for the same parameters as the original sample.	1 - 10 > 10	1 sample is collected in duplicate 1 after every 10 samples or 5 % (whatever is greater)
Field Measurements QC Check Standards pH meter	Record the results of calibration check standards for all field measurement equipment. Record two or more pH readings in field note book until sequential values are within 0.02 pH units.	1 or more 1 or more	Beginning of each sampling event, once every four hours, and again at end of the sampling day Every sample.

TABLE 11.1 Continued.
Quality Control Checks

Type	Description	No. of Samples per Event	Frequency (All parameters unless specified)
Laboratory			
Method Reagent Blank	Analyte-free water	1 or more samples	1 at beginning of a run, after every 10 samples, and at the end.
Replicate Samples	Re-analysis of a sample	1 or more samples	Every sample is analyzed 2 to 4 times
Continuing Calibration Standards	One standard at a level of 1 to 2X the PQL (included in the standard curve) and one intermediate standard	1 or more samples	Analyzed at the beginning of each run, and at a frequency of 5%, thereafter.
Matrix Spikes	One sample from a set (not blanks) is split in two, and one of the duplicates is spiked with a known concentration (3 to 5 times higher than the original expected concentration) prior to sample preparation.	1 or more samples	1 sample in a set or at a frequency of 5%, whichever is greater. Except for organic mercury in sediment and tissue samples, 1 matrix spike is performed on every sample.
Quality Control Check Standards	Standards from an independent source that are certified and traceable (i.e. NIST). Can be interchanged as one of the continuing calibration check standards.	1 or more samples	Analyzed at the beginning of each run to check the initial calibration of the standard curve.
Quality Control Check Samples	Samples of known analytical concentration that are submitted blind to the analyst. These samples are either prepared in house or obtained from an independent source.	1 or more samples	Analyzed in duplicate quarterly.

Matrix spikes samples are prepared by adding a known concentration approximately 2 to 5 times the original expected concentration (*e.g.* 1 ppt for total mercury water samples) to the duplicate bottles or ampoules. The concentrations from the unspiked duplicate is subtracted from the spiked result and the percent recovery is determined by comparing the remainder to the known spike concentration.

Continuing calibration standards (intermediate level) are run at the beginning of each run and at a frequency of 5% thereafter. A QC check standard (a certified standard from an independent source) is run at the beginning of a run to check the calibration curve. A quality control check standard is typically an intermediate or high end standard.

A Standard Reference Material sample (if available) is included in every run to check the accuracy of the analytical procedure. Quality control check samples are prepared in-house or from an NIST or other certifying source and are submitted blind to the analyst on a quarterly basis to check instrument and user performance. If the blind QC check sample results is not acceptable, the results will be reported in the QA report to FDEP.

11.3 Routine Method Used to Assess Precision and Accuracy

Precision and accuracy of each analytical parameter determined in the laboratory is determined on a daily basis. Precision is defined as the agreement or closeness of two or more results. As stated above, SERP performs 2 to 8 replicate analyses on the same sample. For samples analyzed 3 or more times, SERP usually determines the mean (X) and standard deviation (SD) of the replicate analyses and estimates precision in terms of percent relative standard deviation (% RSD) using the following equation:

$$\%\ RSD = \frac{SD}{X} * 100$$

The acceptance criteria for % RSD depends on the analyte/matrix combination (See Table 5.2).

The Relative Percent Difference (RPD) is another parameter used to monitor the precision of our analytical results and it is calculated for Matrix Spike duplicates and/or sample duplicates. The acceptance criteria is usually RPD <= 20 - 30 % (See Table 5.2 for specific analyte/matrix targets).

Replicate results that are outside the +/-2SD range are automatically eliminated from the calculations by the corresponding Microsoft Excel template to ensure precision and reliability of the final results.

Accuracy is defined as the agreement between the analytical results and the known concentration. Accuracy is determined by running matrix spikes (MS) and/or standard reference materials (SRM) and is determined as percent recovery (% R) according to the following equations:

$$\%\ R = \frac{Cs - Cu}{MS \quad S} * 100$$

Where:

Cs = concentration of spiked sample
Cu = concentration in unspiked sample
S = expected concentration of spike in sample
%R = percent recovery

$$\%\ R_{SRM} = \frac{\text{Sample Concentration}}{\text{True Value}} * 100$$

The control limits for accuracy are +/- three standard deviations of the historical percent recovery average , with warning limits set at +/- two standard deviation. When no historical limits are available SERP usually uses an acceptance limit of 75-125% of the expected value (See Table 5.2).

The results obtained for each quality control check are compared to their acceptable limits for precision and accuracy on a daily basis. New limits (both control and warning) based on historical data are calculated on a quarterly basis .

11.4 Method Detection Limits

Method detection limits (MDLs) have been determined according to the EPA procedure described in 40 CFR Part 136, Appendix B, revision 1.11, except that a multiplier of 3 is used instead of the Student's t value. Specifically, seven or more replicate samples containing an analyte at a known low concentration are analyzed according to the appropriate analytical procedure for that analyte. A standard deviation for the replicates is determined and the MDL is computed as 3 times the standard deviation. The practical quantitation limit (PQL) is defined as 12 times the standard deviation. MDLs and PQLs are routinely verified/updated on a yearly basis.

12.0 Data Reduction, Validation and Reporting

12.1 Data Reduction

Data reduction is not necessary for field data, as field measurements are read directly from the field instruments in their appropriate reportable units. The pH meter and SCT meter are automatically compensated for temperature.

All data reduction (both field and laboratory) is performed according to the protocols specified by the analytical methods listed in Section 5. Each technician is responsible for data entry of field data from the field notebook into Microsoft Excel spreadsheets. Instrument produced peak heights are converted to concentrations by comparison to the standard curve by the analytical instrument. Sediment and tissue samples are corrected for dilutions and for the weight of the sample by the analyst.

Replicate sample results are further reduced to provide one data point per sampling location. The mean and standard deviation of each sample result is calculated using Microsoft Excel spreadsheets. Any replicate result that is two standard deviation away from the mean (+ or -) is removed from the replicate data set.

12.2 Data Validation

The Chief Chemist and analytical technicians are responsible for the collection, custody, storage, and analysis of all of the samples. It is their responsibility to check for sample integrity and that the samples are analyzed within the appropriate holding times. They are also responsible for the proper maintenance of all equipment and cleaning of laboratory glassware. They provide the first check on instrument calibration, method blanks, and equipment blank results, and ensure that all method specifications have been met. If problems arise during an analysis, such as failure of proper equipment calibration, or unusual sample results, it is their responsibility to verbally notify the laboratory director as soon as possible.

The QA Officer is responsible for a second check of instrument calibration by comparing the present instrument responses to historical values. The QA Officer checks all sample preparation and instrument logs. In addition, the QA officer checks the results of method blanks, equipment blanks, sample replicates, matrix spikes and field calibration checks and determines that the instrument precision and accuracy is within the QA objectives listed in Section 5. Obvious anomalous results are subject to re-analysis.

Dr. Jones, the director, is responsible for the final review of all data and documents that are submitted to the client (EPA, DOI, NPS, FDEP, SFWMD). Due to his extensive experience in analytical chemistry, Dr. Jones can apply both objective and subjective techniques to data review. From his knowledge of analytical chemistry, Dr. Jones can interpret the data in its environmental context. In addition, through his collection of historical data in South Florida, Dr. Jones can identify potential outliers in a data set.

12.3 Data Reporting

Once the instrument calibration and sample results have been validated, they are entered into input data files. The laboratory technicians are responsible for entering the raw data into the spreadsheet and providing a first check of data entry. The QA officer provides a second check of the input data, spreadsheet calculations and output file formats. Once all of the data has been validated, the QA officer will provide a written statement of validation along with the data report.

For data reports issued to the client for DEP related work, or for reports issued to DEP, the following information will be included:

a. Laboratory name, address, and phone number
b. Client name and/or site name
c. CompQAP number
d. Client or field identification number
e. Sample identification number
f. Method name and number/reference of each analysis
g. Analytical result with applicable data qualifiers
h. Date of sample preparation
i. Time of sample preparation if holding time is less than 48 hours
j. Date of sample analysis
k. Date and time of sample collection
l. Identification of all laboratories providing analytical results, including their CompQAP number

A copy of the final data report is included as Figure 12.1.

12.4 Data Storage

Data input files and final report files are stored on hard drive and floppy disk using names that readily identify a sampling event. Files labeled by sampling event are stored in a locked file cabinet with limited access by SERP employees only. These files contain hard copies of the file input and output as well as all raw laboratory data sheets and field note book sheets. Raw laboratory output data sheets are identified with a date, analysis, analyst initials, and sampling event number. Pursuant to Chapter 62-160 F.A.C., all records will be maintained for a minimum of 3 years.

FIGURE 12.1
Final Data Report

Southeast Environmental Research Program **Florida International University**
Mercury laboratory **OE 148 Miami, FL 33199**
CompQAP No.: _____ **(305) 348-3095**

Client Name: _____
Site Name: _____
Matrix (water, sediment, fish): _____

Sample ID	Bottle #	Sampling Date	Sampling Time	Sampling Depth (m)	Sample Prep Date/Time	Sample Anal. Date/Time	Parameter Name/SOP#	Result /units	QA Code

Other Laboratories providing analytical results:
Lab :_____ CompQAP # :_____ Analyses:_____

Lab :_____ CompQAP # :_____ Analyses:_____

SERP Lab Manager: _____

13.0 Corrective Action

Corrective action is taken whenever the quality assurance objectives have not been met. A summary of the corrective actions for the laboratory and for the field are included in Tables 13.1 and 13.2, respectively.

The analyst, either the Chief Chemist or the technicians, are responsible for providing a first check for compliance, and initiating corrective action procedures as described in Table 13.1. The QA Officer is responsible for a second check for compliance, and initiating corrective action as appropriate. If problems continue, then the analyst and/or QA officer will notify the laboratory director immediately, who may initiate further steps in solving the problem.

Any corrective action taken will be documented in one of the following: analyst log books, instrument log books, or project-specific files. Samples requiring reanalysis will be noted on the analysis sheet.

FDEP recommended corrective action will be initiated as a result of systems or performance audits, split samples or data validation review.

TABLE 13.1
Corrective Actions for the Laboratory

QC Activity	Acceptance Criteria	Recommended Corrective Action
Initial Instrument Blank	Instrument response <MDL	Prepare new blank, if same response determine cause of contamination: reagents, environment, equipment failure; notify Dr. Jones and QA Officer
Initial Calibration Standards	Linear response with R>0.995	Reanalyze standards, if same response, reoptimize instrument, if same response, prepare new standards, notify Dr. Jones and QA Officer
Matrix Spikes	See Table 5.2 and specific SOPs in Appendix C	Reanalyze matrix spike, if same response, prepare and run a new matrix spike, if same response, notify Dr. Jones and QA Officer.
QC Check and Continuing Calibration Standards	See Table 5.2 and specific SOPs in Appendix C	Reanalyze check standard, if same response, prepare new check standard, if same response, prepare new primary and calibration standards, notify Dr. Jones and QA Officer.
Replicate Sample	See Table 5.2 and specific SOPs in Appendix C	Determine cause: baseline drift, carryover, etc. Reanalyze all samples between duplicates, notify Dr. Jones and QA Officer.
Duplicate Sample	See Table 5.2 and specific SOPs in Appendix C	Reanalyze duplicates, reanalyze all samples between duplicates; notify Dr. Jones and QA Officer

TABLE 13.2
Corrective Actions for the Field

QC Activity	Acceptance Criteria	Recommended Corrective Action
Initial Calibration Standards	Value within +/- 5% of expected value	Reanalyze standards, if same response, optimize instrument, if same response, use new standards; notify Dr. Jones and QA Officer.
QC Check Standards	Value within +/- 2 standard deviations of the historical value	Reanalyze QC check standard, if same response, prepare new QC check standard, if same response, recalibrate; notify Dr. Jones and QA Officer.
Equipment/Trip Blank	Value <MDL	Reanalyze blanks: if same response, check recorded cleaning procedures and mark sample trip results for affected and related parameters questionable or invalidate data, as required; notify Dr. Jones and QA Officer.
Duplicate Samples	Value within +/- 1 standard deviation of mean.	Reanalyze duplicates: if same response, mark sample trip results for affected and related parameters questionable or invalidate data as required. If reanalysis show Field Collection to be acceptable, reanalyze all samples analyzed with the Field samples the first time. Notify Dr. Jones and QA Officer.

14.0 Performance and System Audits

Dr. Jones supervises all aspects of field and laboratory activities. He requires the laboratory and all instrumentation to be clean and working at optimum conditions. He is knowledgeable on the inner-workings of each instrument and checks on their performance as well as on the performance of the laboratory personnel continually.

14.1 System Audits

14.1.1 Field Audit

A field audit is conducted at least on an annual basis by the QA Officer. During these audits, the QA Officer will review and evaluate the various components of the measurement system to determine their proper selection and use according the specific Project Quality Assurance Plan. Specifically, the auditor will provide a detail review of sampling technique, field instrument calibration, field decontamination procedures, sample custody, sample preservation, and field note book documentation. The checklist included as Figure 14.1 will be used during the field audit, any discrepancies or deviations will be noted in the checklist and corrected immediately. At the end of the audit, the QA officer will date and sign the checklist stating that the audit was completed, and a copy of the checklist will be put in the project-specific files.

14.1.2 Laboratory Audit

A laboratory system audit is conducted on a weekly basis by the Chief Chemist. These audits consist of an evaluation of all laboratory activities. The Chief Chemist inspects that the procedures and documentation for sample log-in, sample preparation, and sample preservation are appropriate for the methodology and Project QA plan. In addition, the Chief Chemist checks that the samples are analyzed within their appropriate holding times, and that the instrumentation is properly calibrated and that the appropriate type and number of QA samples are run. The checklists included as Figure 14.2 is used during the audit. Deficiencies found during the audit are documented on the checklist as well as in one of the following as considered appropriate for the deficiency: sample log, instrument log, sample preparation log. A copy of the checklist is kept in the project specific files as well as in the QA Officer's notebook.

14.2 Performance Audits

Laboratory performance audits take place continually or at least on a weekly basis. A portion of these audits are conducted by all people in the laboratory including the analyst, Chief Chemist, and QA Officer. Instrument performance is checked continually by the analyst with analysis of standard curves, sample replicates, method blanks, and equipment blanks. Many of the analyses are performed and checked within 24 hours to seven days of sample collection, allowing for any deficiencies to be corrected and samples re-analyzed if needed. The results of these performance audits conducted by

Figure 14.1 Field Audit Checklist

Field Audit Checklist

Auditor: _____ Date of Audit: _____

Y	N	Water Sample Collection	Comments
		* Sampling station advanced from downgradient direction.	
		* A clean person is designated and dons two pair of gloves.	
		* Other crew members don one pair of gloves.	
		* Vacuum system tubing is rinsed three times with sample water prior to sample collection.	
		*Sample is properly labeled, double-bagged, and stored in a cooler.	
		* Microcentrifuge tubes are stored in the dark in a cooler with ice.	
Y	**N**	**Soil/Sediment Sample Collection**	**Comments**
		* Sampling station advanced from downwind direction.	
		* A clean person is designated and dons two pair of gloves.	
		* Sampling equipment clean prior to each sample collected.	
		* Sample are homogenized in the field appropriately.	
		*Sample containers are labeled correctly, bagged, and placed in a cooler.	
Y	**N**	**Porewater Sample Collection**	**Comments**
		* Volume of standing water in the lysimeter is determined correctly.	
		* Three volumes of standard water are purged.	
		* Field parameters are monitored during purging until stable.	
		* Sampling equipment tubing and sample bottles are rinsed three times prior to sample collection.	
		*Clean person dons two pairs of gloves.	
		* Sample containers are labeled correctly, bagged, and placed in a cooler.	

Figure 14.1 Field Audit Checklist (Continued)

Field Audit Checklist (cont.)

Auditor: _____ Date of Audit: _____

Y	N	Tissue Sample Collection	Comments
		* Sample station advanced from downstream direction.	
		*Clean person dons two pairs of gloves.	
		* Fish samples are labeled correctly, bagged, and placed in a cooler.	
Y	**N**	**Field Notebook**	**Comments**
		The following are recorded in the field notebook:	
		* Names of the field crew	
		*Weather conditions	
		* Time of sample collection	
		* Time of QC sample collection	
		*Temperature, Salinity, conductivity, pH of purge water	
		* Volume of water purged.	
		* Sample numbers and station names.	
		* Field equipment cleaning procedures.	
		* Soil/Sediment sample homogenization procedure.	
		* The use of fuel powered units.	
		* Number of QC samples collected.	
Y	**N**	**General**	**Comments**
		* Samples are collected away from engine exhaust.	
		* Appropriate number of QC samples are collected.	
		* Samples are brought to the laboratory on the same day of sample collection.	

Figure 14.1 Laboratory Audit Checklist

Laboratory Audit Checklist

Auditor: _____ Date of Audit: _____

Y	N	Instrument Response	Comments
		* Instrument notebook is up to date.	
		* High standard within 10 % of expected value.	
		* Calibration curve correlation coefficient better than 0.995	
		* Instrument blank is less than MDL.	
		* QC check standard within control limits.	
		* Matrix Spike samples within control limits.	
		* Replicate samples show RPD < 20%	
		* Duplicate samples show RPD < 20%	
		* Instrument maintenance up to date.	
		* Equipment Blank samples less than MDL.	
Y	**N**	**Sample Tracking**	**Comments**
		* Sample Checklist is filled out correctly.	
		* Samples are kept in proper storage	
		* Samples are analyzed within holding times.	
		* Samples are not discarded until QA checked.	
Y	**N**	**Labware**	**Comments**
		* Reagents are stored appropriately.	
		* Waste is disposed properly.	
		* Glassware and bottles are cleaned appropriately.	
		* Check the age of sample bottles.	
		* Standards are labeled and stored appropriately.	
		* Check dates of standards.	

Figure 14.2 Laboratory Audit Checklist (Continued)

Laboratory Audit Checklist (cont.)
Auditor: _____ Date of Audit: _____

Y	N	Data Management	Comments
		* Project Files up to date.	
		* Data input up to date and correct.	
		* Calculations performed correctly.	
		* Output files in correct format.	
		* Data values within 2 standard deviations of historical mean.	
		* Data reports up to date.	

the analyst are on instrument log books and instrument printouts on a daily basis.

The Chief Chemist checks the instrument log books on a weekly basis to check that instruments are running within their appropriate QA objectives. The Chief Chemist may also prepare samples and submit them blind to the analyst to check instrument and user performance. The results of these samples are documented in the Chief Chemist notebook.

The QA Officer will review the results of equipment blanks, matrix spike samples, blind samples, sample replicates, and split samples from another laboratory on at least a semi-annual basis. The QA Officer will determine if instrument accuracy and precision values are within the project QA objectives. The results of the QA Officer's review will be documented in the QA Officer's notebook. Any discrepancies determined between instrument performance and the QA objectives' will be included in the QA report to FDEP.

Currently, SERP is not involved in a regular external audit program; however, we are available to receive on-site audits by FDEP at any time.

15.0 Quality Assurance Reports

SERP will submit quality assurance reports for all Quality Assurance Project Plans at a frequency according to Table V of Appendix D of the FDEP QA Manual. The QA Officer is responsible for the preparation of these reports. In general, if no audits were performed and no significant QA/QC problems have been identified, then SERP will prepare a brief letter stating these facts in lieu of a detailed quality assurance report.

A detailed QA report will be prepared when:

1. Activities were conducted in a manner other than those described by the CompQap or QAPP.
2. Preservation or holding requirements were not met.
3. Quality control checks were unacceptable.
4. Precision, accuracy, or MDL objectives were not met.
5. Corrective action was taken.
6. Internal or external audits were conducted and discrepancies were noted.

According to FDEP guidelines, these QA reports will include the following:

1. Title Page including the time period of the report, the QA Project Plan Title and Plan number, the laboratory name, address and phone number, and the preparer's name and signature.
2. Table of contents if the report is over 10 pages long.
3. The results of performance or system audits to include, date of audit, system tested, name of auditor, parameters analyzed, results of tests, deficiencies or failures, and an explanation of the problem and the corrective action taken.
4. Significant QA/QC problems.
5. Corrective actions taken.

APPENDIX A

Method Validation for Part per Trillion (ppt) Concentrations of Inorganic and Total Mercury in Water, Solid, and Tissue Samples

Limited Use Method Validation

Prepared by and for:

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Ronald D. Jones, Ph.D. Date
SERP Director and Professor

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Nancy A. Black Date
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FDEP QA Officer

1.0 Scope and Application

This method covers the determination of parts per trillion (ppt) levels of total and inorganic mercury in water, soils, sediment, and tissue (fish) samples. A method detection limit of 0.3 ppt is obtainable for water samples at a precision of less than 5% relative standard deviation (%RSD) and an accuracy between 90 and 110%. The concentrations of mercury in sediment and tissue samples are significantly higher than in water samples and can be determined at better precision (<5 %RSD) and accuracy (90 to 110 %R). Linear calibrations up to 1 part per million (ppm) can be obtained and higher concentrations can be measured with dilution.

2.0 Summary of Method

Total and inorganic mercury concentrations are determined by Cold Vapor Atomic Fluorescent Spectrometry (CVAFS). In this method, mercury in a liquid sample is vaporized and stripped from the remaining liquid by a carrier gas (Argon). A sheath gas (also Argon) constrains the mercury vapor to a small stream as it passes by a light source and photomultiplier tube. The mercury concentration is determined by atomic fluorescence.

An PS Analytical (PSA) Merlin Plus mercury analysis system equipped with an autosampler, vapor generator, fluorescence detector and a PC-based integrator package is used to detect total and inorganic mercury. The system is run according to the manual provided by the manufacturer, except lower flow rates are used for the carrier and sheath gases. Low detection levels of 0.3 ppt Hg in water samples are obtained with flow rates of 0.14 L/min and 0.125 L/min for the carrier and sheath gases, respectively. For the higher concentrations of mercury detected in sediment and tissue samples, flow rates of 0.35 L/min for the carrier gas and 0.2 L/min for the sheath gas are used. To accurately regulate the gas flow for optimum conditions, SERP installed an Omega model FMA-78P2 electronic mass flow controller in front of the PSA Merlin Plus instrument.

Water sample preparation includes digestion by a brominating procedure to breakdown all mercury complexes. Sediment samples and tissue samples are digested with concentrated nitric acid in sealed ampoules, and subsequently autoclaved.

An NBS standard of 1000 μg Hg/l is used and diluted to obtain the appropriate linear standard curve. High standards of 10 ppt and 400 ppt are used for water/tissue and sediment samples, respectively. NBS oyster tissue 60 ng/g (566a), NRCC dogfish muscle 4.64 μg/g (DORM-2), NIST sediment nominal 50 μg/g (8407), and NIST sediment 60 ng/g (8406) are used to check the accuracy of the tissue and sediment results.

3.0 Interferences

3.1 Matrix Interferences

There are no matrix interferences with this method. By digesting the water samples with bromine, all mercury complexes including organomercury compounds, sulphide complexes and complexes with organic material (e.g. fulvic acids) are broken down. Additionally, acid digesting and autoclaving results in a complete breakdown of mercury in the sediment and tissue samples.

3.2 Environmental Interferences

Mercury contamination at levels near the method detection limit is a consistent problem as samples easily absorb mercury from the air and improperly cleaned glassware. To minimize contamination, all technicians are required to wear surgical vinyl gloves. In addition, all glassware, acids, reagents, etc. are stored in a mercury-free clean room. The clean room contains a bank of laminar flow hoods equipped with gold and charcoal filters. The floor is covered with flypaper to trap particulates. Potential contamination in the clean room is checked weekly by monitoring acidified (1% HCl) water samples, which are stored open inside the clean room. If significant levels of mercury is detected in these samples (<20 ppt), then the source of the mercury contamination is identified and eliminated. The gold and charcoal filters within the laminar flow hoods are reconditioned if necessary.

Mercury-free DIW is produced by filtering tap water through a Culligan system consisting of activated charcoal and two mixed bed ion exchange cartridges. This water is piped to the clean room where is then passed through a Barnstead Mega-ohm B Pure system. This system is fitted with two filters (Thermolyne: colloid/organic-D0835, and ultrapure-D0809) in line with a 0.22 micron pleated particle filter. Mercury levels are not detectable (<0.1 ppt) in this water by both our laboratory and by and independent laboratory analysis. This is the only water used for all analyses.

3.3 Laboratory Glassware and Sample Bottle Cleaning

Laboratory glassware is kept to a minimum, with Teflon bottles and beakers used when possible. All reusable glass bottles, volumetric flasks, graduated cylinders, and teflon beakers are dedicated to the preparation and storage of a specific reagent, and are rinsed between usage with acid (0.5N HCl and 0.05N HNO_3) and rinsed three times with DIW. One volumetric flask is kept dedicated to making the primary standard and is rinsed only with this standard. Glassware or plastic containers that have come in contact with samples, such as ampoules and scintillation vials are used once then discarded.

Teflon sample bottles that have been used previously are rinsed three times with DIW and filled with 1% HCl. After filling, 1 ml of mixed brominating agent is added for every 50 ml, and the bottle is shaken. This mixture remains in the bottles until used. Prior to their use, 500 µl of hydroxylamine hydrochloride is added to remove the free bromine. The bottle and cap are then rinsed three times with DIW. Sediment cups and 20 ml scintillation vials are non-reusable and

discarded.

4.0 Safety Precautions

Bromine vapors are toxic; therefore, prepare the brominating agent beneath a hood. Keep all containers with the brominating agent securely capped when moved or stored. Neutralize sample bottles with hydroxylamine hydrochloride prior to their use.

Hydroxylamine hydrochloride is a skin and eye irritant and can cause dermatitis. A face shield and gloves must be worn when handling this material.

The exhaust fumes from the atomic fluorescence spectrophotometer are toxic and must be ducted away. The low pressure mercury discharge lamp used for fluorescence determination emits intense U.V. radiation. This lamp must not be viewed directly.

5.0 Apparatus and Materials

Analytical instrumentation includes an PSA Merlin Plus mercury analysis system equipped with the following:

Autosampler	Vapor Generator
Fluorescence Monitor	IBM compatible computer system

In addition, an Omega model FMA-78P2 electronic mass flow controller is installed in front of the PSA Merlin Plus instrument. All of the above equipment is stored beneath a protective hood. All other standard laboratory equipment (Teflon beakers, glassware, pipettes, etc.) is kept within the clean room and dedicated to mercury analysis. A mercury-dedicated refrigerator, oven, and analytical balance are kept within the clean room. Polyethylene scintillation vials (20 ml) with polypropylene caps are used with the autosampler. Water samples suspected of low mercury concentration are injected into the instrument manually from 125 ml teflon bottles.

Additional equipment needed for sediment and tissue sample preparation include:

glass bottled blender	autoclave
syringe	oven
plastic specimen cups	balance
10 ml ampoules	

6.0 Reagents

6.1 Bromination Reagents

0.1 M Potassium Bromate
Heat 8.385 g $KBrO_3$ overnight in a glass scintillation vial (Kimble 74511) at 250^0 C +/- 20^o C in a furnace to remove mercury. After cooling dissolve the potassium bromate in 500 ml of DIW and store in a borosilicate glass bottle with a Teflon cap. Prepare Weekly.

0.2 M Potassium Bromide
Heat 11.9 g KBr overnight in a glass scintillation vial at 250^o C +/- 20^o C to remove mercury. After cooling dissolve the potassium bromide in 500 ml of DIW and store in a borosilicate glass bottle with a Teflon cap. Prepare weekly.

0.05 M Potassium Bromide (KBr) - 0.1 M Potassium Bromate ($KBrO_3$)
Mix equal volumes (100 ml) of bromate and bromide in a borosilicate glass (150 ml) bottle with a Teflon cap. Prepare daily.

6.2 Hydroxylamine Hydrochloride

Dissolve 6 g of $NH_2OH{\cdot}HCl$ in 50 ml of DIW in a 60 ml Teflon bottle. Prepare weekly.

6.3 Stannous Chloride

Add 50 ml of 12 N HCl to 40 g of Stannous Chloride ($SnCl_2$). Bring to 1000 ml with DIW in a borosilicate glass bottle with a Teflon cap. Stannous chloride is purged of any traces of mercury with argon gas continuously throughout the analysis. Prepare fresh daily.

6.4 12 N Hydrochloric Acid

Concentrated HCl (12 N HCl) is dispensed via a pipette or poured into a graduate cylinder, either of which has been previously acid washed and rinsed three times with DIW.

6.5 16 N Nitric Acid

Concentrated Nitric acid (16 N) is dispensed through a pipette, which has been previously washed and rinsed three times with DIW.

6.6 Standards

Due to its high concentration, primary stock standard is prepared and stored outside of the mercury-free clean room. The primary stock standard is made by addition of 100 μl of SEPEX (PLHG4-2X) (1000 μg/ml) to 1000 ml of DIW with 10 ml of trace metal grade HCl in a glass, volumetric flask with a ground glass stopper. This standard is prepared daily. Secondary standards are prepared daily in 500 ml Teflon bottles by adding concentrated HCl (5 ml) to 495 ml of DIW. The primary stock is brought into the clean room and 50 μl - 250 μl (depending on final concentration of 10 ppt - 50 ppt) is added to the bottles containing the water-acid mixture with a pipette.

6.7 Standard Reference Material

The following standard reference materials are used to check the accuracy and precision of the tissue and sediment digestion methods and analyses:

DORM-2 Dogfish muscle (NRCC)
556a Oyster tissue (NBS)
8406 Sediment (NIST)
8407 Sediment (NIST)

These standard reference materials are stored and dried according to the Certificate of Analysis accompanying each standard. The standards are digested following the same procedures used for tissue and sediment samples. They are prepared and analyzed on a daily basis.

7.0 Calibration

A linear calibration curve is run prior to each sample run. For low level water samples, standards include 0, 2.5, 5 and 10 ppt. Standards run for tissue and sediment samples include 0, 100, 250, and 400 ppt. All standards are run in replicate and plotted using the software inherent to the instrument. Linear regression analysis is used to determine the best-fit calibration line with a regression coefficient (r^2) of 0.998 or better. Standard curves outside the acceptable limits are run again, and new standards are prepared if necessary.

The sensitivity of the Merlin Plus System is such that a zero mercury concentration results in a zero result. However, multiple dilutions of the source standard as required to produce the low standard of 10 ppt, often results in error, thereby producing a standard curve that does not intercept the origin. Therefore, once the standard curve is checked for linearity, a new linear calibration curve is recalculated using the high standard and forcing the intercept through zero. Mercury concentrations in samples are then determined by comparing sample peak heights to the new standard curve.

8.0 Quality Control

8.1 Method Detection Limit

The method detection limit (MDL) of 0.3 ppt is determined for this method according to the EPA procedure described in 40 CFR Part 136, Appendix B, revision 1.11, except that a value of 3 is used in place of the Student's T value. Specifically, seven or more replicate samples containing a known, low concentration of mercury are analyzed. The standard deviation of the replicate analyses is determined and the MDL is computed as 3 times the standard deviation.

Table A.1
Method Detection Limit

Matrix	Hg Concentration (ppt)	Mean	Standard Deviation (S)	MDL = 3 x S
Spiked DIW (DIW plus 1ppt Hg)	2.315 2.093 2.216 2.167 2.290 2.093 2.118	2.185	0.085	0.255
Sediment	84.30 ppb 85.42 ppb 82.44 ppb 81.32 ppb 84.30 ppb 82.44 ppb 82.81 ppb	83.29 ppb	1.42 ppb	4.27 ppb

8.2 Precision

Precision is defined as the agreement or closeness of two or more results of the same sample, and is determined in terms of percent relative standard deviation (% RSD) using the following equation:

$$\% \text{ RSD} = \frac{s}{X} * 100,$$

where, s and X represent the standard deviation and mean, respectively, of two or more results of

the same sample. The analytical precision of this method is variable with more precise measurements obtained at higher mercury concentrations.

Table A.2
Precision

Matrix	Hg Concentration	Precision %RSD
Water	0.3 - 30 ppt	< 5%
Water	5 - 500 ppt	< 5%
Water	1 - 1000 ppt	< 5%
Soil/Sediment Slurry	1 ppt - 400 ppt	< 5%
Tissue Slurry	1 ppt - 400 ppt	< 5%

8.3 Accuracy

Accuracy is defined as the agreement between the analytical results and the known concentration. Accuracy is determined by running continuing calibration standards or by check standards and is determined as percent recovery (%R) according to the following equation:

$$\%\ R = \frac{\text{Observed Standard Concentration}}{\text{Known Standard Concentration}} * 100.$$

Instrument accuracy is determined on a daily basis by performing multiple analysis (5 or more replicates) of the high standard. Percent recoveries between 90 and 110% are obtained for water samples with a high standard of 10 ppt. For solid and tissue samples with a high standard of 400 ppt, percent recoveries between 95 and 105% are obtained.

Table A.3
Accuracy

Matrix	Hg Concentration	Accuracy (% Recovery)
Water	0.3 - 30 ppt	90 - 110
Water	5 - 500 ppt	90 - 110
Water	1 - 1000 ppt	90 - 110
Soil/Sediment Slurry	5 - 400 ppt	90 - 110
Tissue Slurry	5 - 400 ppt	90 - 110

8.4 QC Checks

Calibration check samples are run in duplicate following at the beginning and after every 10 samples. For solid and tissue samples, NIST or NBS standards are run after every 20 samples. Accuracy for each of these calibration check samples and standards must be within 90 to 110% or the samples must be run again.

9.0 Sample Collection, Preservation, and Handling

9.1 Water Samples

Water samples are collected in Teflon bottles. Collection is done while wearing shoulder length polyethylene gloves over a pair of vinyl gloves. Surface water samples are collected through a 105 μm nylon screen via a vacuum system to reduce the amount of sediment collected. Samples are double bagged in zip-lock polyethylene bags and placed in a plastic ice chest/cooler used exclusively for low level mercury samples. Samples are returned to the laboratory upon the same day of sample collection and preserved in the mercury-free clean room with 0.5 ml of trace metal grade HCl per 100 ml of sample. Sample analysis within 28 days is recommended.

9.2 Sediment Samples

Sediment samples are collected in polyethylene specimen cups (Elkay non-sterile wide mouth specimen cups with screw caps - 128 ml volume) and placed in polyethylene zip-lock bags. All field samples are kept in a cooler used exclusively for low level mercury samples until they are returned to the laboratory, where they are stored in a freezer. Samples can be stored indefinitely within the freezer; however, analysis within 28 days is recommended.

9.3 Tissue Samples

Fish are collected with a dip net and stored in plastic bags for transport to the laboratory. In the laboratory fish are stored within the freezer indefinitely; however, analysis within 28 days is recommended.

10.0 Sample Extraction/Preparation

10.1 Water Samples

Water samples analyzed for inorganic mercury do not require additional sample preparation prior to analysis. For analysis of total mercury, the samples are placed in an ultraviolet cabinet for 12 hours, allowed to cool, then 2.5 ml of $KBrO_3$/KBr solution is added to 125 ml of each water sample. The sample is left to brominate for one hour in the Hg-free room; after which 500 µl of hydroxylamine hydrochloride is added to the solution to inhibit further reaction. Samples are permitted to settle for at least 10 minutes before analysis.

10.2 Soils and Sediment Samples

Preparation of soil/sediment samples is done outside the mercury-free room, due to their high mercury concentrations. Soil/sediment samples are homogenized and slurried in a glass bottle blender. A mixture of 120 cc of soil/sediment and 50 ml of DIW is blended for three minutes. With a syringe, 10 ml of the slurry is collected for dry weight determination. Another 10 ml is pipeted into a polyethylene specimen cup and diluted to 50 ml with DIW containing 5% HCl to neutralize any carbonate contained in the sediments. The slurry is mixed well, then 1 ml of the slurry is transferred to a 10 ml ampoule with 2 ml of concentrated nitric acid (total volume in the ampoule is 3 ml) and left to sit for 20 minutes. The ampoule is sealed and autoclaved for 1 hour at 105^0C. The ampoules are allowed to cool completely to room temperature, and then 0.5 ml of the ampoule solution is put into a 20 ml polyethylene scintillation vial containing 19.5 ml of DIW and 1% HCl for a dilution of 1:40.

The 10 ml of slurry collected for dry weight is weighed, dried in an oven overnight at 80^oC, and weighed again. Duplicate or triplicate samples are dried and weighed until a constant weight is obtained. This constant weight is then divided by 10 to obtain the dry weight of sediment within the original 1 ml extracted for analysis.

10.3 Tissue Samples

Fish samples are prepared similar to soil/sediment samples, except that the initial addition of HCl to neutralize carbonates is not performed. For small fish (Gambusia sp.), the entire fish is weighed, placed in ampoules and digested. For large fish (bass and catfish), a stainless steel core tube, 4 mm in diameter, is used to collect three tissue plugs from the left fillet of each fish. Care is taken to collect muscle tissue and not scales or bones. The three plugs are combined in a 10 ml ampoule, weighed, and digested according to the procedures described above in Section 10.2. The weight of the tissue sample that is digested and used for analysis is usually between 0.3 g and 0.4 g.

11.0 Sample Cleanup and Separation

Additional sample cleanup and separation are not necessary to separate the mercury from the sample matrix. The bromination process used for the water samples results in a complete conversion of all organic forms of mercury to mercury (II). In addition, the acidification followed by autoclaving the sediment and tissue samples results in a complete digestion of the sample.

12.0 Sample Analysis

The PSA Merlin Plus Fluorescence detector is operated according to the manufacturer specifications with the following modifications:

- An Omega model FMA-7882 mass flow controller with a channel selector is installed at the front of the instrument. This flow controller is used to more accurately regulate the flow rates of the carrier and sheath gases (both argon) while the flow controllers supplied with the instrument on the hydride generator are open to full capacity. For low level mercury in water samples, the optimum flow rates of the carrier and sheath gases are 140 cc/min and 125 cc/min, respectively. For high level mercury, such as in sediment and tissue samples, optimum flow rates of the carrier and sheath gases are 350 cc/min and 200 cc/min, respectively.

- When analyzing water samples from 125 ml teflon bottles, the auto sampler is removed and the switch box is modified to sip only from the right sampling tube.

The procedure for sample analysis includes:

1. Tighten the peristaltic pump (pumps wash water, waste water, sample and stannous chloride).
2. Turn on the wash water to the system
3. Turn on the computer
4. Turn on the gas to the system. The argon (Zero grade) flows through two gas purifiers (charcoal and gold) and a moisture trap before reaching the instrument.
5. Turn on the line stabilizer/conditioner.
6. Check to make sure no tubes are crimped, and that flow is smooth in all tubes before proceeding.
7. Check gas flow at the mass flow controller.
   Note: For low level Hg-concentrations the optimum level of the carrier gas has been determined to be 140 cc/min while the sheath gas level has been optimized at 125 cc/min. At higher Hg-concentrations the carrier gas is 350 cc/min, and the sheath gas is 200 cc/min. The membrane dryer gas flow rate is 1 L/min.
8. Allow the system to run on DIW for 15 minutes.

9. After 15 minutes switch the instrument to $SnCl_2$.
10. Note: the sensitivity dial on the instrument is run at highest sensitivity for water but may be lowered for running of sediment, soil and tissue samples. This method is adequate for samples of the range we have run to date.
11. When the instrument is ready, zero the fluorescence detector and run acidified water (0 ppt) to check baseline response of the instrument and guard against unexplained contamination from reagent preparation. When peak height of D.I. is 0.0-0.3 the standards may be run. Initially one high standard is run to test for consistency of standard preparation and machine function. The range of standards will reflect the concentration of samples to analyze. Eight standards (four concentrations, two replicates) are run for each standard curve. Standards run for low level samples are 2.5, 5, and 10 ppt. Standards, blanks, and high level samples (generally fish, sediments and soil) may be run in plastic scintillation vials. Water samples for total-Hg are digested and analyzed in 125 ml teflon bottles. Digested acidified DIW water samples are analyzed along with the samples as reagent blanks. This number is subtracted from sample values.
12. After running the standards, two samples of the 0 ppt (acidified water) are analyzed before running the samples. Each 125 ml water sample is analyzed at least three times. Tissue and sediment samples are run in replicate. One run of fifty samples plus standards takes approximately 2 hours and uses approximately 10 ml of $SnCl_2$ per sample. A new standard curve is run when the $SnCl_2$ is replaced. In addition to running a full set of standards at the beginning of the analysis for each bottle of stannous chloride, a replicate of the highest standard and zero ppt are run after every 10 samples.

    Note: When sampling from 125 ml teflon bottles, the auto sampler tray is removed and the connections are modified to sample only from the right sampling tube. This is done by disconnecting the right (internal) sampling tube as well as the corresponding tube to the hydride generator and replaced with a longer teflon tube that directly connects the sampling tube to the hydride generator.

Instrument Shutdown:

1. If you are using the results directly from the company supplied computer program, make sure you have printed and/or saved results. This program does not reliably transfer files to ASCII or Excel although it has functions for these tasks.
2. Replace the $SnCl_2$ solution with DIW and flush the instrument for 5 minutes.
3. Turn off the wash water and disconnect tubing from DIW bottle.
4. Run the pump until no more liquid is present in the pump tubing.
5. Turn off the gas.
6. Turn off the line stabilizer and the computer.
7. Release tubing in the Hydride generator and peristaltic pump.
8. Check the waste water container and empty if necessary.

13.0 Calculations

The calculations program supplied with the AFS does not have an adequate curve fitting function for low level mercury concentrations. Therefore, the instrument data is transferred as an ASCII file into an Excel spreadsheet. Sample concentration is determined using the linear calibration curve equation:

$$Y = M * X + B,$$

where Y is the sample concentration, M is the slope of the best-fit line through the calibration points, X is the sample peak height, and b is the intercept of the line with the Y axis. Sample concentration is further corrected for background noise or drift, if any, by subtraction. In addition, if the sample was diluted prior to analysis, the sample concentration is multiplied by the dilution factor. An additional correction for dry and wet sample weight is performed for soil/sediment and tissue samples, respectively.

The concentration of mercury per gram of soil/sediment or of tissue (C_{Hg}) is obtained from the following equation:

$$C_{Hg} = (SC * D.F. * 0.003)/(W)$$

where SC is the sample concentration in ng/l (ppt), D.F. represents the final dilution factor, 0.003 represents the volume of sample in each ampoule in liters, W is either the dry weight of soil/sediment in 1 ml of solution extracted for analysis, or the wet weight of the fish digested in the ampoule (whole fish or plug samples).

14.0 Confirmation

As yet, no other analytical method has been developed to measure sub-part per trillion concentrations of mercury. Confirmation can only be obtained by analysis of duplicate samples by other laboratories with similar capabilities. Laboratories currently used by SERP for confirmation include the EPA laboratory in Athens, GA and Batelle Marine Sciences Laboratory in Sequim, WA.

15.0 Method Performance

This method measures inorganic and total mercury in water samples at concentrations between 0.3 ppt and 1000 ppt with a precision of better than 5% RSD and an accuracy between 90 and 110%. Mercury concentrations between 5 and 500 ppt in sediment and tissue samples are easily measured, with higher concentrations determined following dilution. Accuracy and precision of the higher mercury concentrations in the sediment and tissue samples can be obtained at precision and accuracy levels of better than 5% RSD and between 90 and 110%, respectively.

APPENDIX B

Related Scientific Articles

Contents

. Jones, Jacobson, Jaffe, West-Thomas, Arfstrom, and Alli. 1995. Method Development and Sample Processing of Water, Soil, and Tissue for the Analysis of Total and Organic Mercury by Cold Vapor Atomic Fluorescence Spectrometry. ***Water, Air and Soil Pollution***. 80: 1285-1294.

. Alli, Jaffe, and Jones. 1994. Analysis of organomercury compounds in sediments by capillary GC with atomic fluorescence detection. ***Journal of High Resolution Chromatography.*** Vol 17, pp. 745-748.

. Lee and Mowrer. 1988. Determination of methylmercury in natural waters at the sub-nanograms per liter level by capillary gas chromatography after adsorbent preconcentration.

. Cai, Jaffe, Alli, and Jones. 1996. Determination of organomercury compounds in aqueous samples by capillary gas chromatography-atomic fluorescence spectrometry following solid-phase extraction***. Analytica Chimica Acta*** 334 (1996) 251-259.

. Evaluation of some isolation methods for organomercury determination in soil and fish samples by capillary gas chromatography-atomic fluorescence spectrometry. ***Intern. J. Environ. Anal. Chem.*** Vol. 68 (3), pp. 331-345.

APPENDIX C

SERP Mercury Lab Standard Operating Procedures

SERP Mercury Lab Standard Operating Procedures (SOP)

SOP Number	SOP Title	Issue Date
001-99	Determination of Total Mercury in Water Samples	04/15/99
002-99	Determination of Total Mercury in Soils and Sediments	04/15/99
003-99	Determination of Total Mercury in Fish Samples	04/15/99
004-99	Determination of Organic Mercury in Water Samples	04/15/99
005-99	Determination of Organic Mercury in Soil/Sediment Samples	04/15/99
006-99	Determination of Organic Mercury in Fish Tissue Samples	04/15/99

APPENDIX D
Examples of Instrument Printouts for Total and Organic Mercury Determinations

APPENDIX E

Method Validation for Organomercury Compounds in Water, Sediment, and Tissue Samples

Limited Use Method Validation

Prepared by and for:

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_____ _____
Ronald D. Jones, Ph.D. Date
SERP Director and Professor

_____ _____
Doraida Diaz Date
SERP Quality Assurance Officer

_____ _____
Sylvia S. Labie Date
FDEP QA Officer

1.0 Scope and Application

This method covers the determination of parts per trillion (ppt) levels of organomercury compounds in water, soils, sediment, and tissue (fish) samples. Organomercury compounds that can be detected by the methods described herein include methylmercury (MeHg) and ethylmercury (EtHg). Method detection limits (MDL) of 0.02 ng/L (ppt) and 0.02 ng/g (ppb) are obtainable for both methyl- and ethylmercury in water and sediment/tissue samples, respectively.

2.0 Summary of Method

Organomercury concentrations are determined by capillary gas chromatography coupled with atomic fluorescence spectrometry (GC-AFS) as described by Alli et al. (1994). Initial extracts of sediment and tissue samples are subjected to sodium thiosulfate clean-up and the organomercury species are isolated as their bromide derivatives by acidic KBr and $CuSO_4$ and subsequent extraction into a small volume of dichloromehtane. For water samples, the organomercury compounds are pre-concentrated using a sulfhydryl cotton fiber adsorbent, followed by elution with acidic KBr and $CuSO_4$ and extraction in dichloromethane.

Chromatography is performed with a Hewlett-Packard (Model 5890 Series II) gas chromatograph coupled with an HP (Model 7673) automatic sampler. A Merlin Mercury Fluorescence Detector System, Model 10.023, (P.S. Analytical) is used.

All mercury standards are purchased from Ultra Scientific. Stock standard solutions of methyl- and ethylmercury chloride are prepared by dissolving appropriate amounts of the standards in optima grade methanol (Fisher Scientific). These solutions are stored in dark brown bottles at $<20^{o}C$ and diluted with dichloromethane.

3.0 Interferences

3.1 Matrix Interferences

For analysis of organic mercury in water samples, pH, chloride ion concentration and salinity must be within the domain of 2-5, <0.37 M and <20 $^{o}/o$, respectively. The normally occurring concentrations of ions such as sulfate, calcium, and magnesium, as well as the presence of dissolved organic carbon have no effect on the analysis.
Low levels of recovery are obtained for fish tissue (70%-80%) and soil and sediment samples (70%-85%). The exact cause for the low recovery of organic mercury is unknown yet assumed to be related to the sample matrix. Due to the low levels of recovery for these matrices, matrix spike recoveries must be determined on every sediment and tissue sample.

3.2 Environmental Interference's

Mercury contamination at levels near the method detection limit is a consistent problem as samples easily absorb mercury from the air and improperly cleaned glassware. To minimize contamination all technicians are required to wear surgical vinyl gloves. In addition, all glassware, acids, reagents, etc. are stored in a mercury-free clean room. The clean room contains a bank of laminar flow hoods equipped with gold and charcoal filters. The floor is covered with flypaper to trap particulate.

Mercury-free DIW is produced by filtering tap water through a Culligan system consisting of activated charcoal and two mixed bed ion exchange cartridges. This water is piped to the clean room where it is then passed through a Barnstead Mega-ohm B Pure system. This system is fitted with two filters (Thermolyne: colloid/organic-D0835, and ultrapure-D0809) in line with a 0.22 micron pleated particle filter. Organic mercury levels are not detectable (<0.02 ppt) in this water. This is the only water used for all analyses.

3.3 Laboratory Glassware and Sample Bottle Cleaning

Laboratory glassware is kept to a minimum, with Teflon bottles and beakers used when possible. All reusable glass bottles, volumetric flasks, graduated cylinders, and teflon beakers are dedicated to the preparation and storage of a specific reagent, and are rinsed between usage with acid (0.5N HCl and 0.05N HNO_3) and rinsed three times with DIW. Glassware or plastic containers that have come in contact with samples, such as ampules and scintillation vials are used once then discarded.

Teflon sample bottles that have been used previously for the collection of water samples are rinsed three times with DIW and filled with 1% HCl. After filling, 1 ml of mixed brominating agent is added for every 50 ml, and the bottle is shaken. This mixture remains in the bottles until used. Prior to their use, 500 µl of hydroxylamine hydrochloride is added to remove the free bromine. The bottle and cap are then rinsed three times with DIW. Sediment cups and 20 ml scintillation vials are non-reusable and discarded.

4.0 Safety Precautions

Bromine vapors are toxic; therefore, prepare the brominating agent beneath a hood. Keep all containers with the brominating agent securely capped when moved or stored. Neutralize sample bottles with hydroxylamine hydrochloride prior to their use.

Dichloromethane is a skin, eye, and respiratory irritant. A face shield and gloves must be worn when handling this material. This material should only be handled under a fume hood.

The exhaust fumes from the atomic fluorescence spectrophotometer are toxic and must be ducted away. The low pressure mercury discharge lamp used for fluorescence determination emits intense U.V. radiation. This lamp must not be viewed directly.

5.0 Apparatus and Materials

A schematic diagram of the GC-AFS system used in this work is shown in Figure I and the optimum operating conditions are summarized in Table I. A Hewlett-Packard (Model 5890 Series II) gas chromatograph coupled with an HP (Model 7673) automatic sampler is used. A fused-silica, bonded phase megabore column (15 m x 0.53 mm i.d., 1 μm non-polar DB-1 coating, J & W Scientific) and the splitless injection mode is employed. The effluent from the column is led through a pyrolyzer (P.S. Analytical Ltd., UK), positioned inside the GC oven via a piece of 65 cm length of deactivated fused-silica (0.53 mm i.d., J & W Scientific), which is connected to the column with a glass "press fit" union (J & W Scientific). The Hg atoms formed in the pyrolysis unit are transferred from the outlet end of the deactivated fused-silica tubing to the fluorescence detector (teflon transfer line, 0.5 mm i.d., Alltech Associates). The transfer line is passed through a small hole on the top of the GC oven to a Merlin Mercury Fluorescence Detector, and the connections are made *via* teflon unions.

A real time chromatographic control and data acquisition system (E-Lab, Version 4.10R, OMS TECH, INC.) is interfaced with the GC and AFS detector system. Additional equipment needed for sediment and tissue sample preparation include:

glass bottled blender	oven
syringe	balance
plastic specimen cups	
10 ml ampules	
autoclave	

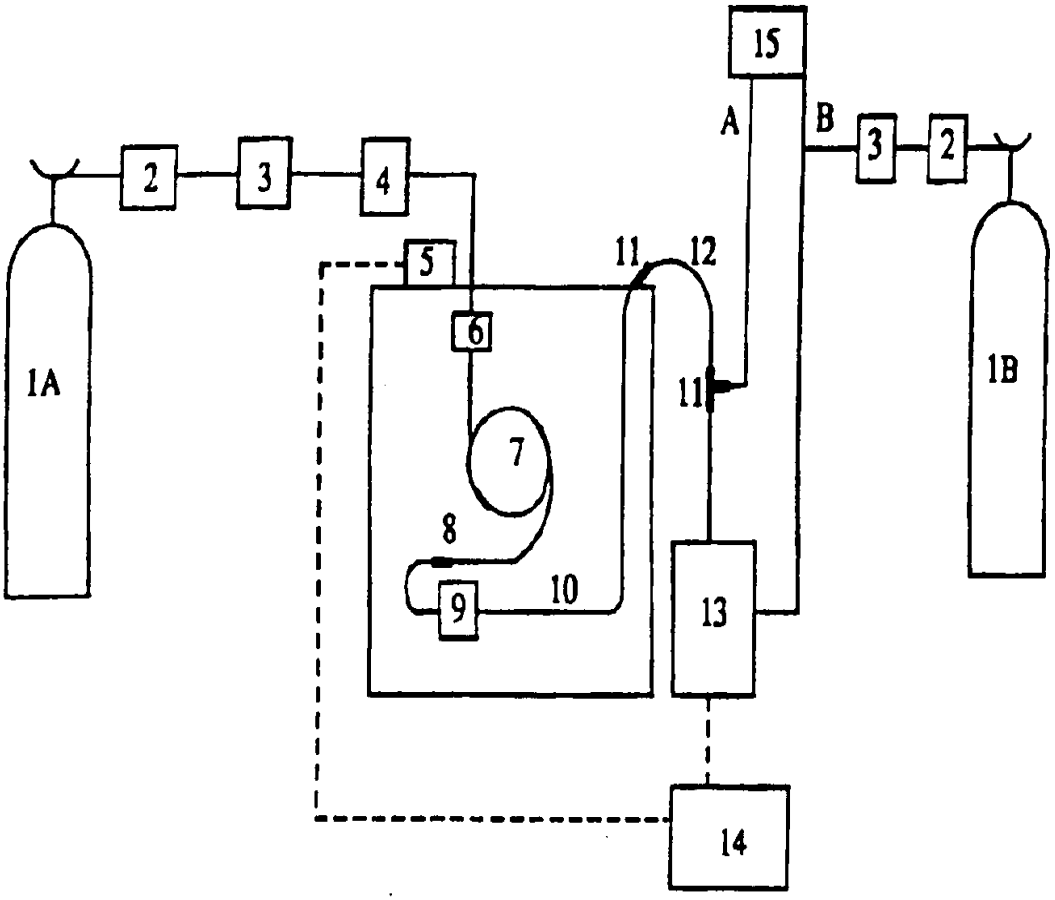

Figure I. Gas Chromatographic-Atomic Fluorescence Spectrometric System. 1A: Helium, 1B: Argon, 2: Oxygen trap, 3: Mercury trap, 4: Moisture trap, 5: Automatic sampler, 6: Injector, 7: Column, 8: Press-fit union, 9: Pyrolyzer, 10: Deactivated fused-silica 0.53mm i.d., 11: Teflon unions, 12: Teflon transfer line 0.5mm i.d., 13: Atomic Fluorescence detector, 14: E-Lab chromatographic control and data acquisition system, 15: Mass flow controller-Channel A make-up, Channel B sheath gas.

Table I. Optimized operating conditions of GC-AFS.

Gas chromatograph.	
Injector temperature	250^0C
Temperature program	1 min at 40^0C, 60^0C/min to 140^0C, 3 min at 140^0C, 50^0C/min to 200^0C, 10 min at 200^0C.
Pyrolyzer temperature	800^0C
Column flow	4.0 ml/min
Make-up flow	60 ml/min
Atomic fluorescence system	
Sheath gas flow	300 ml/min
Integrate time	0.25s
Calibration range	1000 (most sensitive)
Fine gain	10 (maximum)
Recorder output voltage	1V
Damping switch (for signal smoothing)	On

6.0 Reagents

6.1 Bromination Reagents

0.1 M Potassium Bromate
Heat 8.385 g $KBrO_3$ overnight in a glass scintillation vial (Kimble 74511) at 250^0 C +/- 20^o C in a furnace to remove mercury. After cooling dissolve the potassium bromate in 500 ml of DIW and store in a borosilicate glass bottle with a Teflon cap. Prepare Weekly.

0.2 M Potassium Bromide
Heat 11.9 g KBr overnight in a glass scintillation vial at 250^o C +/- 20^o C to remove mercury. After cooling dissolve the potassium bromide in 500 ml of DIW and store in a borosilicate glass bottle with a Teflon cap. Prepare weekly.

0.05 M Potassium Bromide (KBr) - 0.1 M Potassium Bromate ($KBrO_3$)
Mix equal volumes (100 ml) of bromate and bromide in a borosilicate glass (150 ml) bottle with a Teflon cap. Prepare daily.

6.2 Acidic Potassium Bromide

Dissolve 180 g of potassium bromide in 200 mL of DIW. Add 50 ml of trace metal grade sulfuric acid to 100 ml of DIW. Combine the two solutions in a 1 liter flask. After the solution has cooled to room temperature bring the flask up to 1 L with DIW. Store the solution in the mercury-free room in a 1 liter glass bottle with a Teflon lined cap. Prepare as needed.

6.3 1.0 M Copper Sulfate, 0.5 M Copper Chloride, 0.01 M Sodium Thiosulfate

Dissolve the appropriate amounts of each of these salts in DIW. Store in separate glass 0.5 L bottles with Teflon line caps in the mercury-free room. Prepare as needed. All solutions are extracted with dichloromethane prior to use.

6.4 Dichloromethane

Dichloromethane is stored in its original glass container and dispensed through a pipette.

6.4 Standards

All Hg standards are purchased from Ultra Scientific. Stock standard solutions of methyl- and ethylmercury chloride are prepared by dissolving appropriate amounts of the standards in optima grade methanol (Fisher Scientific). These solutions are stored in dark brown bottles, under a hood outside the mercury-free room at <20°C.

A secondary standard is prepared on a weekly basis by diluting the primary standard by 100 in methanol. Working standards are prepared on a daily basis by diluting the appropriate amount of the secondary standard in a solution of 0.8 μL DI water and 0.3 ml acidic $KBr/CuSO_4$ (3:1), then extracting with 100 μL dichloromethane. A five point standard curve is produced within the linear range of 0 to 6.67 pg Hg/μL for water samples and of 0.0 to 6.0 pg Hg/μL for solid samples. (Note, that every standard and sample is injected into the GC-AFS in 5 μL volumes.)

6.5 Gases

All gases are supplied by Liquid Carbonic Specialty Gases and are of zero grade quality. Helium (99.995%) is used as the carrier gas (GC), passed first through an oxygen trap, then through a Hg trap (gold-activated carbon) and a moisture trap prior to the GC. Argon (99.998%) is employed as the make-up gas and the sheath gas for the GC-AFS system and is also passed through moisture and Hg traps before use. Its flow is regulated by a mass flow controller (Omega) equipped with two channels, channel A (make-up flow) and channel B (sheath gas flow, see Figure I).

6.5 Synthesis of Sulfydryl-cotton (SHC) fiber adsorbent.

This synthesis follows the procedure used by Lee and Mowrer (1989). A mixture is first prepared by adding the following reagents in sequence to round bottom flask: 100 ml thioglycolic acid, 60 ml acetic anhydride, 40 ml acetic acid (36%) and 0.30 ml concentrated sulfuric acid. The mixture is allowed to cool to 45°C, then 30 g of cotton wool are added and allowed to soak thoroughly in the mixture. The reaction bottle is placed in an oven for 3 to 4 days at 40°C, then the product is placed in a filter-funnel with suction filtration and washed thoroughly with deionized water to remove traces of thioglycolic acid. The SHC fiber obtained is dried at 40°C for 24 h and stored at room temperature (20°C).

7.0 Calibration

A linear calibration curve is run prior to each sample run (See section 6.4). Linear regression analysis is used to determine the best-fit calibration line with a regression coefficient (R^2) of 0.995 or better. Standard curves outside the acceptable limits are run again, and new standards are prepared if necessary.

8.0 Quality Control

8.1 Method Detection Limit

The method detection limit (MDL) is determined for this method according to the EPA procedure described in 40 CFR Part 136, Appendix B, revision 1.11, except that a value of 3 is used in place of the Student's T value. Specifically, seven or more reagent blanks are analyzed and the instrument baseline noise is determined. The standard deviation of the replicate analyses is determined and the MDL is computed as 3 times the standard deviation.

Table A.1
Method Detection Limit

Matrix	MeHg Conc. EtHg Conc.	Mean	Standard Deviation (S)	MDL = 3 x S
Water	0.0092 ng/L 0.0090 ng/L 0.0092 ng/L 0.0090 ng/L 0.0135 ng/L 0.0132 ng/L 0.0221 ng/L 0.0216 ng/L 0.0221 ng/L 0.0216 ng/L 0.0105 ng/L 0.0102 ng/L 0.0117 ng/L 0.0114 ng/L 0.0141 ng/L 0.0138 ng/L 0.0234 ng/L 0.0228 ng/L 0.0092 ng/L 0.0090 ng/L	MeHg 0.0145 ng/L EtHg 0.0142 ng/L	MeHg 0.0055 ng/L EtHg 0.0057 ng/L	MeHg 0.017 ng/L EtHg 0.017 ng/L
Sediment / Tissue	0.028 ng/g 0.005 ng/g 0.005 ng/g 0.010 ng/g 0.011 ng/g 0.008 ng/g 0.003 ng/g 0.014 ng/g 0.015 ng/g 0.005 ng/g 0.014 ng/g 0.020 ng/g 0.014 ng/g 0.015 ng/g 0.010 ng/g 0.004 ng/g 0.014 ng/g 0.010 ng/g	MeHg 0.013 ng/g EtHg 0.010 ng/g	MeHg 0.007 ng/g EtHg 0.005 ng/g	MeHg 0.021 ng/g EtHg 0.015 ng/g

8.2 Precision

Precision is defined as the agreement or closeness of two or more results of the same sample, and is determined in terms of percent relative standard deviation (% RSD) using the following equation:

$$\% \text{ RSD} = \frac{s}{X} * 100,$$

where, s and X represent the standard deviation and mean, respectively, of two or more results of the same sample. The analytical precision of this method is variable with more precise measurements obtained at higher mercury concentrations.

Table A.2
Precision

Matrix	MeHg and EtHg Precision %RSD
Water	<10
Sediment/Tissue	<10

8.3 Accuracy

Accuracy is defined as the agreement between the analytical results and the known concentration. Accuracy is determined by running continuing calibration standards, check standards, and matrix spike samples and is determined as percent recovery (%R) according to the following equation:

$$\% \text{ R} = \frac{\text{Observed Standard Concentration}}{\text{Known Standard Concentration}} * 100.$$

Instrument accuracy is determined on a daily basis by performing matrix spike samples. Percent recoveries between 95 - 105% and between 80 - 120% for methyl- and ethylmercury, respectively, in water. For solid and tissue samples recoveries of 70 - 85% and 70 - 80% are obtained, respectively. Due to these low recoveries, matrix spike samples must be done on every tissue and sediment sample in order to compensate for matrix effects.

Table A.3
Accuracy

Matrix	Accuracy (% Recovery)	
	MeHg	EtHg
Water	95 - 105	80 - 120
Soil/Sediment	70 - 85	70 - 85
Tissue	70 - 80	70 - 80

8.4 QC Checks

Calibration check samples are run in duplicate following the standard curve at the beginning and after every 10 samples. Three replicate samples are prepared for each solid and tissue sample. Two of the replicates are run as replicate samples, while one of the samples is spiked to serve as a matrix spike. The percent recovery is determined for all solid and tissue samples, and this recover factor is applied to each sample. A standard reference material for methylmercury in tissue is available from the Canadian National Research Council (DORM-2 Dogfish muscle and liver). Standard reference material for ethylmercury is not yet available.

9.0 Sample Collection, Preservation, and Handling

9.1 Water Samples

Water samples are collected in Teflon bottles. Collection is done while wearing shoulder length polyethylene gloves over a pair of vinyl gloves. Surface water samples are collected through a 105 μm nylon screen via a vacuum system to reduce the amount of sediment collected. Samples are double bagged in zip-lock polyethylene bags and placed in a plastic ice chest/cooler used exclusively for mercury samples. Samples are returned to the laboratory upon the same day of sample collection, acidified with HCl to a pH<2, then stored in the mercury-free clean room and

analyzed within 28 days. Immediately prior to processing for analysis the sample pH is increased to above 3 with NaOH.

9.2 Sediment Samples

Sediment samples are collected in polyethylene specimen cups (Elkay non-sterile wide mouth specimen cups with screw caps - 128 ml volume) and placed in polyethylene zip-lock bags. All field samples are kept in a cooler used exclusively for low level mercury samples until they are returned to the laboratory, where they are stored in a freezer. Samples can be stored indefinitely within the freezer; however, analysis within 28 days is recommended.

9.3 Tissue Samples

Fish are collected with a dip net and stored in plastic bags for transport to the laboratory. In the laboratory fish are stored within the freezer indefinitely and analyzed within 28 days.

10.0 Sample Extraction/Preparation

10.1 Water Samples

The determination of organic mercury in water samples involves an adsorbent pre-concentration of the organomercurials onto sulfydryl-cotton fibers. The sulfydryl-cotton (SFC) fiber columns are made of a 5 ml screening column (Fisher Scientific) containing 0.16 g of SFC fiber, packed 1 cm high. The water sample is passed through the column by vacuum. Five-ml of an acidic potassium bromide and 1.0 M copper sulfate mixture (2:1) are then pipetted on the surface of the adsorbent and the eluate is collected in a 6 ml glass vial. This is extracted with 0.25 ml dichloromethane on a shaker and centrifuged as described above. The dichloromethane layer is then transferred to a 2 ml glass sampling vial and subjected to GC analysis.

10.2 Soils, Sediment and Tissue Samples

Preparation of soil/sediment samples is done outside the mercury-free room, due to their high mercury concentrations. Soil/sediment samples are homogenized and slurred in a glass bottle blender. A mixture of 120 cc of soil/sediment and 50 ml of DIW is blended for three minutes. With a syringe, 10 ml of the slurry is collected for dry weight determination.

The extraction procedure for soil/sediment and tissue samples consists of three steps. **Step 1.** A 1.0-5.0 g portion of the homogenized sample is placed in a 20 ml borosilicate glass scintillation vial (Kimble, #74511). To the vial 5 ml distilled water, 3.0 ml of 1.0 M copper sulfate and 3.0 ml of acidic potassium bromide solution are added. The mixture is shaken for 1 hr at 330 rpm (Gyrotory Shaker Model G2). Dichloromethane (5 ml) is added and the mixture is shaken for 24

h at 330 rpm and then centrifuged for 10 min at 5000 g in a Sorvall Model RC-5 refrigerated centrifuge (Dupont). **Step 2.** An exactly known volume of the dichloromethane layer (3.0-4.0 ml) is transferred to a 7.0 ml borosilicate glass scintillation vial (Kimble, #0333726) and 1.0 ml of 0.01 M sodium thiosulfate is added. The mixture is shaken for 20 min at 330 rpm and centrifuged at high speed in a IEC clinical centrifuge. **Step 3.** The aqueous layer (0.8 - 0.9 ml) is placed in a 1.5 ml microcentrifuge tube (Fisherbrand, Fisher Scientific), and 0.3 ml of acidic KBr and $CuSO_4$ mixture (3:1) and 0.1 ml dichloromethane are added. The contents are mixed for 1 min on a Vortex Genie mixer and centrifuged for 2 min at high speed (16,749 x g) in a Hermle centrifuge. The dichloromethane is transferred to a 2.0 ml glass sampling vial containing a few crystals of anhydrous sodium sulphate and subjected to GC analysis. Injections of 5.0 μL are used. Samples spiked with known concentrations of methyl- and ethylmercury chloride are extracted to evaluate the recovery factor used for quantification.

The 10 ml of slurry collected for dry weight is weighed, dried in an oven overnight at 80^{o}C, and weighed again. Duplicate or triplicate samples are dried and weighed until a constant weight is obtained. This constant weight is then divided by 10 to obtain the dry weight of sediment within the original 1 ml extracted for analysis.

10.3 Tissue Samples

Fish samples are prepared similar to soil/sediment samples. For small fish (Gambusia sp.) the entire fish is weighted and placed in the 20 ml borosilicate glass scintillation vial. For large fish (bass and catfish), a stainless steel core tube, 4 mm in diameter, is used to collect three tissue plugs from the left fillet of each fish. The three plugs are weighed then placed in the borosilicate glass scintillation vial and digested as for the soil and sediment samples.

11.0 Sample Cleanup and Separation

The sample cleanup and separation procedures are described in detail above under Section 10.0 Sample Extraction/Preparation.

12.0 Sample Analysis

The procedure for sample analysis is as follows:

1. Turn on main switch (controls computer plus AF detector).
2. Turn on conditioner (controls gas meter).
3. Screw teflon tubing that carries helium from the GC column to AF detector.
4. Turn on pyrolyzer.
5. Fill up solvent containers with CH_2Cl_2.
6. Place samples in the autosampler and write sequence of samples.
7. Press "zero" in the AF detector several times until it stabilize (it takes about an hour or more).
8. Type "elab" at the prompt sign.
9. Select "method".
10. Select "retrieve". Hit "enter" twice.
11. Hit "escape", select "go".
12. Enter total # of samples to be run and press "enter".
13. Enter file name and press "enter".
14. Press "start" in the autosampler.
15. A curve is created at the beginning of each run by running the following concentrations: 0.0, 0.83, 1.67, 3.33, and 6.67 pg/μL for water samples; 0.0, 2.0, 4.0, and 6.0 pg/μL for solid samples.
16. A blank is run following the calibration curve.
17. A low and a high standard are run to check the calibration curve.
18. A blank sample is run prior to running the actual samples.
17. High standards are run after each set of ten samples. The run ends with a high standard followed by a blank.
18. All samples are run in duplicate.
19. For sediment and tissue samples two duplicate samples and one matrix spike sample are run for every sample.
20. QC check standard made from a different source is used to confirm calibration.

A consistent system for determining peak responses has been established by properly selecting threshold level of the data acquisition program (see attached chromatograms).

13.0 Calculations

Sample concentration is determined using the linear calibration curve equation:

$$H = M * X + B,$$

where H is the sample concentration in pg/□l, M is the slope of the best-fit line through the

calibration points, X is the sample peak height, and b is the intercept of the line with the Y axis. Sample concentration is further corrected for background noise or drift, if any, by subtraction.

The formulas used to calculate the final organomercury results are as follows (see attached sheet):

Column A: sample No. (a, b, c, d)
B (gram): wet sample weight
C (gram): dry sample weight
C=B * R
R: sample dry/wet ratio
D (ng/g): standard concentration spiked into sample based on dry weight
D=1000/(C*1000)
E (ml): volume of CH_2Cl_2 transferred at the first extraction step initially, 5 ml of CH_2Cl_2 is added to sample for extraction).
F (ml): volume of $Na_2S_2O_3$ transferred at the back extraction procedure (initially, 1.0 ml of $Na_2S_2O_3$ is added to the CH_2Cl_2 extract).
G (*E+5): peak area
H (pg/□l): concentration of organomercury in sample
I= (H * V / C) * (5.0 / E * 1.0 / F) * 1/1000
V(□l): volume of CH_2Cl_2 added at the final extraction step.
5.0/E: correction factor for the first CH_2Cl_2 extraction step.
1.0/F: correction factor for the $Na_2S_2O_3$ back extraction step.
J (ng/g): average concentration of unspiked samples (a & b).
K (ng/g): standard deviation of concentration of unspiked samples.
L (%): recovery of the spiked sample
L=(I –J) / D * 100
M(%): averages of the recoveries (L).
N (ng/g): recovery corrected sample concentration.
O (ng/g): recovery corrected standard deviation.

14.0 Confirmation

As yet, no other analytical method has been developed to measure sub-part per trillion concentrations of organic mercury compounds. Confirmation can only be obtained by analysis of duplicate samples by other laboratories with similar capabilities. Laboratories currently used by SERP for confirmation include the EPA laboratory in Athens, GA and Batelle Marine Sciences Laboratory in Sequim, WA.

15.0 Method Performance

This method measures organic mercury compounds (methylmercury) in water and sediment/tissue samples at method detection limits of 0.02 ppt and 0.02 ppb, respectively. Following the same procedures described herein, similar detection limits can be obtained for ethylmercury.

16.0 References

Alli, A., R. Jaffé, and R. Jones. 1994. Analysis of organomercury compounds in sediments by capillary GC with atomic fluorescence detection. Journal of High Resolution Chromatography. 17:745-748.

Lee, Y.H. and Mowrer, J. 1989. Determination of methylmercury in natural waters at the sub-nanograms per liter level by capillary gas chromatography after adsorbent preconcentration. Anal. Chim. Acta. 221:259-268.

APPENDIX A

Method Validation for Part per Trillion (ppt) Concentrations of Inorganic and Total Mercury in Water, Solid, and Tissue Samples

Limited Use Method Validation

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1.0 Scope and Application

This method covers the determination of parts per trillion (ppt) levels of total and inorganic mercury in water, soils, sediment, and tissue (fish) samples. A method detection limit of 0.3 ppt is obtainable for water samples at a precision of less than 5% relative standard deviation (%RSD) and an accuracy between 90 and 110%. The concentrations of mercury in sediment and tissue samples are significantly higher than in water samples and can be determined at better precision (<5 %RSD) and accuracy (90 to 110 %R). Linear calibrations up to 1 part per million (ppm) can be obtained and higher concentrations can be measured with dilution.

2.0 Summary of Method

Total and inorganic mercury concentrations are determined by Cold Vapor Atomic Fluorescent Spectrometry (CVAFS). In this method, mercury in a liquid sample is vaporized and stripped from the remaining liquid by a carrier gas (Argon). A sheath gas (also Argon) constrains the mercury vapor to a small stream as it passes by a light source and photomultiplier tube. The mercury concentration is determined by atomic fluorescence.

An PS Analytical (PSA) Merlin Plus mercury analysis system equipped with an autosampler, vapor generator, fluorescence detector and a PC-based integrator package is used to detect total and inorganic mercury. The system is run according to the manual provided by the manufacturer, except lower flow rates are used for the carrier and sheath gases. Low detection levels of 0.3 ppt Hg in water samples are obtained with flow rates of 0.14 L/min and 0.125 L/min for the carrier and sheath gases, respectively. For the higher concentrations of mercury detected in sediment and tissue samples, flow rates of 0.35 L/min for the carrier gas and 0.2 L/min for the sheath gas are used. To accurately regulate the gas flow for optimum conditions, SERP installed an Omega model FMA-78P2 electronic mass flow controller in front of the PSA Merlin Plus instrument.

Water sample preparation includes digestion by a brominating procedure to breakdown all mercury complexes. Sediment samples and tissue samples are digested with concentrated nitric acid in sealed ampules, and subsequently autoclaved.

An NBS standard of 1000 μg Hg/l is used and diluted to obtain the appropriate linear standard curve. High standards of 10 ppt and 400 ppt are used for water/tissue and sediment samples, respectively. NBS oyster tissue 60 ng/g (566a), NRCC dogfish muscle 4.64 μg/g (DORM-2), NIST sediment nominal 50 μg/g (8407), and NIST sediment 60 ng/g (8406) are used to check the accuracy of the tissue and sediment results.

3.0 Interferences

3.1 Matrix Interferences

To overcome matrix interferences due to organic substances in the water samples, the samples are left in an ultraviolet cabinet for 12 hours. By digesting the water samples with bromine afterward, any remaining mercury complexes including organomercury compounds, sulphide complexes and complexes with organic material (e.g. fulvic acids) are broken down. Additionally, acid digesting and autoclaving results in a complete breakdown of mercury in the sediment and tissue samples.

3.2 Environmental Interferences

Mercury contamination at levels near the method detection limit is a consistent problem as samples easily absorb mercury from the air and improperly cleaned glassware. To minimize contamination, all technicians are required to wear surgical vinyl gloves. In addition, all glassware, acids, reagents, etc. are stored in a mercury-free clean room. The clean room contains a bank of laminar flow hoods equipped with gold and charcoal filters. The floor is covered with flypaper to trap particulates. Potential contamination in the clean room is checked weekly by monitoring acidified (1% HCl) water samples, which are stored open inside the clean room. If significant levels of mercury is detected in these samples (<20 ppt), then the source of the mercury contamination is identified and eliminated. The gold and charcoal filters within the laminar flow hoods are reconditioned if necessary.

Mercury-free DIW is produced by filtering tap water through a Culligan system consisting of activated charcoal and two mixed bed ion exchange cartridges. This water is piped to the clean room where is then passed through a Barnstead Mega-ohm B Pure system. This system is fitted with two filters (Thermolyne: colloid/organic-D0835, and ultrapure-D0809) in line with a 0.22 micron pleated particle filter. Mercury levels are not detectable (<0.1 ppt) in this water by both our laboratory and by and independent laboratory analysis. This is the only water used for all analyses.

3.3 Laboratory Glassware and Sample Bottle Cleaning

Laboratory glassware is kept to a minimum, with Teflon bottles and beakers used when possible. All reusable glass bottles, volumetric flasks, graduated cylinders, and teflon beakers are dedicated to the preparation and storage of a specific reagent, and are rinsed between usage with acid (0.5N HCl and 0.05N HNO_3) and rinsed three times with DIW. One volumetric flask is kept dedicated to making the primary standard and is rinsed only with this standard. Glassware or plastic containers that

have come in contact with samples, such as ampules and scintillation vials are used once then discarded.

Teflon sample bottles that have been used previously are rinsed three times with DIW and filled with 1% HCl. After filling, 1 ml of mixed brominating agent is added for every 50 ml, and the bottle is shaken. This mixture remains in the bottles until used. Prior to their use, 500 µl of hydroxylamine hydrochloride is added to remove the free bromine. The bottle and cap are then rinsed three times with DIW. Sediment cups and 20 ml scintillation vials are non-reusable and discarded.

4.0 Safety Precautions

Bromine vapors are toxic; therefore, prepare the brominating agent beneath a hood. Keep all containers with the brominating agent securely capped when moved or stored. Neutralize sample bottles with hydroxylamine hydrochloride prior to their use.

Hydroxylamine hydrochloride is a skin and eye irritant and can cause dermatitis. A face shield and gloves must be worn when handling this material.

The exhaust fumes from the atomic fluorescence spectrophotometer are toxic and must be ducted away. The low pressure mercury discharge lamp used for fluorescence determination emits intense U.V. radiation. This lamp must not be viewed directly.

5.0 Apparatus and Materials

Analytical instrumentation includes an PSA Merlin Plus mercury analysis system equipped with the following:

Autosampler	Vapor Generator
Fluorescence Monitor	IBM compatible computer system

In addition, an Omega model FMA-78P2 electronic mass flow controller is installed in front of the PSA Merlin Plus instrument. All of the above equipment is stored beneath a protective hood. All other standard laboratory equipment (Teflon beakers, glassware, pipets, etc.) is kept within the clean room and dedicated to mercury analysis. A mercury-dedicated refrigerator, oven, and analytical balance are kept within the clean room. Polyethylene scintillation vials (20 ml) with polypropylene caps are used with the autosampler. Water samples suspected of low mercury concentration are injected into the instrument manually from 125 ml teflon bottles.

Additional equipment needed for sediment and tissue sample preparation include:

glass bottled blender	autoclave
syringe	oven
plastic specimen cups	balance
10 ml ampules	

6.0 Reagents

6.1 Bromination Reagents

0.1 M Potassium Bromate

Heat 8.385 g $KBrO_3$ overnight in a glass scintillation vial (Kimble 74511) at 250° C +/- 20° C in a furnace to remove mercury. After cooling dissolve the potassium bromate in 500 ml of DIW and store in a borosilicate glass bottle with a Teflon cap. Prepare Weekly.

0.2 M Potassium Bromide

Heat 11.9 g KBr overnight in a glass scintillation vial at 250° C +/- 20° C to remove mercury. After cooling dissolve the potassium bromide in 500 ml of DIW and store in a borosilicate glass bottle with a Teflon cap. Prepare weekly.

0.05 M Potassium Bromide (KBr) - 0.1 M Potassium Bromate ($KBrO_3$)

Mix equal volumes (100 ml) of bromate and bromide in a borosilicate glass (150 ml) bottle with a Teflon cap. Prepare daily.

6.2 Hydroxylamine Hydrochloride

Dissolve 6 g of $NH_2OH \cdot HCl$ in 50 ml of DIW in a 60 ml Teflon bottle. Prepare weekly.

6.3 Stannous Chloride

Add 50 ml of 12 N HCl to 40 g of Stannous Chloride ($SnCl_2$). Bring to 1000 ml with DIW in a borosilicate glass bottle with a Teflon cap. Stannous chloride is purged of any traces of mercury with argon gas continuously throughout the analysis. Prepare fresh daily.

6.4 12 N Hydrochloric Acid

Concentrated HCl (12 N HCl) is dispensed via a pipette or poured into a graduate cylinder, either of which has been previously acid washed and rinsed three times with DIW.

6.5 16 N Nitric Acid

Concentrated Nitric acid (16 N) is dispensed through a pipet, which has been previously washed and rinsed three times with DIW.

6.6 Standards

Due to its high concentration, primary stock standard is prepared and stored outside of the mercury-free clean room. The primary stock standard is made by addition of 100 µl of SEPEX (PLHG4-2X) (1000 µg/ml) to 1000 ml of DIW with 10 ml of trace metal grade HCl in a glass, volumetric flask with a ground glass stopper. This standard is prepared daily. Secondary standards are prepared daily in 500 ml Teflon bottles by adding concentrated HCl (5 ml) to 495 ml of DIW. The primary stock is brought into the clean room and 50 µl - 250 µl (depending on final concentration of 10 ppt - 50 ppt) is added to the bottles containing the water-acid mixture with a pipet.

6.7 Standard Reference Material

The following standard reference materials are used to check the accuracy and precision of the tissue and sediment digestion methods and analyses:

DORM-2 Dogfish muscle (NRCC)
556a Oyster tissue (NBS)
8406 Sediment (NIST)
8407 Sediment (NIST)

These standard reference materials are stored and dried according to the Certificate of Analysis accompanying each standard. The standards are digested following the same procedures used for tissue and sediment samples. They are prepared and analyzed on a daily basis.

7.0 Calibration

A linear calibration curve is run prior to each sample run. For low level water samples, standards include 0, 2.5, 5 and 10 ppt. Standards run for tissue and sediment samples include 0, 100, 250, and 400 ppt. All standards are run in replicate and plotted using the software inherent to the instrument. Linear regression analysis is used to determine the best-fit calibration line with a regression coefficient (r^2) of 0.998 or better. Standard curves outside the acceptable limits are run again, and new standards are prepared if necessary.

The sensitivity of the Merlin Plus System is such that a zero mercury concentration results in a zero result. However, multiple dilutions of the source standard as required to produce the low standard of 2.5 ppt, often results in error, thereby producing a standard curve that does not intercept the origin. Therefore, once the standard curve is checked for linearity, a new linear calibration curve is recalculated using the high standard and forcing the intercept through zero. Mercury concentrations in samples are then determined by comparing sample peak heights to the new standard curve.

8.0 Quality Control

8.1 Method Detection Limit

The method detection limit (MDL) of 0.3 ppt is determined for this method according to the EPA procedure described in 40 CFR Part 136, Appendix B, revision 1.11, except that a value of 3 is used in place of the Student's T value. Specifically, seven or more replicate samples containing a known, low concentration of mercury are analyzed. The standard deviation of the replicate analyses is determined and the MDL is computed as 3 times the standard deviation.

Table A.1
Method Detection Limit

Matrix	Hg Concentration (ppt)	Mean	Standard Deviation (S)	MDL = 3 x S
Spiked DIW (DIW plus 1ppt Hg)	2.315 2.093 2.216 2.167 2.290 2.093 2.118	2.185	0.085	0.255
Sediment	84.30 ppb 85.42 ppb 82.44 ppb 81.32 ppb 84.30 ppb 82.44 ppb 82.81 ppb	83.29 ppb	1.42 ppb	4.27 ppb

8.2 Precision

Precision is defined as the agreement or closeness of two or more results of the same sample, and is determined in terms of percent relative standard deviation (% RSD) using the following equation:

$$\% \text{ RSD} = \frac{s}{X} * 100,$$

where, s and X represent the standard deviation and mean, respectively, of two or more results of the same sample. The analytical precision of this method is variable with more precise measurements obtained at higher mercury concentrations.

Table A.2
Precision

Matrix	Hg Concentration	Precision %RSD
Water	0.3 - 30 ppt	< 5%
Water	5 - 500 ppt	< 5%
Water	1 - 1000 ppt	< 5%
Soil/Sediment Slurry	1 ppt - 400 ppt	< 5%
Tissue Slurry	1 ppt - 400 ppt	< 5%

8.3 Accuracy

Accuracy is defined as the agreement between the analytical results and the known concentration. Accuracy is determined by running continuing calibration standards or by check standards and is determined as percent recovery (%R) according to the following equation:

$$\% \text{ R} = \frac{\text{Observed Standard Concentration}}{\text{Known Standard Concentration}} * 100.$$

Instrument accuracy is determined on a daily basis by performing multiple analysis (5 or more replicates) of the high standard. Percent recoveries between 90 and 110% are obtained for water samples with a high standard of 10 ppt. For solid and tissue samples with a high standard of 400 ppt, percent recoveries between 95 and 105% are obtained.

Table A.3
Accuracy

Matrix	Hg Concentration	Accuracy (% Recovery)
Water	0.3 - 30 ppt	90 - 110
Water	5 - 500 ppt	90 - 110
Water	1 - 1000 ppt	90 - 110
Soil/Sediment Slurry	5 - 400 ppt	90 - 110
Tissue Slurry	5 - 400 ppt	90 - 110

8.4 QC Checks

Calibration check samples are run in duplicate following at the beginning and after every 10 samples. For solid and tissue samples, NIST or NBS standards are run after every 20 samples. Accuracy for each of these calibration check samples and standards must be within 90 to 110% or the samples must be run again.

9.0 Sample Collection, Preservation, and Handling

9.1 Water Samples

Water samples are collected in Teflon bottles. Collection is done while wearing shoulder length polyethylene gloves over a pair of vinyl gloves. Surface water samples are collected through a 105 μm nylon screen via a vacuum system to reduce the amount of sediment collected. Samples are double bagged in zip-lock polyethylene bags and placed in a plastic ice chest/cooler used exclusively for low level mercury samples. Samples are returned to the laboratory upon the same day of sample collection and preserved in the mercury-free clean room with 0.5 ml of trace metal grade HCl per 100 ml of sample. Sample analysis within 28 days is recommended.

9.2 Sediment Samples

Sediment samples are collected in polyethylene specimen cups (Elkay non-sterile wide mouth specimen cups with screw caps - 128 ml volume) and placed in polyethylene zip-lock bags. All field samples are kept in a cooler used exclusively for low level mercury samples until they are returned to the laboratory, where they are stored in a freezer. Samples can be stored indefinitely within the freezer; however, analysis within 28 days is recommended.

9.3 Tissue Samples

Fish are collected with a dip net and stored in plastic bags for transport to the laboratory. In the laboratory fish are stored within the freezer indefinitely; however, analysis within 28 days is recommended.

10.0 Sample Extraction/Preparation

10.1 Water Samples

Water samples analyzed for inorganic mercury do not require additional sample preparation prior to analysis. For analysis of total mercury, the samples are placed in an ultraviolet cabinet for 12 hours, allowed to cool, then 2.5 ml of $KBrO_3/KBr$ solution is added to 125 ml of each water sample. The sample is left to brominate for one hour in the Hg-free room; after which 500 μl of hydroxylamine hydrochloride is added to the solution to inhibit further reaction. Samples are permitted to settle for at least 10 minutes before analysis.

10.2 Soils and Sediment Samples

Preparation of soil/sediment samples is done outside the mercury-free room, due to their high mercury concentrations. Soil/sediment samples are homogenized and slurried in a glass bottle blender. A mixture of 120 cc of soil/sediment and 50 ml of DIW is blended for three minutes. With a syringe, 10 ml of the slurry is collected for dry weight determination. Another 10 ml is pipetted into a polyethylene specimen cup and diluted to 50 ml with DIW containing 5% HCl to neutralize any carbonate contained in the sediments. The slurry is mixed well, then 1 ml of the slurry is transferred to a 10 ml ampule with 2 ml of concentrated nitric acid (total volume in the ampule is 3 ml) and left to sit for 20 minutes. The ampule is sealed and autoclaved for 1 hour at 105°C. The ampules are allowed to cool completely to room temperature, and then 0.5 ml of the ampule solution is put into a 20 ml polyethylene scintillation vial containing 19.5 ml of DIW and 1% HCl for a dilution of 1:40.

The 10 ml of slurry collected for dry weight is weighed, dried in an oven overnight at 80°C, and weighed again. Duplicate or triplicate samples are dried and weighed until a constant weight is obtained. This constant weight is then divided by 10 to obtain the dry weight of sediment within the original 1 ml extracted for analysis.

10.3 Tissue Samples

Fish samples are prepared similar to soil/sediment samples, except that the initial addition of HCl to neutralize carbonates is not performed. For small fish (Gambusia

sp.), the entire fish is weighed, placed in ampules and digested. For large fish (bass and catfish), a stainless steel core tube, 4 mm in diameter, is used to collect three tissue plugs from the left fillet of each fish. Care is taken to collect muscle tissue and not scales or bones. The three plugs are combined in a 10 ml ampule, weighed, and digested according to the procedures described above in Section 10.2. The weight of the tissue sample that is digested and used for analysis is usually between 0.3 g and 0.4 g.

11.0 Sample Cleanup and Separation

Additional sample cleanup and separation are not necessary to separate the mercury from the sample matrix. The bromination process used for the water samples results in a complete conversion of all organic forms of mercury to mercury (II). In addition, the acidification followed by autoclaving the sediment and tissue samples results in a complete digestion of the sample.

12.0 Sample Analysis

The PSA Merlin Plus Fluorescence detector is operated according to the manufacturer specifications with the following modifications:

- An Omega model FMA-7882 mass flow controller with a channel selector is installed at the front of the instrument. This flow controller is used to more accurately regulate the flow rates of the carrier and sheath gases (both argon) while the flow controllers supplied with the instrument on the hydride generator are open to full capacity. For low level mercury in water samples, the optimum flow rates of the carrier and sheath gases are 140 cc/min and 125 cc/min, respectively. For high level mercury, such as in sediment and tissue samples, optimum flow rates of the carrier and sheath gases are 350 cc/min and 200 cc/min, respectively.

- When analyzing water samples from 125 ml teflon bottles, the auto sampler is removed and the switch box is modified to sip only from the right sampling tube.

The procedure for sample analysis includes:

1. Tighten the peristaltic pump (pumps wash water, waste water, sample and stannous chloride).
2. Turn on the wash water to the system
3. Turn on the computer

4. Turn on the gas to the system. The argon (Zero grade) flows through two gas purifiers (charcoal and gold) and a moisture trap before reaching the instrument.
5. Turn on the line stabilizer/conditioner.
6. Check to make sure no tubes are crimped, and that flow is smooth in all tubes before proceeding.
7. Check gas flow at the mass flow controller.
   Note: For low level Hg-concentrations the optimum level of the carrier gas has been determined to be 140 cc/min while the sheath gas level has been optimized at 125 cc/min. At higher Hg-concentrations the carrier gas is 350 cc/min, and the sheath gas is 200 cc/min. The membrane dryer gas flow rate is 1 L/min.
8. Allow the system to run on DIW for 15 minutes.
9. After 15 minutes switch the instrument to $SnCl_2$.
10. Note: the sensitivity dial on the instrument is run at highest sensitivity for water but may be lowered for running of sediment, soil and tissue samples. This method is adequate for samples of the range we have run to date.
11. When the instrument is ready, zero the fluorescence detector and run acidified water (0 ppt) to check baseline response of the instrument and guard against unexplained contamination from reagent preparation. When peak height of D.I. is 0.0-0.3 the standards may be run. Initially one high standard is run to test for consistency of standard preparation and machine function. The range of standards will reflect the concentration of samples to analyze. Eight standards (four concentrations, two replicates) are run for each standard curve. Standards run for low level samples are 2.5, 5, and 10 ppt. Standards, blanks, and high level samples (generally fish, sediments and soil) may be run in plastic scintillation vials. Water samples for total-Hg are digested and analyzed in 125 ml teflon bottles. Digested acidified DIW water samples are analyzed along with the samples as reagent blanks. This number is subtracted from sample values.
12. After running the standards, two samples of the 0 ppt (acidified water) are analyzed before running the samples. Each 125 ml water sample is analyzed at least three times. Tissue and sediment samples are run in replicate. One run of fifty samples plus standards takes approximately 2 hours and uses approximately 10 ml of $SnCl_2$ per sample. A new standard curve is run when the $SnCl_2$ is replaced. In addition to running a full set of standards at the beginning of the analysis for each bottle of stannous chloride, a replicate of the highest standard and zero ppt are run after every 10 samples.

APPENDIX B

Jones, Jacobson, Jaffe, West-Thomas, Arfstrom, and Alli. 1994. Method Development and Sample Processing of Water, Soil, and Tissue for the Analysis of Total and Organic Mercury by Cold Vapor Atomic Fluorescence Spectormetry. Water, Air and Soil Pollution.

Method Development and Sample Processing of Water, Soil, and Tissue for the Analysis of Total and Organic Mercury by Cold Vapor Atomic Fluorescence Spectrometry

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Abstract. Atomic Fluorescence-based methods have been developed for measuring ultratrace levels of mercury (Hg) in environmental (water, soil) and biological (fish tissue) samples. In addition, methods for preparation of water, soil, and tissue samples have been developed. For the analysis of total Hg in soil, sediment and fish the samples are digested with concentrated nitric acid in sealed glass ampules, and subsequently autoclaved. Water samples are digested using standard brominating procedures. A Merlin Plus, PS Analytical atomic fluorescence spectrometer (AFS) system equipped with an autosampler, vapor generator, fluorescence detector and a PC based integrator package is used in the determination of total Hg. The determination of Hg mercury species in water, without pre-derivitization, involves adsorbent pre-concentration of the organomercurials onto sulfydryl-cotton fibers. The organic Hg compounds are eluted with a small volume of acidic KBr and $CuSO_4$ and extracted into dichloromethane. Sediment, soil and tissue samples are homogenized and the organomercurials first released from the sample by the combined action of acidic KBr and $CuSO_4$ and extracted into dichloromethane. The initial extracts are subjected to thiosulfate clean-up and the organomercury species are isolated as their chloride derivatives by cupric chloride addition and subsequent extraction into a small volume of dichloromethane. Analysis of organic Hg compounds is accomplished by capillary column chromatography coupled with atomic fluorescence detection.

1. Introduction

Mercury is a widely distributed pollutant in the environment and has gained considerable toxicological concern in recent years. In some cases, the desired quantitation levels of this metal challenge the detection limits of the instrumentation and methods in current use (Swift and Campbell, 1993; Kammin and Knox, 1992). This has certainly encouraged the development of sensitive, reliable and precise methods for the analysis of Hg. Further, the organic forms of Hg, particularly methylmercury (CH_3Hg^+), are far more toxic than the inorganic forms (Hg^{2+}, Hg^0) of the pollutant (Rubi *et al.*, 1992; Bryan and Langston, 1992). In efforts to project long-term health risks and the ecological impact associated with trace amounts of Hg in the environment, reliable quantitation and accurate speciation at increasing lower levels are necessary.

The open-vessel digestion procedures and detection methods for total Hg analysis in water samples that are commonly used are based on acid leaching and permanganate/persulfate oxidation followed by cold vapor atomic absorption (CVAAS), (Szakács *et al.*, 1980; Van Delft and Vos, 1988). One of the most commonly used analytical technique for the determination of organomercurials is gas chromatography with electron capture detection (GC-

ECD), (Rubi *et al.*, 1992; O'Reilly, 1982) with or without pre-derivatization of the organic mercury compounds. The instrumentation and sample preparation of the existing methods strongly limit the ultimate sensitivity and efforts to lower the detection limits have not been entirely successful (Swift and Campbell, 1993). In addition, the ECD is an unselective detector and the column has to be tediously conditioned with large injections of Hg (II) chloride to alleviate poor chromatographic response to organomercurials (Hight and Capar, 1984; Rubi *et al.*, 1992; Uthe *et al.*, 1972; Bryan and Langston, 1992; Bulska *et al.*, 1992). These disadvantages demonstrate the need for the development of new methods in this field.

This paper describes atomic fluorescence-based methods for analyzing total Hg and organic Hg compounds at low part-per-trillion levels in environmental and biological samples. The atomic fluorescence (AFS) method (Bloom, 1989; Alli *et al.*, 1994) has become increasingly important compared to CVAAS, since the instrumental detection limit of this method is about 1 picogram or less and at least one order of magnitude better than for CVAAS (Lindqvist, 1993). Total Hg analysis involves three stages: sample digestion, cold vapor generation and atomic fluorescence detection.

In water samples, the difficulty in measuring MeHg and other organomercurials lies in concentrating these compounds from solution. This work employs a sulfydryl cotton fibre medium (Lee and Mowrer, 1989) which effectively adsorbs and preconcentrates trace levels of organomercurials. The organic Hg compounds are eluted with acidic potassium bromide and extracted into dichloromethane and subjected to GC analysis with AFS detection. Soil, sediment and tissue samples are treated with acidic potassium bromide and copper sulfate, and extracted with dichloromethane. The initial extracts are subjected to sodium thiosulfate clean-up subseqent to capillary gas chromatography with atomic fluorescence detection (Alli *et al.*, 1994).

2. Materials and Methods

2.1 SAMPLE COLLECTION AND PREPARATION

Surface water samples are collected in 2 L Teflon (Nalgene) bottles using a vacuum system. Samples are screened (105 μm Nytex netting) to prevent the collection of large particles with the water samples. All tubings and fittings used in the sampling system are constructed of teflon (SERP, internal SOP, 1994). The samples are collected by a "clean person" using double gloves (short vinyl gloves under shoulder length polyethylene gloves, OakTech) and a double bagging technique. All samples are placed in zip-lock polyethylene bags (Fisher Scientific), then in an additional plastic sample bag and placed in an icechest/cooler. In the clean room, concentrated hydrochloric acid, (trace metal grade, Fisher Scientific) is added to the water samples for preservation.

Surface, soil and sediment samples are collected using either a stainless steel spade, trowel or Eckman dredge. These samples are placed into wide mouth polyethylene specimen cups (125 ml, Fisher Scientific). Subsurface soil or sediment samples are collected in polycarbonate core tubes. Upon arrival to the laboratory the samples are immediately frozen to preserve their chemical integrity.

Fish samples are collected using a dip net, with the sampler wearing two pairs of gloves. The fish are placed in zip-lock sample bags, labelled, and stored in a cooler with ice for transport to the laboratory. Fish samples remain frozen until ready for analysis.

2.2 SAMPLE DIGESTION FOR TOTAL MERCURY DETERMINATION

Water Samples. Water samples are digested in a 125 mL teflon bottle with 1

mL HCl and 2.5 mL potassium bromate ($KBrO_3$)/potassium bromide (KBrO) mixture overnight (Szakács *et. al.*, 1980; Bloom and Fitzgerald, 1988). These samples are prepared and remain (in capped bottles) in the Hg-clean room. Prior to analysis, 500 µL hydroxylamine hydrochloride is added to destroy excess bromine and the samples thoroughly shaken.

Soil and Sediment Samples. Soils (such as peat, marls and marly peat) are first homogenized by adding 30 to 50 mL of deionized water and blended for 3 minutes to a uniform consistency with a blender (Osterizer). From the homogenized slurry 5 mL is diluted into 45 mL of 0.6N HCl to neutralize any carbonates, in a clean specimen cup. Of this mixture 1 mL is placed in a 10 mL ampule with 2 mL of concentrated nitric acid (HNO_3), (trace metal grade, Fisher Scientific). Volcanic soils are first sieved through a #80 mesh stainless steel sieve. In a 10 mL ampule, 0.5 g of the sieved sample is digested with 1 mL of 0.6N HCl and 2 mL HNO_3. Digested soil and sediment samples are left to stand under a fume hood for 20 minutes. The ampules are subsequently sealed and autoclaved for 1 h at 151°C. Before analysis the digestates are diluted with 0.12N HCl solution in a 20 mL polyethylene vial.

Fish Samples. To quantify total Hg in small fish (< 0.4 g, < 30 mm in length) the entire fish is weighed and placed in 10 mL ampules and digested using 1 mL deionized water and 2 mL concentrated HNO_3. After standing 20 minutes under a fume hood, the ampules are sealed and autoclaved as described above. For the analysis of larger fish (approximately 30 cm or longer), 3 tissue plugs (stainless steel core tube, 4 mm in diameter) are taken from the left side (using only muscle tissue), and combined to obtain a representative sample (approximately 0.4 g). The samples are then processed as indicated above for soil and sediment.

These digestion procedures result in the conversion of organic forms of Hg to inorganic mercury (Hg^{2+}). The digested samples are introduced to the cold vapor generator, at which point tin (II) chloride is used to effectively reduce inorganic mercury (Hg^{2+}) to its elemental gaseous form (Hg^0), prior to detection by atomic fluorescence.

2.3 INSTRUMENTATION AND ANALYSIS

A PS Analytical Merlin Mercury Fluorescence Detector System used in this study was supplied by P.S. Analytical Ltd. (UK). This system incorporates an Autosampler, Vapor Generator, Fluorescence Monitor and an IBM compatible Computer System. Instrument operating conditions for ultratrace and high levels of Hg concentrations are given in Table I.

Table I. Optimised operating conditions of the AFS System for total-Hg concentrations.

Ultratrace levels		*High levels*	
Carrier gas mL/min	125	Carrier gas mL/min	200
Sheath gas mL/min	150	Sheath gas mL/min	350
Calibration range	1000	Calibration range	100
Fine grain	4.0	Fine grain	2.5
Damping Switch	On	Damping Switch	On

Cold Vapor Generation. In the continuous flow vapor generator system, Hg(II) is reduced to Hg(0) following the addition of tin(II) chloride. The volatile Hg is stripped from the solution (in the gas liquid separator) by a carrier gas (argon). The rate of argon flow depends on whether the analysis is for ultratrace or high levels of Hg determination (P.S. Analytical, 1992).

Atomic Fluorescence Detection. A sheath gas (also argon) is used to channel the Hg vapor through a chimney past a light source and a photomultiplier tube that are at right angles to each other. With a specific high intensity Hg lamp source (Cathodeon Ltd., Cambridge) and a fixed 254 nm filter, efficient isolation of the required excitation and emission wavelengths is achieved (P.S. Analytical, 1992).

Reagents. All reagents used in total Hg analysis are of certified ACS grade and obtained commercially from Fisher Scientific, unless otherwise stated. A Barnstead B-pure system (located in the Hg-clean room) produces all deionized water used in making up reagents, sample digestates, calibration solutions, stock solution and quality control standards. This water is first filtered through a Culligan system consisting of activated charcoal and two mixed bed ion exchange cartridges before being piped to the Hg-clean room. 0.1 N $KBrO_3$, 0.2 N KBrO and 1.7 M hydroxylamine hydrochloride solutions are made up by dissolving the apppropriate amounts of the salts in deionized water. The $KBrO_3$ and KBrO salts are heated overnight in a glass vial at 250°C to remove adsorbed Hg. The digesting solution is made up daily by mixing equal volumes (100 mL) of 0.1N $KBrO_3$ and 0.2N KBrO solutions. All solutions are prepared weekly and stored in borosilicate bottles with teflon lined caps.

Standards. Working standards are prepared daily from a Hg stock solution (100 ng/mL) and diluted to the desired concentration. The stock solution is also made up daily from a commercially available mercury standard (1000 µg/mL, SPEX Industries, Edison, NJ). Calibration solutions are made up in 500 mL teflon bottles and stabilized by adding 5 mL concentrated HCl. No certified material exists for quality control of Hg in water near ultratrace levels. Soil (NIST sediment nominal value 60 ng/g, 8406) and tissue (NBS 1566a Oyster Tissue, 64 ng/g) quality control standards are obtained from the National Institute of Standards and Technology (Gaithersburg, MD).

2.4 ORGANIC MERCURY DETERMINATION

Sediment, Soil and Tissue Samples. A 1.0-5.0 g portion of the homogenized sample (as prepared above) is placed in a 20 mL borosilicate glass scintillation vial (Kimble, #74511). To the vial 5 mL distilled water, 3.0 mL of 1.0 M copper sulfate and 3.0 mL of acidic potassium bromide solution are added. The mixture is shaken for 1 hr at 330 rpm (Gyrotory Shaker Model G2). Dichloromethane (5 mL) is added and the mixture is shaken for 24 h at 330 rpm and then centrifuged for 10 min at 5000 x g in a Sorvall Model RC-5 refrigerated centrifuge (Dupont). An exactly known volume of the dichloromethane layer (3.5-4.0 mL) is transferred to a 7.0 mL borosilicate glass scintillation vial (Kimble, #0333726) and 1.0 mL of 0.01 M sodium thiosulfate is added. The mixture is shaken for 20 min at 330 rpm and centrifuged at high speed in a IEC clinical centrifuge. The aqueous layer (0.9 mL) is placed in a 2.0 mL microcentrifuge tube (Fisherbrand, Fisher Scientific), and 0.3 mL of 0.5 M copper chloride and 0.3 mL dichloromethane are added. The contents are mixed for 1 min on a Vortex Genie mixer and centrifuged for 2 min at high speed (16,749 x g) in a Hermle centrifuge. The dichloromethane is transferred to a 2.0 mL glass sampling vial containing a few crystals of anhydrous sodium sulphate and subjected to GC analysis. Injections of 5.0 µL are used. Samples spiked with known concentrations of methyl - and ethlymercury chloride are

extracted to evaluate the recovery factor used for quantification.

Water Samples. The sulfydryl-cotton (SFC) fibre columns are made of 1 mL disposable pipette tips containing 0.1 g of SFC fibre, packed loosely and as evenly as possible. Two SFC columns are connected in series and the water sample is passed through these by vacuum. One mL of acidic potassium bromide and 0.5 mL of 1.0 M copper sulfate are then pipetted on the surface of the adsorbent and the eluate is collected in a 2 mL micro-centrifuge tube (Fisher Scientific). This is extracted with 0.2 mL dichloromethane on a Vortex Genie mixer for 1.5 min and centrifuged as described above. The dichloromethane layer is then transferred to a 2 mL glass sampling vial containing a few crystals of anhydrous sodium sulfate and subjected to GC analysis.

2.5 INSTRUMENTATION AND ANALYSIS

A schematic diagram of the GC-AFS system used in this work is shown in Figure I and the optimum operating conditions are summarized in Table II. A

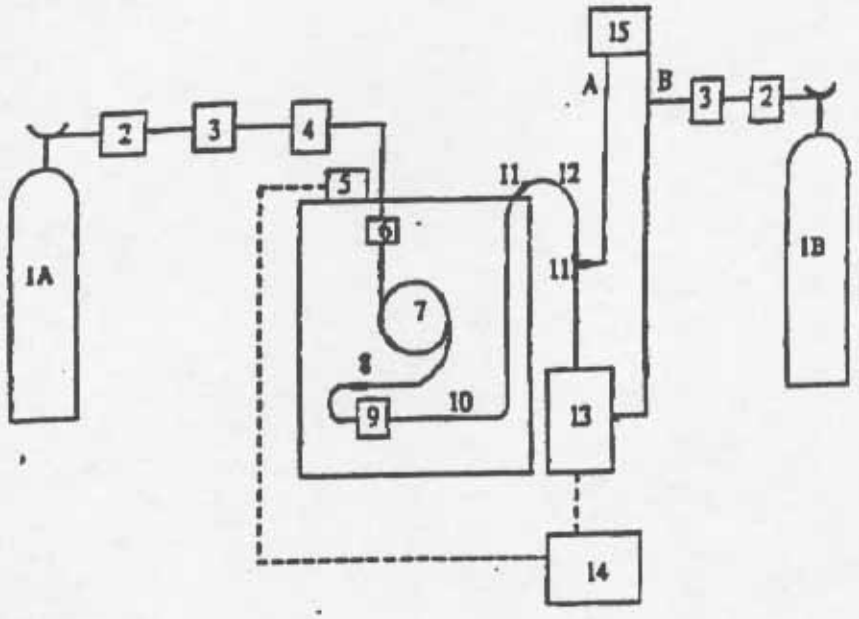

Figure I. Gas Chromatographic-Atomic Fluorescence Spectrometric System. 1A: Helium, 1B: Argon, 2: Oxygen trap, 3: Mercury trap, 4: Moisture trap, 5: Automatic sampler, 6: Injector, 7: Column, 8: Press-fit union, 9: Pyrolyser, 10: Deactivated fused-silica 0.53mm i.d., 11: Teflon unions, 12: Teflon transfer line 0.5mm i.d., 13: Atomic Fluorescence detector, 14: E-Lab chromatographic control and data acquisition system, 15: Mass flow controller-Channel A make-up, Channel B sheath gas.

Hewlett-Pakard (Model 5890 Series II) gas chromatograph coupled with an HP (Model 7673) automatic sampler is used. A fused-silica, bonded phase megabore column (15 m x 0.53 mm i.d., 1 μm non-polar DB-1 coating, J & W Scientific) and the splitless injection mode is employed. The effluent from the

column is led through a pyrolyser (P.S. Analytical Ltd., UK), positioned inside the GC oven via a piece of 65 cm length of deactivated fused-silica (0.53 mm i.d., J & W Scientific), which is connected to the column with a glass "press fit" union (J & W Scientific). The Hg atoms formed in the pyrolysis unit are transferred from the outlet end of the deactivated fused-silica tubing to the fluorescence detector (teflon transfer line, 0.5 mm i.d., Alltech Associates). The transfer line is passed through a small hole on the top of the GC oven to a Merlin Mercury Fluorescence Detector, and the connections are made *via* teflon unions.

Table II. Optimised operating conditions of GC-AFS.

Gas chromatograph.	
Injector temperature	250°C
Temperature program	1 min at 40°C, 60°C/min to 140°C, 3 min at 140°C, 50°C/min to 200°C, 10 min at 200°C.
Pyrolyser temperature	800°C
Column flow	4.0 mL/min
Make-up flow	60 mL/min
Atomic fluorescence system	
Sheath gas flow	300 mL/min
Integerate time	0.25s
Calibration range	1000 (most sensitive)
Fine gain	10 (maximum)
Recorder output voltage	1V
Damping switch (for signal smoothing)	On

A real time chromatographic control and data acquisition system (E-Lab, Version 4.10R, OMS TECH, INC.) is interfaced with the GC and AFS detector system. In this work, the detection limit is defined as the amount of Hg necessary to give a peak area equal to three times the standard deviation of the background signal.

Gases. All gases are supplied by Liquid Carbonic Speciality Gases and are of zero grade quality. Helium (99.995%) is used as the carrier gas (GC), passed first through an oxygen trap, then through a Hg trap (gold-activated carbon) and a moisture trap prior to the GC. Argon (99.998%) is employed as the make-up gas and the sheath gas for the GC-AFS system and is also passed through moisture and Hg traps before use. Its flow is regulated by a mass flow controller (Omega) equipped with two channels, channel A (make-up flow) and channel B (sheath gas flow, see Figure I).

Reagents. Double deionized water produced by a Barnstead B-Pure system is used in all solutions. Certified ACS grade potassium bromide, copper(II) sulfate, copper(II) chloride and sodium thiosulfate (Fisher Scientific) are used throughout this work. The acidic potassium bromide solution is prepared by dissolving 180 g in 200 mL water. Trace metal grade concentrated sulphuric acid (50 mL, Fisher Scientific) is added to 100 mL of water. After cooling to room temperature the solutions are mixed and made up to 1 L with water. Copper sulfate (1.0 M), copper chloride (0.5 M) and sodium thiosulfate (0.01 M) solutions are prepared by dissolving appropriate amounts of the salts in water. All solutions are extracted with dichloromethane prior to use.

Standards. All Hg standards are purchased from Ultra Scientific. Stock standard solutions of methyl- and ethylmercury chloride are prepared by dissolving appropriate amounts of the standards in optima grade methanol (Fisher

Scientific). These solutions are stored in dark brown bottles and diluted with dichloromethane to give working standards of the desired concentrations when required.

Synthesis of Sulfydryl-cotton (SHC) fiber adsorbent. This synthesis follows the procedure used by Lee and Mowrer (1989). A mixture is first prepared by adding the following reagents in sequence to round bottom flask: 100 mL thioglycolic acid, 60 mL acetic anhydride, 40 mL acetic acid (36%) and 0.30 mL concentrated sulfuric acid. The mixture is allowed to cool to 45°C, then 30 g of cotton wool are added and allowed to soak thoroughly in the mixture. The reaction bottle is placed in an oven for 3 to 4 days at 40°C, then the product is placed in a filter-funnel with suction filtration and washed thoroughly with deionized water to remove traces of thioglycolic acid. The SHC fiber obtained is dried at 40°C for 24 h and stored in the refrigerator.

3. Results and Discussion

Internal standard operating procedures (SERP, Internal SOP, 1994) for quality assurance purposes have been developed for ultratrace levels of Hg determination. The method detection limit (MDL, ppt), accuracy (%R) and precision (%RSD) for the various matrices and analytes considered in this study are shown in Table III. An MDL of 0.3 ng/L is achieved which is based on the US EPA method used for the calculation of this parameter. This number can also be viewed as the instrument detection limit, since the matrix of determination is a spiked blank that did not undergo the standard Hg digestion procedure. The method detection limit obtained is based on the analysis of seven replicate samples of spiked reagent blank water stabilized with concentrated HCl conducted on 3 nonconsecutive days. The standard deviation for each set of analyses is multiplied by the students' t value for a 99% confidence level and a standard deviation estimate with $n-1$ degrees of freedom is, $t = 3.14$ for seven replicates (US EPA, 1993). As shown in Table III, the MDL for water samples has a precision of about 5% relative standard deviation (%RSD) and recovery between 90 and 110%. The concentrations of Hg in sediment and tissue samples are significantly higher than water samples and can be determined at better precision (<5 %RSD) and accuracy (95 to 105 %R). The MDL is reevaluated every 6 months (SERP, Internal SOP, 1994).

In total-Hg determination, samples are prepared and analyzed according to the internal SOP established. The optimized operating conditions of the AFS System are listed in Table I, and as indicated, these parameters vary markedly depending on whether ultratrace levels or high levels of Hg are to be measured. In addition, the calibration levels used in the generation of daily calibration curves also depend on the level of Hg to be monitored in the sample. For ultratrace levels of Hg determination, calibration levels are 0, 10, 20 and 30 ng/L. Calibration levels for soil, sediment and fish samples are 0, 100, 250 and 500 ng/L. The linear correlation coefficient met EPA Contract Laboratory Program requirements of $\leq$ 0.995 (Inorganic USEPA CLP SOW 3/90).

Quality control checks are performed on NIST soil and tissue standards that are digested and autoclaved. Subsequent analysis of the digestates (1:20 dilution) yielded recovery values of 90 to 110% (58 to 70 ng/g) for the tissue standard and 95 to 105 % (57 to 63 ng/g) for soil standard. These values are within the acceptance criteria window for soil and tissue standards of $\pm$10%.

Table III. Precision, recovery and method detection limits for inorganic, total and organic Hg.

Analyte	Matrix	Precision (%RSD)	Recovery (%)R	MDL
Inorganic Hg	Water	≃5	90 - 110	0.3 ng/L
Total Hg	Water	≃5	90 - 110	0.3 ng/L
Total Hg	Tissue (NBS oyster tissue 1566a 64 ng/g)	<5	90 - 110	—
Total Hg	Soils, sediments (NIST sediment 8406 60 ng/g)	<5	95 - 105	—
Organic Hg ($MeHg^+$, $EtHg^+$)	Water	<5	98 - 110	0.02 ng/L
Organic Hg ($MeHg^+$, $EtHg^+$)	Soils, sediments, tissue	<5	67 - 80	—

In the analysis of organomercurials, the mercuric chloride conditioning of the GC column is associated with many drawbacks (Rubi *et al.*, 1992) and this procedure is a major limitation of the analytical technique. The aqueous phase ethylation technique derivatizes both inorganic mercury (Hg^{2+}) and ethylmercury ($C_2H_5Hg^+$) to diethylmercury [$(C_2H_5)_2Hg$] and thus the quantification of these species inherent in the sample can become difficult. These disadvantages indicate the need for the development of more straight-foward methods in the analysis of organic Hg compounds.

This work employed a capillary column for higher efficiency separation and a mercury fluorescence detector which affords better selectivity and sensitivity compared to the ECD. The configuration of the GC-AFS System is outlined in Figure I and the optimized operating conditions is shown in Table II. The extraction of soil, sediment and tissue samples involve a thiosulfate clean-up step and with this procedure, no mercuric chloride conditioning is necessary (Alli *et al.*, 1994). In addition, the thiosulfate back-extraction step effectively removes sample matrix interferences (high-molecular-weight compounds, possibly containing sulfur), which cause rapid stationary phase deterioration (Alli *et al.*, 1994; Lansens *et al.*, 1991; O'Reilly, 1982). A typical chromatogram of a sediment sample is shown in Figure II. Note that both methyl- and ethylmercury are efficiently separated.

In natural waters, organomercurials are present in very low concentrations and this is one of the major limitations in analyzing these compounds. The SHC fiber lends efficiently to solid phase extraction (SPE)

and allows for the analysis of trace levels of organomercurials. Further, since the SHC fiber has a high selectivity for organic mercury compounds, it avoids the extraction of extraneous compounds which causes severe column problems.

Quantitative data are obtained using the calibration curves generated daily. The chlorides of methyl- and ethylmercury are used to create the standard calibration curves expressed in terms of peak area *vs* organomercury chloride concentration (pg Hg/5 μL injection). The relative standard deviation of the signal for a 2 pg Hg/5 μL standard was 1.5% for peak area measurements (n = 3). The linear range used for the generation of calibration curves is 0 and

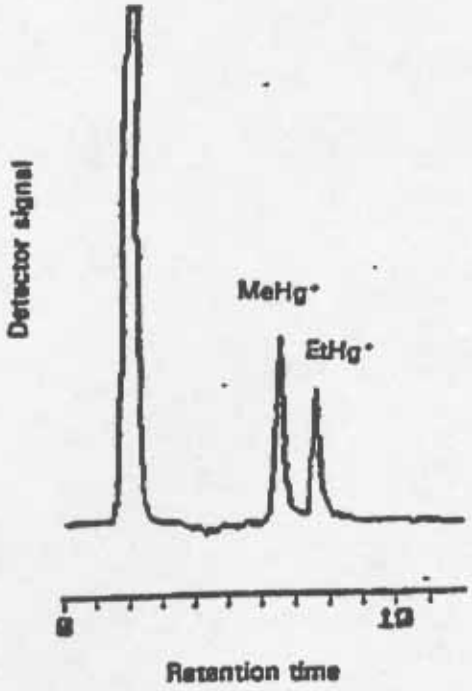

Figure II. (A) Chromatogram of pure organic mercury standards on new column. DMM: 2.00 pg Hg, MMC: 3.79 pg Hg, EMC: 2.50 pg Hg, (B) Chromatogram of sediment sample after thiosulfate clean-up (MM: 1945.7 pg Hg/g, EM: 1236.6 pg Hg/g)

4 pg Hg/μL and the linear correlation coefficients are typically 0.998 and 0.999 for methylmercury chloride and ethylmercury chloride respectively.

Quality control is maintained by determination of % recoveries for each sample. The recovery factor (%R) varies between 67 and 80% for soil, sediment and tissue samples, compared to 98 and 110% for water samples (Table III). This establishes the importance for determining a recovery factor for each sample since this value is influenced by differences in sample matrices which affect the partitioning of organic Hg compounds. Further support for this determination (%R) is evidenced by the lack of official standard materials (for organic Hg analysis) and the current need for internal standard(s).

4. Conclusion

Sealed ampule digestion of environmental and biological samples for total Hg determination described in this article is a relatively new method which provides accuracy of 95 - 105% recovery of Hg^0. Digestion of soil, sediment and fish samples in sealed 10 mL ampules is a clean and straightforward method for Hg determination. When these samples are autoclaved they liquify which makes it very easy to dilute samples suitable for AFS detection. Closed vessel digestion followed by cold vapor generation and atomic fluorescence detection has yielded detection limits that allow the quantification of ultratrace levels of Hg in water samples. The preparation techniques and use of an Hg-clean room have made it possible to reduce significantly contamination of samples.

In the speciation of organic Hg by GC, sample matrix interferences become adsorbed or bound to the stationary phase of the column after various injections, exerting a negative effect on the efficiency of the analysis. With a thiosulfate back-extraction step, these interferences can be effectively removed, allowing efficient analysis of the organomercurials. In water samples, the organic Hg compounds can be efficiently preconcentrated onto the SHC fiber, which also provide a sample clean-up. The column life becomes considerably longer with the clean-up step and can be used routinely for the analysis of organomercurials with no apparent loss in efficiency.

Acknowledgements

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APPENDIX C

Standard Operating Procedures for Total and Inorganic Mercury Analysis in Water, Sediment and Tissue

STANDARD OPERATING PROCEDURES FOR TOTAL AND INORGANIC MERCURY ANALYSIS IN WATER, SEDIMENT AND TISSUE

Southeast Environmental Research Program

Florida International University
University Park
Miami, Florida 33199

Version 10, November 19, 1997

(Previous versions written July 27, 1993; September 13, 1993; September 21, 1993; November 4, 1993; April 1, 1994; April 18, 1994; May 20, 1994; April 18, 1996; and September 15,1997.)

MERCURY

COLLECTION AND STORAGE

Water samples are collected in Teflon (FEP) bottles. Collection of samples is done using vinyl double gloves (Polyethylene shoulder length PPE glove, OakTech) bagging technique. Samples are then placed in zip-lock polyethylene bags, then placed in an additional plastic sample bag in a plastic ice chest/cooler. In the laboratory 10 ml of trace metal grade HCl is added to preserve samples. These additions are done in an Hg-clean room (described below). Soil samples are collected in polyethylene specimen cups (Elkay non-sterile wide mouth specimen cups with screw caps - 128 ml volume) and placed in polyethylene zip-lock bags. All field samples are kept in a cooler until they are returned to the laboratory. These coolers are used exclusively for low level Hg samples.

Acidified water samples may be stored in Teflon (Nalgene FEP) bottles in the Hg-free room or refrigerator. Refrigeration at 2°C and freezing at -20°C are used for storage of low level Hg samples. Teflon (FEP) bottles and storage of water samples in an Hg-clean room or refrigerator is recommended.

Water samples in Polyethylene (Nalgene LDPE) bottles stored in the Hg-clean room show high accumulation of Hg (refrigerator stored samples show minor accumulation of Hg) and cannot be used for storage of low level Hg samples. The plastic leaches mercury into the samples. This effect is facilitated by acid washing. Mercury accumulated in acid washed bottles to approximately 70-80 ppt with accumulation in non-washed bottles at 15 ppt in 30 days. Samples having higher levels of mercury (sediment and tissue), storage vessel type is not as critical.

ANALYTICAL METHODS

CLEAN ROOM

All glassware, acids, reagents, etc. are stored in the Hg-clean room. It is equipped with a bank of laminar flow hoods, a separate water supply and gold-charcoal filter apparatus, refrigeration unit, drying oven, analytical balance, and a "flypaper" covered floor which is changed when needed.

Contamination is checked weekly by monitoring acidified (1% HCl) replicate water samples which are stored open in the Hg-clean room. Data on this quality control monitoring is stored both as an Excel file on computer and as hard copy in a data notebook. If significant levels of Hg are found (>20 ppt) the source of contamination will be located and if necessary, gold and charcoal filters will be reconditioned.

CLEANING PROCEDURES

Teflon bottles which have been previously used for samples are rinsed three times with DIW and filled with 125 ml of 1% HCl. To this, 1 ml of mixed brominating agent (see reagent section for preparation) for every 50 ml of acid water is added and the bottle is shaken. This mixture remains in the bottles until it is used. Prior to using these bottles 500 µl of hydroxylamine hydrochloride is added to remove all free bromine. The bottle, bottle mouth and cap are then rinsed three times in DIW. Reusable teflon bottles (used for standards) are not rinsed.

Reusable laboratory glassware (graduating cylinders) are rinsed three times with DIW. The volumetric flask used for making up the primary standard, and reagent storage bottles are not rinsed.

Sediment cups, 20 ml scintillation vials (both plastic and glass) are non-reusable and discarded.

WATER SAMPLE PREPARATION FOR Hg ANALYSIS

Water samples may be analyzed for inorganic Hg and for total Hg. For inorganic Hg analysis samples are acidified as mentioned above and are then ready for analysis. Samples to be analyzed for total mercury are prepared using the following reagents:

REAGENTS

1) Mercury-free Water:

Tap water is first filtered through a Culligan system consisting of activated charcoal and two mixed bed ion exchange cartridges and then piped to the mercury-clean room. It is then passed through a Barnstead Mega-ohm B Pure system. This system is fitted with two filters (Thermolyne: colloid/organic-D0835, and ultrapure-D0809) in line with A 0.22 micron pleated particle filter. Mercury levels are not detectable by both our methods and independent laboratory analysis (<0.1 ppt). The only water available for use in the Hg laboratory is this Hg-free water. All reference to DIW in this SOP should be assumed to be Hg-free water as described above.

2) Bromination reagents:

0.1 M Potassium Bromate:
Heat 8.385 g $KBrO_3$ overnight in a glass scintillation vial (Kimble 74511) at 250°C ± 20°C in a furnace to remove mercury. After cooling dissolve the potassium bromate in 500 ml of deionized water and store in a borosilicate bottle. **Prepare weekly.**

0.2 M Potassium Bromide:
Heat 11.9 g KBr overnight in a glass scintillation vial at 250°C ± 20°C to remove mercury. After cooling dissolve the potassium bromide in 500 ml of deionized water and store in a

borosilicate bottle. **Prepare weekly.**

0.05 M Potassium Bromide (KBr): 0.1 M Potassium Bromate ($KBrO_3$)
Mix equal volumes (100 ml) of bromate and bromide in a 150 ml screw cap teflon borosilicate bottle. **Prepare daily.**

3) Hydroxylamine Hydrochloride
Dissolve 6.0 g of $NH_2OH.HCl$ in 50 ml of deionized water in a 60 ml teflon bottle. **Prepare weekly.**

4) Stannous Chloride: To 40 g of Stannous Chloride ($SnCl_2$) add 50 ml of 12 N HCl. Bring to 2000 ml using Hg-free deionized water in a borosilicate glass bottle. **Prepare daily.** The stannous chloride is made Hg-free by purging it with argon for 20 minutes before running samples. Thereafter, the purging continues throughout the entire analysis.

5) 12 N HCl: Concentrated HCl (12 N HCl) is poured into a graduate cylinder which has been previously acid washed and rinsed three times with DIW.

6) Wash water: 150 ml of concentrated HCl (12N HCl) is added to 15 L of DIW in a 15 L teflon bottle (Nalgene lowboy) and shaken.

DIGESTION OF WATER SAMPLES

Samples are placed in an ultraviolet cabinet for 12 hours, allowed to cool, and then brominated for one hour in the Hg-clean room. 125 ml of acidified sample (0.625 ml 12 N HCl is added to each 125 ml sample) is brominated by adding 2.5 ml $KBrO_3$/KBr mixed reagent (as described above, Reagents section #2) to each sample bottle. Then, 500 µl of hydroxylamine hydrochloride is added to the solution to inhibit further reaction. Samples are permitted to settle for at least 10 min before analysis.

SOILS AND SEDIMENTS (CARBONATE AND CLASTIC)

Preparation of soil and sediment samples is done outside the Hg-clean room. Sediment samples are homogenized and slurried using a glass bottled blender (which is cleaned in between samples by rinsing three times with tap water). 120 ml of sediment is slurried with 50 ml of distilled water. This mixture is then blended for 3 minutes. Using a syringe, 10 ml of slurry are removed, placed in a polyethylene specimen cup and diluted by adding 40 ml of 5% HCl (The HCl acts to neutralize carbonate sediments prior to digestion. It is necessary to prevent a violent reaction when the ampule is subsequently sealed and autoclaved). After mixing, 1 ml of this solution is transferred to a 10 ml ampule using a 1000 cc syringe (with the tip cut off). Nitric acid (2 ml conc. HNO_3) is added to the ampule which is left to stand for 20 minutes. The ampule is then sealed and autoclaved for 1 hr at 105°C. Ampules must be cooled completely before further processing.

To process ampule contents, pipette 0.5 ml of the digested solution into a 20 ml polyethylene scintillation vial (Kimble # 58504) containing 20.0 ml of 0.12 N HCl solution.

PLANT AND ANIMAL TISSUES

Animal and plant tissue are treated the same as sediments. Initial dilutions of homogenate vary with the type of tissue. In addition the HCl step used to neutralize carbonates is not used for tissue analysis. For small fish (such as Gambusia), the entire fish is weighed, placed in ampules and digested as for sediments. For large fish (bass and catfish), using a stainless steel core tube, three 4 mm cores (without scales or bones) from the left fillet are combined and weighed. The cores are weighed in 10 ml ampules and digested as above for sediments. The weight of tissue sample that is used for analysis is usually between 0.3 g and 0.4 g. Tissue samples weighing more than 0.4 g tend to explode the ampules in the autoclave.

DIGESTION OF STANDARD REFERENCE MATERIAL AND SPIKED MATERIAL

A series of method tests have been run both with spiked tissue, spiked sediment, and NBS or NIST certified samples to test for digestion efficiency. NBS oyster tissue (566a), NRCC dogfish muscle (DORM-2), NIST sediment nominal 50 μg/g (8407) and 60 μg/g (8406) were used in these tests. In addition, a sample of certified material is digested and run with each analysis of tissue or sediment. Digestion efficiency is between 98-102% in all cases.

SAMPLE STORAGE AFTER PREPARATION

Sediment and tissue samples may be stored in the sealed ampules for an indefinite period after they have been autoclaved.

STANDARD PREPARATION

All preparation and storage of working standards is done in a Hg-clean room. Secondary standard is prepared and stored outside the Hg-clean room (because of its high Hg concentration). The primary stock standard is made by addition of 100 μl of NBS certified secondary Hg standard (SEPEX PLHG4-2X) (1000 μg/ml) to deionized water plus 10 ml of trace metal grade HCl, and made up to 1 L in a 1000 ml flask. **This standard is prepared daily.** Working standards are made in 500 ml teflon (FEP) bottles. For each working standard, concentrated HCl (5 ml) is added to 495 ml of deionized water. When acids and brominating agents are added, the external laboratory hood is turned on creating a negative pressure in the area where acid addition is being done. The primary stock is then brought into the Hg-clean room and depending on final concentration, the required amount of primary stock is added to the bottles containing the water-acid mixture. **Working**

standards are made up daily.

The working standard used for low level Hg-concentration (e.g. water samples) are 0, 2.5, 5 and 10 ppt. In the 500 ml acid-water mixture, 0, 12.5, 25 and 50 µl of the stock are used respectively. The 5 ppt standard is used as the continuing calibration standard and the 1 ppt is run as a low level check. All pipettes, micropipettes and pipette tips are calibration checked before use, using an analytical balance. The temperature of the clean room is approximate 21°C.

Secondary standards used for tissue and sediment analysis are 0, 100, 250 and 400 ppt. These required standards are made up in the 500 ml acid-water mixture, using 0, 0.5, 1.25 and 2.0 ml of the secondary standard respectively. The 250 ppt standard is used as the continuing calibration standard while the 100 ppt is run as a low level check .

ANALYTICAL INSTRUMENTAL TECHNIQUE

Cold Vapor Atomic Fluorescence Spectrometry (CVAFS) is the method used for Hg determination. The system used is a PSA Merlin Plus supplied by Questron corporation, Princeton, New Jersey 08543. This system contains an autosampler, vapor generator, fluorescence monitor, and an IBM-compatible computer system as the electronic data interface. In the CVAFS method, $SnCl_2$ is mixed with the liquid sample fed by the auto sampler, which then enters a gas liquid fritted separator. The sample flows through peristaltic pump tubing. As mercury enters the vapor phase it is stripped and carried along a gas stream (Argon- Zero grade) to the detector. The method detection limit (MDL) is approximately 0.255 ppt (S.D X 3). Baseline noise translates into variation of between 0.087-0.185 ppt.

Modifications to the apparatus are:

1) The pump in the hydride generator has been changed to a peristaltic pump.

2) Modification of the computer output using Excel to permit more accurate representation of the peak height data.

3) An Omega model FMA-7882 mass flow controller with a channel selector is installed at the front of the instrument. This flow controller is used to more accurately regulate flow rates of the carrier and sheath gas, while the flow controllers on the hydride generator are open to full capacity.

Procedure for operating the instrument:

1) Tighten the peristaltic pump (pumps wash water, waste water, sample, and stannous chloride).

2) Turn on the wash water to the system.

3) Turn on the computer.

4) Turn on the gas to the system. The argon (Zero grade) flows through two gas purifiers (charcoal and gold) before reaching the instrument.

5) Turn on the line stabilizer/conditioner.

6) Check to make sure no tubes are crimped, and that flow is smooth in all tubes before proceeding.

7) Check gas flow at the mass flow controller.
Note: For low level Hg-concentrations the optimum level of the carrier gas has been determined to be 0.14 L/min, while the sheath gas level has been optimized at 0.125 L/min. At higher Hg-concentrations the carrier gas is 0.35 L/min, and the sheath gas is 0.2 L/min.

8) Allow the system to run on DIW for 15 minutes.

9) After 15 minutes switch the instrument to $SnCl_2$.

10) Note: the sensitivity dial on the instrument is run at highest sensitivity for water but may be lowered for running of sediment, soil and tissue samples. This method is adequate for samples of the range we have run to date.

11) When the instrument is ready, zero the fluorescence detector and run acidified water (0 ppt) to check baseline response of the instrument and guard against unexplained contamination from reagent preparation. When peak height of DIW is ≤MDL (0.0-0.3) the standards may be run. Initially run one high standard to test for consistency of standard preparation and machine function. The range of standards will reflect the concentration of samples to analyze. Eight standards (four concentrations, two replicates) are run for each standard curve. Standards run for low level samples are 2.5, 5, and 10 ppt.

12) Three reagent blanks containing different amounts of reagents are digested to determine the amount of mercury added to each sample by the reagents. the reagent blanks are all made up in DIW from the Hg-clean room, so it is important that the amount of Hg in the water be determined. To reliably calculate the mercury concentration in the reagent blanks, the following procedure is used:

Reagent blank I - 1.5 ml $KBrO_3$/KBr mixture + 0.25 ml hydroxylamine hydrochloride.
Reagent blank II -2.50 ml $KBrO_3$/KBr mixture + 0.50 ml hydroxylamine hydrochloride.
Reagent blank III-5.00 ml $KBrO_3$/KBr mixture + 1.00 ml hydroxylamine hydrochloride.

After the reagent blanks are analyzed, the best fit line is obtained: $y = m x + b$, where y represents the peak height unit, x represents the volume of reagents in each sample (3 ml)

and b represents the contamination by water. The mercury concentration is then obtained from the standard curve.

All reagent blanks are made up to a final volume of 125 ml. The slope obtained from this curve give the concentration of mercury in the reagents. This number is subtracted from sample values.

12) Standards, blanks, and high level samples (generally fish, sediments and soil) may be run in plastic scintillation vials. Water samples for total-Hg are digested and analyzed in 125 ml teflon bottles. Each 125 ml water sample is analyzed at least three times. Tissue and sediment samples are run in replicate. When using the autosampler, fifty samples including standards takes approximately 2-1/2 hours. Each sample uses 10 ml of $SnCl_2$ per sample. A new standard curve is run when the $SnCl_2$. In addition to running a full set of standards at the beginning of the analysis for each bottle of stannous chloride, a replicate of the highest standard and zero ppt are run after every 10 samples.

Note: When sampling from 125 ml teflon bottles, the auto sampler tray is removed and the connections are modified to sample only from the right sampling tube. This is done by disconnecting the right (internal) sampling tube as well as the corresponding tube to the hydride generator and replaced with a longer teflon tube that directly connects the sampling tube to the hydride generator. After every 12 analyses and at the end of the run, the mid-level and 0 ppt standards are analyzed in duplicate.

Instrument Shutdown:

1) If you are using the results directly from the company supplied computer program, make sure you have printed and/or saved results. This program does not reliably transfer files to ascii or Excel although it has functions for these tasks.
2) Replace the $SnCl_2$ solution with DIW and flush the instrument for 5 minutes.
3) Turn off the wash water.
4) Run the pump until no more liquid is present in the pump tubing.
5) Turn off the gas.
6) Turn off the line stabilizer and the computer.
7) Release tubing in the Hydride generator and peristaltic pump.
8) Check the waste water container and empty if necessary.

Computer Procedure:

1) Choose LIBRARY, press select to choose methods and to see methods stored.
2) For ANALYSIS choose "analyze", "batch". Specify batch (sample) size. The computer will ask you whether the sample tray is in position and if you wish to change the sample tray. If you respond 'NO' twice, the instrument will then align the sample tray to the run you have specified. It will then be necessary to return to the analyze menu and check batch etc. you may then respond "YES" to the questions about tray position. This response will

initiate the run. The instrument will analyze 50 samples. If you have more than 50 samples you must re-select analyze and you can choose a reference number which reflects the actual number of samples you are running.
3) To run standards, select "CALIBRATION" then select new curve.

DATA TRANSFER

We do not find the program supplied with the CVAFS adequate for our needs. Specifically, the curve fitting function is not adequate for low level samples (< 1 ppt). We therefore after printing results from the machine, save the data as an ascii file and transfer it into an excel spreadsheet.

DATA HANDLING

For water samples, during each run a sample is spiked with 1 ppt mercury concentration. The mercury concentration of the sample is then subtracted from the mercury concentration of the spiked sample. Fish and soil samples are not spiked for total mercury analysis and the recovery is determined by using NIST reference materials.

THE STANDARD CURVE
Currently the NIST standard available for Hg is 1000 µg/ml. With appropriate dilutions, standards can be made reproducible to 10 ppt using standard dilution and pipetting procedures. The intercept location in the standard curve calculation becomes critical to proper calculation of concentrations. We have found that the most reliable and reproducible results are generated by running a set of standards, and then checking if the standard regression is acceptable (linear coefficient >0.98). If the regression coefficient is acceptable and the mid-level continuing standards are ± 10% of the expected value, the curve is dropped parallel through the origin. The peak height units for each sample are then compared to this new curve to determine Hg concentration. This method is used for water samples and has also been found to be comparable to traditional estimating procedures used in sediment, soil and tissue analysis.

All data is printed as hard copy and stored on computer disks. We maintain a back-up copy for each disk.

APPENDIX D

Laboratory Documentation (Sample Prep Logs, Analytical Analysis, Standard Curves, Sample Calculations) for Water, Sediment, and Tissue Samples

Table 1. PREPARATION OF SEDIMENT SAMPLES FOR ORGANIC MERCURY ANALYSIS

Name of project: _____ No. of samples: _____

Technician: _____ Date of Analysis: _____

SOP #/ Date: _____

Sample No.	Cup Wt. (g)	Weight of wet sed. (g)	Weight of dry sed. + cup	Weight of dry sed.	Ratio of dry/wet wt.

Blend 125cc of soil with DI water.

_____

Date Time Init

Weigh 5mL of slurry and dry in dyring oven at 80°C overnight.

_____

Date Time Init.

Take out oven and allow to cool.

_____

Date Time Init

Weigh dry sediment

_____

Date Time Init.

Table 2. PRE-TREATMENT FOR SEDIMENT ORGANOMERCURY ANALYSIS

Samples:
Technican:

Number of samples:
Date:

Samp. No	W.S. g	1st. V	2nd. V	Ref	Samp. No	W.S g	1st V	2nd V	Ref

Spiked amount: ul of 5pg/ul solution.
Last step CH2Cl2: ul
Please do not forget reference stardand.

Table 3. GC-RESULTS FOR ORGANOMERCURY ANALYSIS

GC file name:
GC analysis date:

Pretreatment Date:

Samp. No	GC ID	MeHg	EtHg	Ref	Samp. No	GC ID	MeHg	EtHg	Ref

SOUTHEAST ENVIRONMENTAL RESEARCH PROGRAM
ORGANIC MERCURY ANALYSIS
DATA INTEGRATION

Name of project:_____ No. of samples:_____

Technician:_____ Date of analysis:_____

SAMPLE ID	FILE NAME	METHYL Hg		ETHYL Hg	
		Auto int.	Manual int.	Auto int.	Manual int.

Tab 1.PREPARATION OF WATER SAMPLES FOR ORG-HG ANALYSIS

Sample source:
Technican:

Sample numbers:
Prep. Date:

Samp.No.	Vol.ml	Orig.PH	Buffer	Fin.PH	CH2Cl2	notes

Reference Stardands:

amount of 5pg/ul mixed stardand added: _____ ul
amount of CH2Cl2 added: _____ ul

SOUTHEAST ENVIRONMENTAL RESEARCH PROGRAM
TOTAL MERCURY ANALYSIS

NAME OF PROJECT: _____ MATRIX: _____

DATE OF ANALYSIS: _____

TECHNICIAN: _____

REF. #	SAMPLE ID	DILUTION	COMMENTS

SOUTHEAST ENVIRONMENTAL RESEARCH PROGRAM
TOTAL MERCURY IN WATER

SOP # AND REVISION DATE: ______________ NAME OF PROJECT: ________________

DATE OF PREPARATION: ______________

DATE OF ANALYSIS. ________________

TECHNICIAN: ________________________

REF. #	SAMPLE ID	BOTTLE #	DESCRIPTION

DIGESTION PROCEDURE

Split water samples into 125mL teflon bottles and acidify with 0.625mL 12N HCl. Then place the samples in the ultraviolet cabinet for 12 h, allow to cool, then brominate for 1 h.

REAGENTS:

Weigh out 8.385g of $KBrO_3$ and 11.9g of KBr Heat overnight in a muffle furnace at 250°C.

Date Time Init.

Take out of muffle furnace and allow to cool

Date Time Init.

Dissolve each reagent in DDI water and make up to a final volume of 500mL. Store reagents in borosilicate bottles and they last for 1 week.

Date Time Init.

Mix equal vol. (100mL) of $KBrO_3$ and KBr reagents are and store in a borosilicate bottle. Make this daily.

Date Time Init.

Dissolve 12g of hydroxylammine hydrochloride in 100mL DDI water. Make this weekly and store in a borosilicate bottle.

Date Time Init.

Add 2.5mL of mixed reagent to each sample and digest for 1 h.

Date Time Init.

After digesting for 1h add 0.5 mL hydroxylamine hydrochloride to each sample.

SOUTHEAST ENVIRONMENTAL RESEARCH PROGRAM
TOTAL MERCURY IN FISH

:P AND REVISION DATE._____ NAME OF PROJECT:

:TE OF PREPARATION:_____ _____

:CHNICIAN:_____

:MPLE ID	WT. OF FISH	SEX	LENGTH	COMMENTS

DIGESTION PROCEDURE:

Determine the length and sex of fish and weigh in a 10mL glass ampule.

Date Time Init.

Add 1mL of DDI water in 2mL conc. HNO3. Leave for 20 min. under the hood. Then seal.

Date Time Init.

Place in autoclave in a water-bath for 1 hour at 105° C.

Date Time Init.

Date of analysis:

Date Time Init.

SOUTHEAST ENVIRONMENTAL RESEARCH PROGRAM
TOTAL MERCURY IN SOIL

SOP No. AND REVISION DATE. ____________________

DATE OF PREPARATION: ____________________

TECHNICIAN: ____________________

NAME OF PROJECT

____

SAMPLE ID	DESCRIPTION	CUP WT.	WET WT.	DRY WT.

DIGESTION PROCEDURE:

Blend '125 cc of soil with DI water. Add 45mL of 0.6N HCl to 5mL of slurry and leave for 1h.

Date Time Init.

Take 1mL of acidified slurry and pipette into a 10mL glass ampule Add 2mL conc. HNO_3. Leave for 20 min under the hood.

Date Time Init.

Weigh 5mL of slurry and dry in drying oven (80°C) overnight.

Date Time Init.

Take out of oven

Date Time Init.

Place in autoclave in a waterbath for 1 hour at 105° C.

Date Time Init.

DATE	MODEL	WEIGHT	OK/NOT	INITIAL
10 27.97	P100 Clean	0.200	✓	IM
10 28.97	P1000 Clean (2)	0.999	✓	IM
10 28.97	P100 Clean	0.200	✓	IM
10 28 97	P100 Hg	0.099	✓	IM
10 29 97	P100 Hg	0.099	✓	IM
10 29.97	P100 Clean	0.100	✓	IM
30 97	P100 Hg	0.100	✓	IM
10 30.97	P100 Clean	0.101	✓	IM
31.97	P100 Clean	0.100	✓	IM
10.31.97	P1000 Clean	1.001	✓	IM
10 31 97	P100 Hg	10.101	✓	IM
10 31 97	P5000 Clean	4.993	✓	IM
11-02-97	P1000 Clean (2)	0.997	✓	IM
11-02-97	P100 Clean	0.100	✓	IM
11-02-97	P100 Hg	0.100	✓	IM
11-04-97	P100 Hg Lab	0.100	✓	IM
11-04-97	P100 Clean	0.099	✓	IM
11-04-97	P200 Clean	0.200	✓	IM
11-04-97	P1000 Clean (2)	1.001	✓	IM
11-04-97	P5000 Clean	5.012	✓	IM
11-5-97	P100 Hg	0.099	✓	IM
11-05-97	P100 Clean	0.099	✓	IM
11-05-97	P1000 Clean (2)	1.002	✓	IM
11-05-97	P5000 Clean	4.998	✓	IM
11/6/97	P200 Clean	0.200	✓	NOT.
11-10/97	P200 Clean	0.199	✓	IM
11-10-97	P100 Clean	0.099 IM	✓	IM
11-10 97	P100 Hg	0.996	✓	IM
11-10-97	P5000 Clean	4.967	✓	IM

Water (Merlin) sent to LPT1 on 11 Nov 94

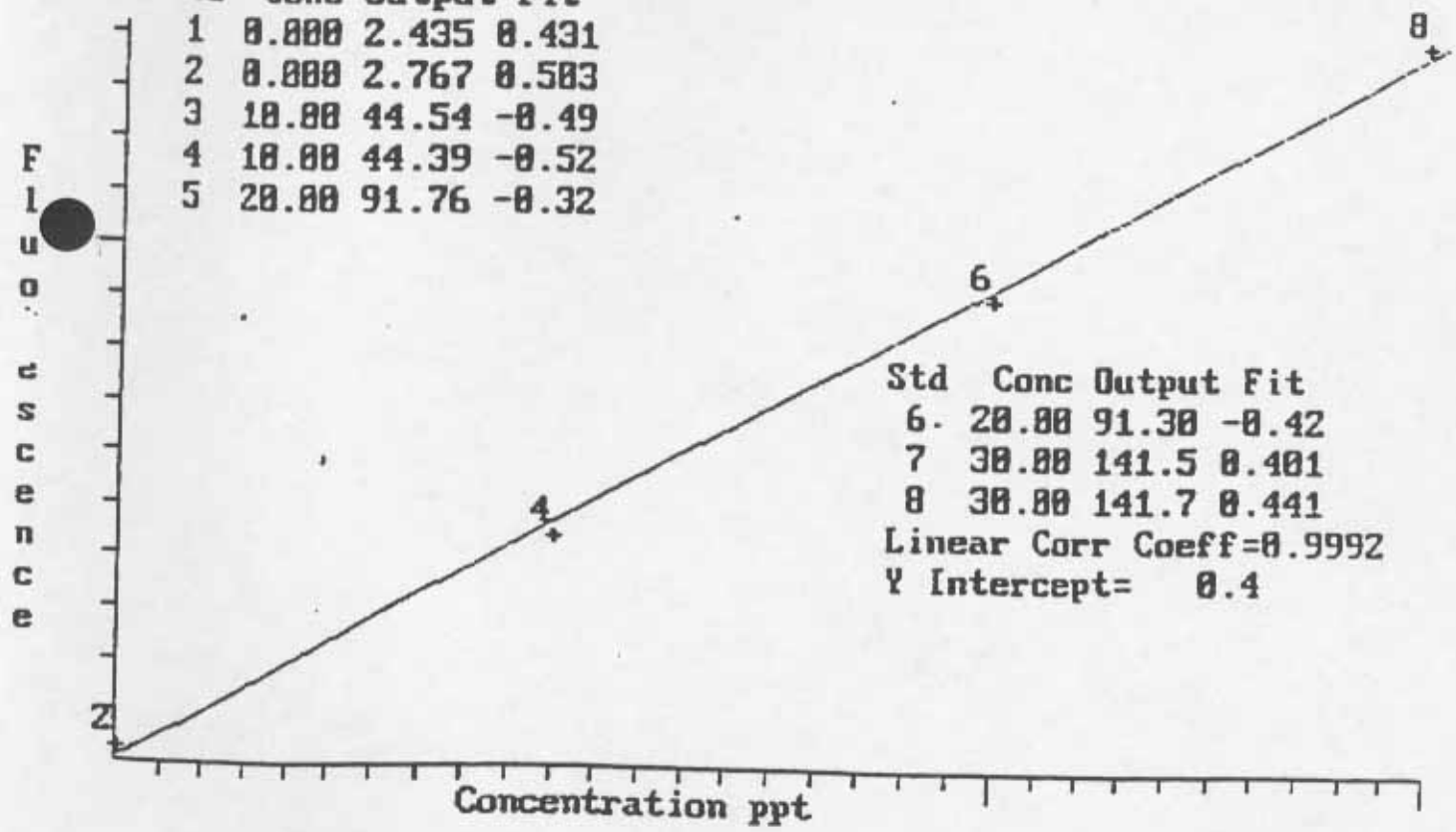
	Tag	Ref	Output	Concentration	Runs	SD	Time	Date
1	EPAF0994	001	0.0	0.000ppt	1	0.00ppt	21:45	10 Nov
	EPAF0994	002	1.1	0.000ppt	1	0.00ppt	21:49	10 Nov
	EPAF0994	003	0.0	0.000ppt	1	0.00ppt	21:54	10 Nov
4	EPAF0994	004	188.1	>30.00ppt	1	0.00ppt	22:01	10 Nov
5	EPAF0994	005	0.0	0.000ppt	1	0.00ppt	22:06	10 Nov
6	EPAF0994	006	2.6	0.000ppt	1	0.00ppt	22:10	10 Nov
7	EPAF0994	007	0.1	0.000ppt	1	0.00ppt	22:14	10 Nov
8	EPAF0994	008	0.4	0.000ppt	1	0.00ppt	22:18	10 Nov
9	EPAF0994	009	144.6	55.68ppt	1	0.00ppt	22:24	10 Nov
10	EPAF0994	010	2.8	0.000ppt	1	0.00ppt	22:28	10 Nov
11	Std 1	Hg	2.4	0.00ppt	1	0.00 O/P	22:32	10 Nov
12	Std 2	Hg	2.8	0.00ppt	1	0.00 O/P	22:36	10 Nov
13	Std 3	Hg	44.5	10.00ppt	1	0.00 O/P	22:40	10 Nov
14	Std 4	Hg	44.4	10.00ppt	1	0.00 O/P	22:44	10 Nov
15	Std 5	Hg	91.8	20.00ppt	1	0.00 O/P	22:49	10 Nov
16	Std 6	Hg	91.3	20.00ppt	1	0.00 O/P	22:53	10 Nov
17	Std 7	Hg	141.6	30.00ppt	1	0.00 O/P	22:58	10 Nov
18	Std 8	Hg	141.7	30.00ppt	1	0.00 O/P	23:03	10 Nov
19	EPAF0994	011	1.5	0.234ppt	1	0.00ppt	23:09	10 Nov
20	EPAF0994	001	5.4	1.071ppt	1	0.00ppt	23:13	10 Nov
21	EPAF0994	002	5.3	1.054ppt	1	0.00ppt	23:18	10 Nov
22	EPAF0994	003	5.4	1.080ppt	1	0.00ppt	23:22	10 Nov
23	EPAF0994	004	5.3	1.053ppt	1	0.00ppt	23:26	10 Nov
24	EPAF0994	005	4.1	0.793ppt	1	0.00ppt	23:30	10 Nov
	EPAF0994	006	5.6	1.117ppt	1	0.00ppt	23:34	10 Nov
	EPAF0994	007	4.9	0.971ppt	1	0.00ppt	23:38	10 Nov
27	EPAF0994	008	4.0	0.768ppt	1	0.00ppt	23:42	10 Nov
29	EPAF0994	009	6.0	1.207ppt	1	0.00ppt	23:46	10 Nov
	EPAF0994	010	0.7	0.052ppt	1	0.00ppt	23:51	10 Nov
30	EPAF0994	011	0.6	0.034ppt	1	0.00ppt	23:55	10 Nov
31	EPAF0994	012	0.6	0.034ppt	1	0.00ppt	23:59	10 Nov
32	EPAF0994	013	144.4	31.01ppt	1	0.00ppt	00:05	11 Nov
33	EPAF0994	014	143.5	30.81ppt	1	0.00ppt	00:11	11 Nov
34	EPAF0994	015	2.4	0.415ppt	1	0.00ppt	00:15	11 Nov
35	EPAF0994	016	2.6	0.477ppt	1	0.00ppt	00:19	11 Nov
36	EPAF0994	017	1.2	0.168ppt	1	0.00ppt	00:23	11 Nov
37	EPAF0994	018	0.0	0.000ppt	1	0.00ppt	00:27	11 Nov
38	EPAF0994	019	0.2	0.000ppt	1	0.00ppt	00:31	11 Nov
39	EPAF0994	020	0.5	0.014ppt	1	0.00ppt	00:35	11 Nov
40	EPAF0994	021	1.4	0.201ppt	1	0.00ppt	00:39	11 Nov
41	EPAF0994	022	1.3	0.187ppt	1	0.00ppt	00:43	11 Nov
42	EPAF0994	023	0.3	0.000ppt	1	0.00ppt	00:48	11 Nov
43	EPAF0994	024	0.2	0.000ppt	1	0.00ppt	00:52	11 Nov
44	EPAF0994	025	0.7	0.067ppt	1	0.00ppt	00:56	11 Nov
45	EPAF0994	026	1.3	0.192ppt	1	0.00ppt	01:01	11 Nov
46	EPAF0994	027	2.5	0.438ppt	1	0.00ppt	01:05	11 Nov
47	EPAF0994	028	0.3	0.000ppt	1	0.00ppt	01:09	11 Nov
	EPAF0994	029	143.1	30.72ppt	1	0.00ppt	01:15	11 Nov
	EPAF0994	030	141.8	30.46ppt	1	0.00ppt	01:20	11 Nov
50	EPAF0994	031	2.8	0.504ppt	1	0.00ppt	01:25	11 Nov
51	EPAF0994	032	3.4	0.631ppt	1	0.00ppt	01:29	11 Nov
52	EPAF0994	033	1.4	0.201ppt	1	0.00ppt	01:33	11 Nov
53	EPAF0994	034	0.9	0.094ppt	1	0.00ppt	01:37	11 Nov
	EPAF0994	035	0.4	0.000ppt	1	0.00ppt	01:41	11 Nov

63	EPAF0994	044	0.9	0.104ppt	1	0.00ppt	02:1	11	Nov
64	EPAF0994	045	143.1	30.72ppt	1	0.00ppt	02:24	11	Nov
65	EPAF0994	046	141.2	30.32ppt	1	0.00ppt	02:30	11	Nov
66	EPAF0994	047	3.5	0.670ppt	1	0.00ppt	02:34	11	Nov
67	EPAF0994	048	3.6	0.689ppt	1	0.00ppt	02:38	11	Nov
68	EPAF0994	049	0.1	0.000ppt	1	0.00ppt	02:42	11	Nov
69	EPAF0994	050	1.5	0.220ppt	1	0.00ppt	02:46	11	Nov
70	EPAF0994	051	0.4	0.001ppt	1	0.00ppt	02:50	11	Nov
71	EPAF0994	052	1.9	0.314ppt	1	0.00ppt	02:54	11	Nov
72	EPAF0994	053	1.3	0.187ppt	1	0.00ppt	02:58	11	Nov
73	EPAF0994	054	2.6	0.466ppt	1	0.00ppt	03:02	11	Nov
74	EPAF0994	055	141.2	30.31ppt	1	0.00ppt	03:08	11	Nov
75	EPAF0994	056	140.8	30.24ppt	1	0.00ppt	03:12	11	Nov
76	EPAF0994	057	2.1	0.359ppt	1	0.00ppt	03:16	11	Nov
77	EPAF0994	058	1.9	0.306ppt	1	0.00ppt	03:20	11	Nov

```
Reading Std 1 Run    1 Peak Height=     2.4   Peak Area=    61.0
   Std 1      Hg       2.4       0.00ppt      1     0.00 O/P    22:32
Reading Std 2 Run    1 Peak Height=     2.8   Peak Area=   143.5
   Std 2      Hg       2.8       0.00ppt      1     0.00 O/P    22:36
Reading Std 3 Run    1 Peak Height=    44.5   Peak Area=  2549.5
   Std 3      Hg      44.5      10.00ppt      1     0.00 O/P    22:40
Reading Std 4 Run    1 Peak Height=    44.4   Peak Area=  2543.5
   Std 4      Hg      44.4      10.00ppt      1     0.00 O/P    22:44
Reading Std 5 Run    1 Peak Height=    91.8   Peak Area=  5265.5
   Std 5      Hg      91.8      20.00ppt      1     0.00 O/P    22:49
Reading Std 6 Run    1 Peak Height=    91.3   Peak Area=  5236.0
   Std 6      Hg      91.3      20.00ppt      1     0.00 O/P    22:53
Reading Std 7 Run    1 Peak Height=   141.6   Peak Area=  8185.8
   Std 7      Hg     141.6      30.00ppt      1     0.00 O/P    22:58
Reading Std 8 Run    1 Peak Height=   141.7   Peak Area=  8087.0
   Std 8      Hg     141.7      30.00ppt      1     0.00 O/P    23:03
```

Hg in Water (Merlin) Fit : Least Squares Straight Line
Slope=4.64229 Term2=0.00000 Term3=0.00000 No Reslope

Std	Conc	Output	Fit
1	0.000	2.435	0.431
2	0.000	2.767	0.583
3	10.00	44.54	-0.49
4	10.00	44.39	-0.52
5	20.00	91.76	-0.32



Std	Conc	Output	Fit
6	20.00	91.30	-0.42
7	30.00	141.5	0.401
8	30.00	141.7	0.441

Linear Corr Coeff=0.9992
Y Intercept= 0.4

Printed from TouchStone 10 Nov 94

```
PAF0994 Ref 011 Run    1 Peak Height=     1.5   Peak Area=    15.8
PAF0994      011       1.5       0.234ppt      1     0.00ppt     23:09
```

11-11-94
SOUTH FLORIDA WATER MANAGEMENT DIVISION
STRUCTURE SAMPLING
TOTAL MERCURY ANALYSIS
WATER SAMPLES COLLECTED NOVEMBER 3 1994

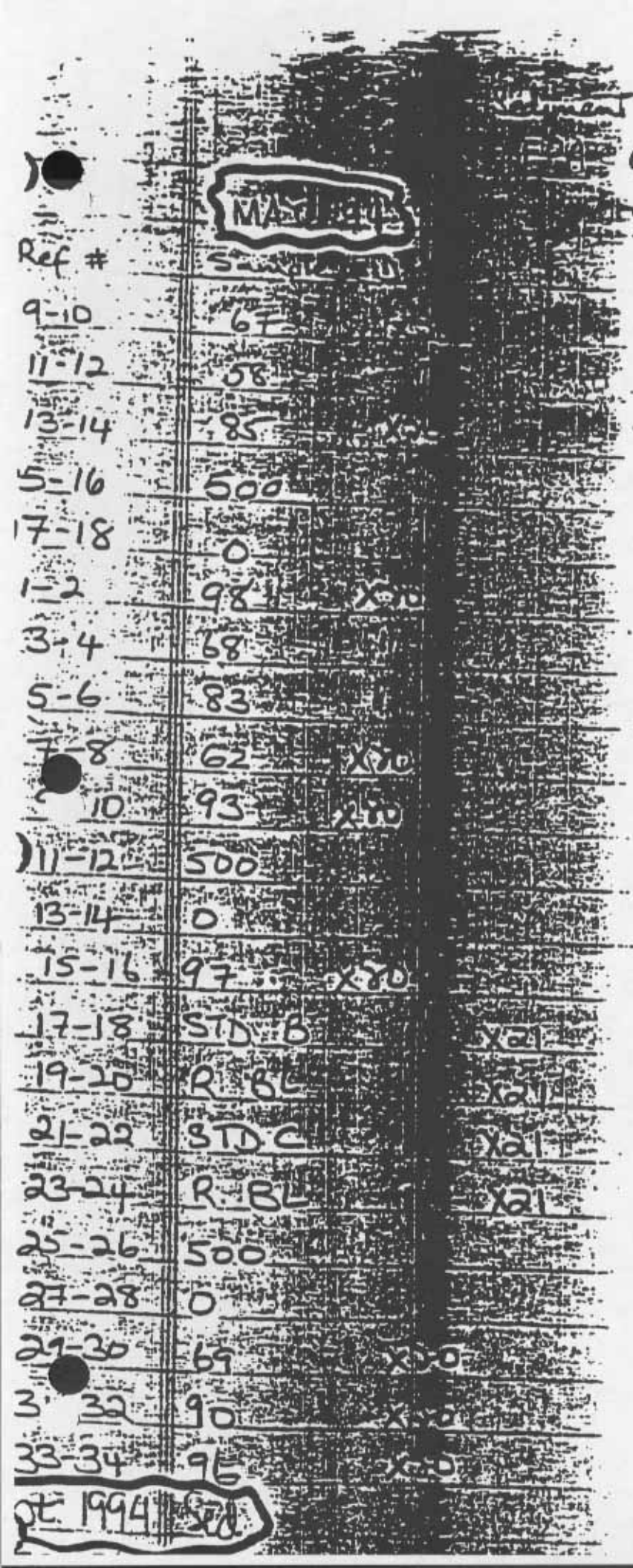
Y	X
30	143.5
30	143.1
30	141.8
30	143.1
30	141.2
30	141.2

Regression Output:

Constant	0
Std Err of Y Est	0.218825
R Squared	ERR
No. of Observations	6
Degrees of Freedom	5
X Coefficient(s)	0.210788
Std Err of Coef.	0.000628

SAMPLE ID	P.H.	Hg-CONC (PPT)	MEAN	S.D.
REAGENT BLANK	0			
A #24	5.4	1.14		
	5.3	1.12		
	5.4	1.14	1.13	0.01
S5A #24	5.3	1.12		
	5.6	1.18	1.15	0.03
S5A #24	4.0	0.84		
	4.9	1.03	0.94	0.09
S7 #43	0.0	0.00		
FIELD	0.2	0.04	0.02	0.02
BLANK				
S7 #43	1.4	0.30		
FIELD	1.3	0.27	0.28	0.01
BLANK				
S7 #43	0.3	0.06		
FIELD	0.2	0.04		
BLANK	0.7	0.15	0.08	0.05

SAMPLE ID	P.H.	Hg-CONC (PPT)	MEAN	S.D.
S8 #59	0.9	0.19		
	0.4	0.08	0.14	0.05
S8 #59	1.0	0.21		
	1.4	0.30	0.25	0.04
S8 #59	1.0	0.21		
	1.6	0.34	0.27	0.06
S151 #66	2.0	0.42		
	1.5	0.32	0.37	0.05
S151 #66	0.1	0.02		
	0.4	0.08	0.05	0.03
S151 #66	1.9	0.40		
	1.3	0.27	0.34	0.06



Sediment Samples. Volume
Canal Survey

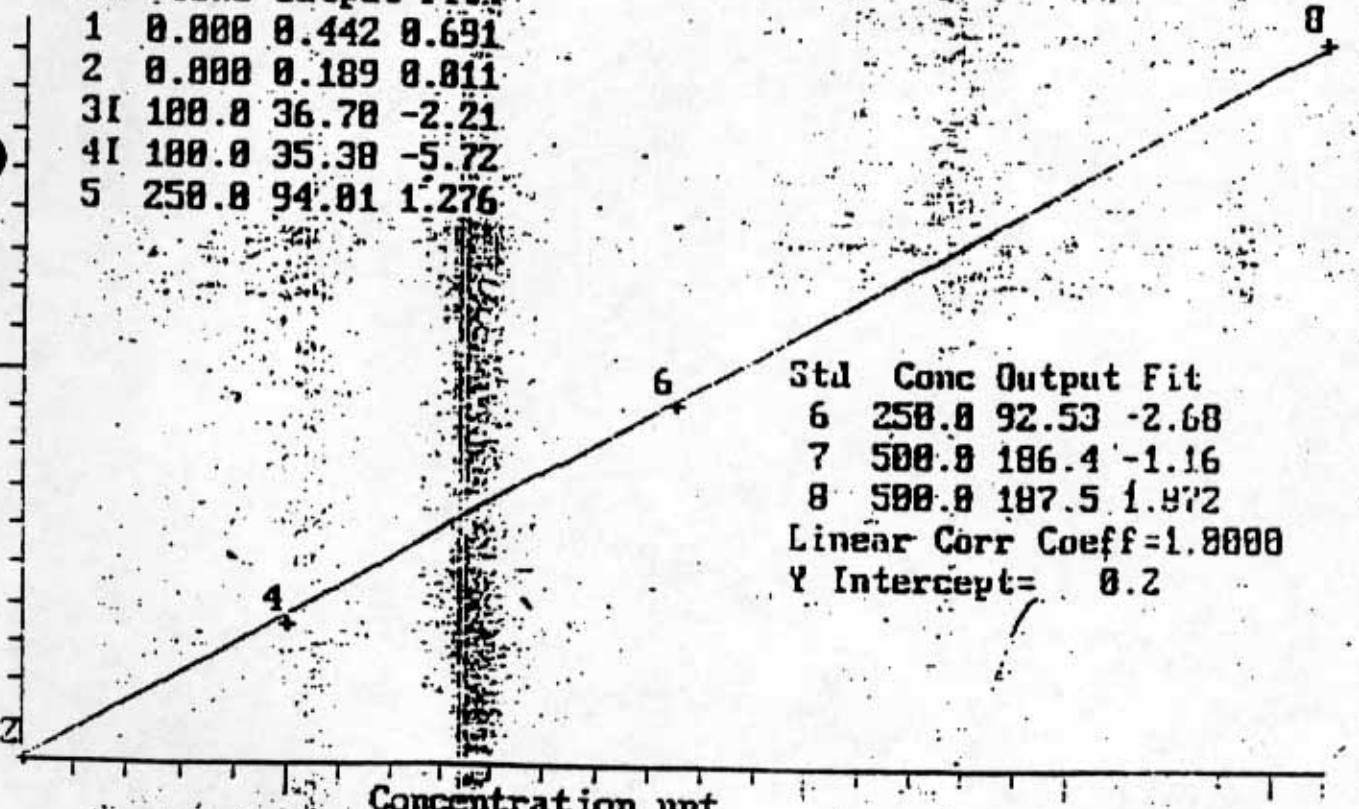
Ref #			
9-10	67		
11-12	58		
13-14	85	X10	
15-16	500		
17-18	0		
1-2	98	X10	
3-4	68		
5-6	83		
7-8	62	X10	
9-10	93	X10	
11-12	500		
13-14	0		
15-16	97	X10	
17-18	STD B	X21	
19-20	R. BL	X21	
21-22	STD C	X21	
23-24	R. BL	X21	
25-26	500		
27-28	0		
29-30	69	X10	
31-32	90	X10	
33-34	96	X10	
41-42	0	ppt	
43-44	118	X2	
45-46	133	X2	
47-48	103	X2	
49-50	STD F	X21	
51-52	R. BL	X21	
53-54	500		
55-56	0		

Oct 1994 Sed

eading Std 1 Run 1 Peak Height= 0.4 Peak Area=
Std 1 Hg 0.4 0.0ppt
ng Std 2 Run 1 Peak Height= 0.2 Peak Area=
d 2 Hg 0.2 0.0ppt
leading Std 3 Run 1 Peak Height= 36.7 Peak Area= 1670.4
Std 3 Hg 36.7 100.0ppt
leading Std 4 Run 1 Peak Height= 35.4 Peak Area= 1586.7
Std 4 Hg 35.4 100.0ppt
leading Std 5 Run 1 Peak Height= 94.0 Peak Area= 4714.
Std 5 Hg 94.0 250.0ppt
eading Std 6 Run 1 Peak Height= 92.5 Peak Area=
Std 6 Hg 92.5 250.0ppt
eading Std 7 Run 1 Peak Height= 186.5 Peak Area=
Std 7 Hg 186.9 500.0ppt
eading Std 8 Run 1 Peak Height= 187.6 Peak Area=
Std 8 Hg 187.6 500.0ppt

Hg in Water (Merlin) Fit : Least Squares Straight Line
Slope=0.37341 Term2=0.00000 Term3=0.00000 No Reslope

Std	Conc	Output	Fit
1	0.000	0.442	0.691
2	0.000	0.189	0.011
3I	100.0	36.70	-2.21
4I	100.0	35.38	-5.72
5	250.0	94.01	1.276

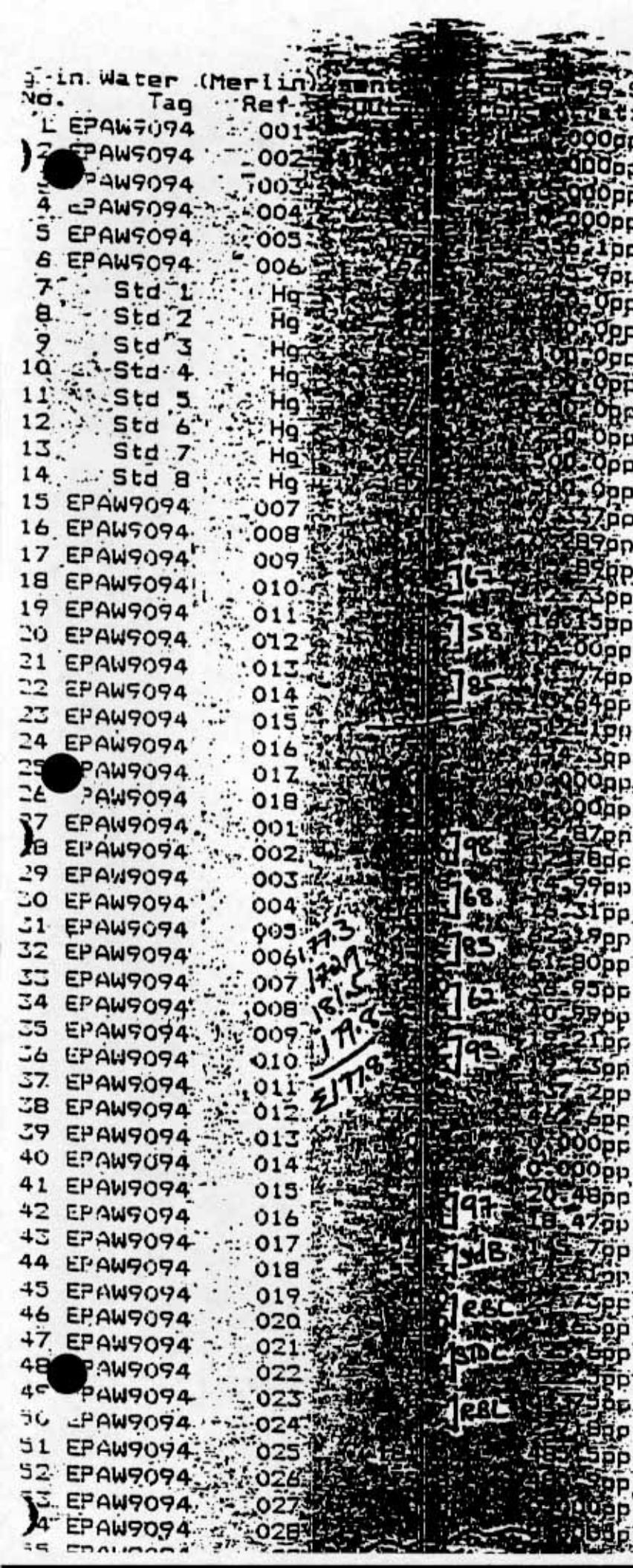


Std	Conc	Output	Fit
6	250.0	92.53	-2.68
7	500.0	186.4	-1.16
8	500.0	187.5	1.872

Linear Corr Coeff=1.0000
Y Intercept= 0.2

Printed from TouchStone 19 Sep 94

AW9094 Ref 007 Run 1 Peak Height= 0.3 Peak Area=
A 94 007 0.3 0.337ppt 1 0.0ppt
A 94 Ref 008 Run 1 Peak Height= 0.3 Peak Area=
A 094 008 0.3 0.289ppt 1 0.0ppt
AW9094 Ref 009 Run 1 Peak Height= 6.1 Peak Area=
AW9094 009 6.1 5.89ppt 1 0.0ppt
AW9094 Ref 010 Run 1 Peak Height= 4.9 Peak Area=
AW9094 010 4.9 2.73ppt 1 0.0ppt



3-in. Water (Merlin) ... 19-Sep 94

No.	Tag	Ref	...	Runs	SD	Time	Date
1	EPAW9094	001	...000ppt	1	0.[illegible]ppt	06:34	19 Sep 94
2	EPAW9094	002	...000ppt	1	0.[illegible]ppt	[illegible]:36	19 Sep 94
3	EPAW9094	003	...000ppt	1	0.[illegible]ppt	06:41	19 Sep 94
4	EPAW9094	004	...000ppt	1	0.[illegible]ppt	[illegible]:50	19 Sep 94
5	EPAW9094	005	...338.1ppt	1	0.[illegible]ppt	[illegible]:53	19 Sep 94
6	EPAW9094	006	...[illegible]ppt	1	0.[illegible]ppt	[illegible]:56	19 Sep 94
7	Std 1	Hg	...[illegible]0ppt	1	0.[illegible] 0/F	07:06	19 Sep 94
8	Std 2	Hg	...[illegible]0ppt	1	0.[illegible] 0/F	07:09	19 Sep 94
9	Std 3	Hg	...[illegible]0ppt	1	0.[illegible] 0/F	[illegible]:13	19 Sep 94
10	Std 4	Hg	...[illegible]0ppt	1	0.[illegible] 0/F	[illegible]:17	19 Sep 94
11	Std 5	Hg	...[illegible]0ppt	1	0.[illegible] 0/F	[illegible]:21	19 Sep 94
12	Std 6	Hg	...[illegible]0ppt	1	0.[illegible] 0/F	[illegible]:25	19 Sep 94
13	Std 7	Hg	...[illegible]0ppt	1	0.[illegible] 0/F	[illegible]:29	19 Sep 94
14	Std 8	Hg	...[illegible]0ppt	1	0.[illegible] 0/F	[illegible]:33	19 Sep 94
15	EPAW9094	007	...337ppt	1	0.[illegible]ppt	[illegible]:37	19 Sep 94
16	EPAW9094	008	...[illegible]89ppt	1	0.[illegible]ppt	[illegible]:41	19 Sep 94
17	EPAW9094	009	...[illegible]89ppt	1	0.[illegible]ppt	[illegible]:45	19 Sep 94
18	EPAW9094	010	...[illegible]3ppt	1	0.[illegible]ppt	[illegible]:49	19 Sep 94
19	EPAW9094	011	...[illegible]15ppt	1	0.[illegible]ppt	[illegible]:53	19 Sep 94
20	EPAW9094	012	...[illegible]00ppt	1	0.[illegible]ppt	07:57	19 Sep 94
21	EPAW9094	013	...[illegible]7ppt	1	0.[illegible]ppt	[illegible]:41	19 Sep 94
22	EPAW9094	014	...[illegible]64ppt	1	0.[illegible]ppt	[illegible]:45	19 Sep 94
23	EPAW9094	015	...[illegible]1ppt	1	0.[illegible]ppt	[illegible]:49	19 Sep 94
24	EPAW9094	016	...[illegible]3ppt	1	0.[illegible]ppt	[illegible]:53	19 Sep 94
25	EPAW9094	017	...000ppt	1	0.[illegible]ppt	[illegible]:57	19 Sep 94
26	EPAW9094	018	...000ppt	1	0.[illegible]ppt	08:01	19 Sep 94
27	EPAW9094	001	...87ppt	1	0.[illegible]ppt	08:05	19 Sep 94
28	EPAW9094	002	...[illegible]8ppt	1	0.[illegible]ppt	[illegible]:09	19 Sep 94
29	EPAW9094	003	...[illegible]9ppt	1	0.[illegible]ppt	[illegible]:13	19 Sep 94
30	EPAW9094	004	...[illegible]1ppt	1	0.[illegible]ppt	[illegible]:17	19 Sep 94
31	EPAW9094	005	...[illegible]9ppt	1	0.[illegible]ppt	[illegible]:21	19 Sep 94
32	EPAW9094	006	...[illegible]80ppt	1	0.[illegible]ppt	[illegible]:25	19 Sep 94
33	EPAW9094	007	...[illegible]95ppt	1	0.[illegible]ppt	[illegible]:29	19 Sep 94
34	EPAW9094	008	...[illegible]99ppt	1	0.[illegible]ppt	[illegible]:33	19 Sep 94
35	EPAW9094	009	...[illegible]1ppt	1	0.[illegible]ppt	[illegible]:37	19 Sep 94
36	EPAW9094	010	...[illegible]3ppt	1	0.[illegible]ppt	[illegible]:41	19 Sep 94
37	EPAW9094	011	...[illegible]2ppt	1	0.[illegible]ppt	[illegible]:45	19 Sep 94
38	EPAW9094	012	...[illegible]6ppt	1	0.[illegible]ppt	[illegible]:49	19 Sep 94
39	EPAW9094	013	...000ppt	1	0.[illegible]ppt	[illegible]:53	19 Sep 94
40	EPAW9094	014	...000ppt	1	0.[illegible]ppt	[illegible]:57	19 Sep 94
41	EPAW9094	015	...[illegible]8ppt	1	0.[illegible]ppt	[illegible]:40	19 Sep 94
42	EPAW9094	016	...[illegible]7ppt	1	0.[illegible]ppt	[illegible]:44	19 Sep 94
43	EPAW9094	017	...[illegible]7ppt	1	0.[illegible]ppt	[illegible]:48	19 Sep 94
44	EPAW9094	018	...[illegible]ppt	1	0.[illegible]ppt	[illegible]:52	19 Sep 94
45	EPAW9094	019	...[illegible]ppt	1	0.[illegible]ppt	[illegible]:56	19 Sep 94
46	EPAW9094	020	...[illegible]5ppt	1	0.[illegible]ppt	[illegible]:54	19 Sep 94
47	EPAW9094	021	...[illegible]5ppt	1	0.[illegible]ppt	[illegible]:58	19 Sep 94
48	EPAW9094	022	...[illegible]5ppt	1	0.[illegible]ppt	[illegible]:02	19 Sep 94
49	EPAW9094	023	...[illegible]5ppt	1	0.[illegible]ppt	[illegible]:06	19 Sep 94
50	EPAW9094	024	...[illegible]8ppt	1	0.[illegible]ppt	[illegible]:10	19 Sep 94
51	EPAW9094	025	...[illegible]5ppt	1	0.[illegible]ppt	[illegible]:14	19 Sep 94
52	EPAW9094	026	...[illegible]3ppt	1	0.[illegible]ppt	[illegible]:18	19 Sep 94
53	EPAW9094	027	...000ppt	1	0.[illegible]ppt	[illegible]:22	19 Sep 94
54	EPAW9094	028	...000ppt	1	0.[illegible]ppt	[illegible]:26	19 Sep 94
55	EPAW9094	[illegible]	...[illegible]ppt	1	0.[illegible]ppt	[illegible]:30	19 Sep 94

64	EPAW9094	038	90.8				
65	EPAW9094	039	88.62ppt	1			Sep 9
66	EPAW9094	040	549.2ppt	1	0.000pt	:38	19 Sep 9
67	EPAW9094	041	444.0ppt	1	0.0ppt	:40	19 Sep 9
68	EPAW9094	042	0.000ppt	1	0.000pt	:43	19 Sep 9
69	EPAW9094	043	0.000ppt	1	0.0ppt	:45	19 Sep 9
70	EPAW9094	044	92.79ppt	1	0.0ppt	:48	19 Sep 9
71	EPAW9094	045	104.0ppt	1	0.000pt	:50	19 Sep 9
72	EPAW9094	046	189.5ppt	1	0.000pt	:52	19 Sep 9
73	EPAW9094	047	188.8ppt	1	0.0ppt	9:55	19 Sep 9
74	EPAW9094	048	28.20ppt	1	0.000pt	09:57	19 Sep 9
75	EPAW9094	049	29.26ppt	1	0.000pt	9:59	19 Sep 9
76	EPAW9094	050	96.48ppt	1	0.00pt	10:02	19 Sep 9
77	EPAW9094	051	95.06ppt	1	0.000pt	10:04	19 Sep 9
78	EPAW9094	052	0.279ppt	1	0.000pt	10:07	19 Sep 9
79	EPAW9094	053	0.968ppt	1	0.0ppt	10:09	19 Sep 9
80	EPAW9094	054	421.8ppt	1	0.000pt	10:12	19 Sep 9
81	EPAW9094	055	439.9ppt	1	0.0ppt	10:15	19 Sep 9
82	EPAW9094	056	0.000ppt	1	0.000pt	10:17	19 Sep 9
			0.000ppt	1	0.000pt	10:20	19 Sep 9

177.3

172.9

~~179.8~~

166

164.5

E

EVERGLADES SEDIMENT SAMPLE
COLLECTED MAY AND SEPTEMBER 1994

Y	X
500.000	177.300
500.000	172.900
500.000	181.500
500.000	179.800

Regression Output:

Constant	0.000
Std Err of Y Est	10.507
R Squared	ERR
No. of Observations	4.000
Degrees of Freedom	3.000
X Coefficient(s)	2.810
Std Err of Coef.	0.000

SEDIMENT SAMPLES
CANAL SURVEY
SAMPLES COLLECTED MAY 1994

SAMPLE I.D.	P.H.	CORR P.H	CONC (ppt)	DILUTION FACTOR	CORR CONC.	SAMPLE WT. GRMS	ngHg/g sed (ppb)	NORMALIZED CONC (PPB)	MEAN	S.D
6/15/94										
R. BL.	0.675									
67	6.10	5.225	14.68	20.00	293.648	1.382	0.03	31.87		
	4.90	4.025	11.31	20.00	226.208	1.382	0.03	24.55	28.21	3.66
58	6.20	5.325	14.96	20.00	299.268	0.907	0.02	49.49		
	6.20	5.325	14.96	20.00	299.268	0.907	0.02	49.49	49.49	0.00
85	5.30	4.425	12.43	20.00	248.688	1.317	0.03	28.32		
	5.30	4.425	12.43	20.00	248.688	1.317	0.03	28.32	28.32	0.00
98	5.00	4.125	11.59	20.00	231.828	2.555	0.05	13.61		
	5.00	4.125	11.59	20.00	231.828	2.556	0.05	13.61	13.61	0.00
66	5.80	4.925	13.84	20.00	276.788	0.744	0.01	55.60		
	6.30	5.425	16.24	20.00	304.888	0.744	0.01	61.47	58.64	2.83
83	23.40	22.525	63.30	20.00	1265.919	0.966	0.02	196.57		
	23.30	22.425	63.01	20.00	1260.299	0.966	0.02	195.70	196.13	0.44
62	14.00	13.125	36.88	20.00	737.633	0.857	0.02	129.11		
	15.50	14.625	41.10	20.00	821.934	0.857	0.02	143.86	136.48	7.38
93	7.40	6.525	18.34	20.00	366.709	0.309	0.01	178.01		
	7.30	6.425	18.05	20.00	361.089	0.309	0.01	175.29	176.65	1.36
97	7.80	6.925	19.46	20.00	389.169	1.334	0.03	43.76		
	7.10	6.225	17.49	20.00	349.849	1.334	0.03	39.34	41.55	2.21
STD	54.60	53.725	150.97	21.00	3170.348	0.122		78.09		
	53.30	52.425	147.32	21.00	3093.634	0.122		76.20		
	47.10	46.225	129.89	21.00	2727.768	0.115		71.41		
	46.00	45.125	126.80	21.00	2662.856	0.115		69.71	73.85	3.413

Y	X
500.000	166.000
500.000	157.700
500.000	164.500

Regression Output:

Constant	0.000
Std Err of Y Est	13.587
R Squared	ERR
No. of Observations	3.000
Degrees of Freedom	2.000
X Coefficient(s)	3.071
Std Err of Coef.	0.048

SAMPLE I.D.	P.H.	CORR. P.H	CONC (ppt)	DILUTION FACTOR	CORR. CONC.	SAMPLE WT. GRM	ngHg/g sed (ppb)	NORMALIZED WT. (PPB)	MEAN	S.
69	5.70	4.825	14.82	20.00	296.351	3.681	0.07	12.08		
	5.70	4.825	14.82	20.00	296.351	3.681	0.07	12.08	12.08	(
96	19.10	18.225	55.97	20.00	1119.379.	0.551	0.01	304.73		
	18.70	17.825	54.74	20.00	1094.811	0.551	0.01	298.04	301.39	
130	11.40	10.525	32.32	20.00	646.445	1.096	0.02	88.47		
	11.30	10.425	32.02	20.00	640.303	1.096	0.02	87.63	88.05	(
144	34.10	33.225	102.03	20.00	2040.679	2.082	0.04	147.02		
	33.30	32.425	99.58	20.00	1991.543	2.082	0.04	143.48	145.25	
118	34.80	33.925	104.18	20.00	2083.673	5.837	0.12	53.55		
	39.00	38.125	117.08	20.00	2341.637	5.837	0.12	60.18	56.86	
133	71.00	70.125	215.35	20.00	4307.076	5.942	0.12	108.73		
	70.70	69.825	214.43	20.00	4288.650	5.942	0.12	108.26	108.50	(
103	10.70	9.825	30.17	20.00	603.451	1.690	0.03	53.56		
	11.10	10.225	31.40	20.00	628.019	1.690	0.03	55.74	54.65	
STD	36.200	35.325	108.48	21.000	2278.144	0.113		60.268		
	35.700	34.825	106.95	21.000	2245.898	0.113		59.415	59.84	(

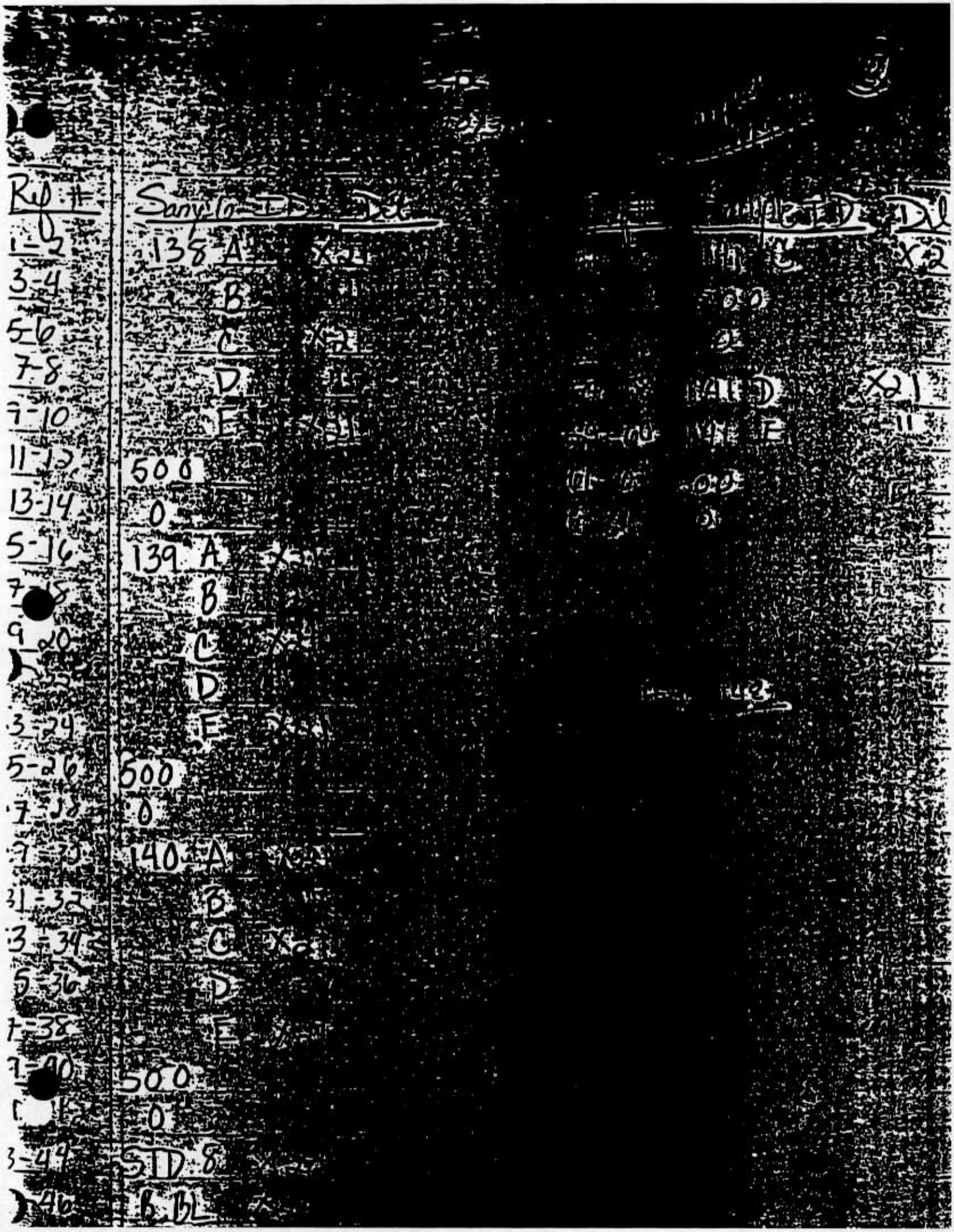
TOTAL MERCURY ANALYSIS
EPA SEDIMENT SAMPLES

CANAL SURVEY - MAY 1994

SAMPLE ID	MEAN	S.D
58	49.49	0.00
62	136.48	7.34
67	28.21	3.66
68	58.64	2.83
69	12.08	0.00
83	196.13	0.44
85	28.32	0.00
93	176.65	1.36
96	301.39	3.34
97	41.55	2.21
98	13.61	0.00

CANAL SURVEY - SEPTEMBER 1994

SAMPLE ID	MEAN	S.D
103	54.65	1.09
118	56.86	3.31
130	88.05	0.42
133	108.50	0.23
144	145.25	1.77
STD	69.18	7.17



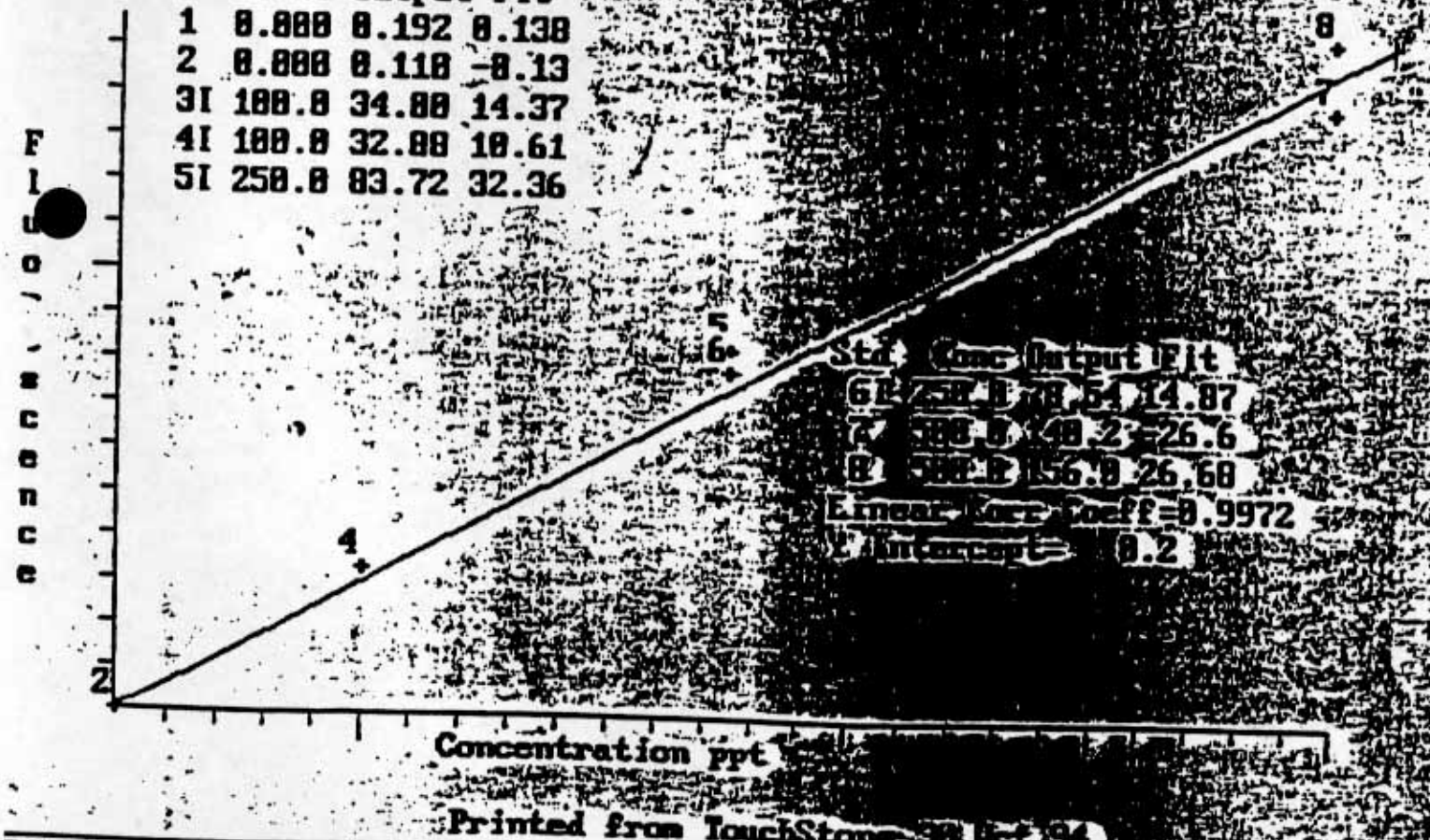
Gambusia Fish Samples
EPA Canal Survey
Collected May 1994

Number	Date	Total Length (mm)	Weight (g)	Sex
51 A	5/12	30	0.212	M
B		23	0.146	M
C		24	0.101	M
D		25	0.136	M
E		23	0.087	M
52 A	5/12	33	0.357	F
B		31	0.314	F
C		26	0.178	F
D		28	0.229	F
E		31.5	0.325	F
53 A	5/12	24	0.141	F
B		26	0.172	M
C		25	0.156	F
D		27.5	0.202	M
E		28	0.218	F
54 A	5/12	22	0.078	M
B		25.5	0.138	M
C		30	0.27	M
D		27	0.201	M
E		29	0.240	M

```
Reading Std 1 Run   1 Peak Height=
   Std 1      Hg      0.2
Reading Std 2 Run   1 Peak Height=
   Std 2      Hg      0.1
Reading Std 3 Run   1 Peak Height=   34.0
   Std 3      Hg     34.0      100.0ppt
Reading Std 4 Run   1 Peak Height=   32.9
   Std 4      Hg     32.9      100.0ppt
Reading Std 5 Run   1 Peak Height=   83.7
   Std 5      Hg     83.7      250.0ppt
Reading Std 6 Run   1 Peak Height=   78.5
   Std 6      Hg     78.5      250.0ppt
Reading Std 7 Run   1 Peak Height=  140.2
   Std 7      Hg    140.2      500.0ppt
Reading Std 8 Run   1 Peak Height=  156.0
   Std 8      Hg    156.0      500.0ppt
```

Hg in Water (Merlin) Fit : Least Squares Straight Line
Slope=0.29597 Term2=0.00000 Term3=0.00000 No Reslope

Std	Conc	Output	Fit
1	0.000	0.192	0.138
2	0.000	0.110	-0.13
3I	100.0	34.00	14.37
4I	100.0	32.00	10.61
5I	250.0	83.72	32.36



Std	Conc	Output	Fit
6I	250.0	78.54	14.87
7	500.0	140.2	26.6
8	500.0	156.0	26.68

Linear Corr Coeff=0.9972
Intercept= 0.2

Printed from TouchStone 28 Oct 94

```
PAF0994 Ref 007 Run   1 Peak Height=
PAF0994     007       0.4      0.923ppt
PAF0994 Ref 008 Run   1 Peak Height=
PAF0994     008       0.0      0.000ppt
```

No.	Tag	Ref	Output	Concentration	Date
1	EPAF0994	001	0.0	0.000ppt	Oct 94
2	EPAF0994	002	0.8	0.481ppt	Oct 94
3	EPAF0994	003	161.3	453.2ppt	30 Oct 94
4	EPAF0994	004	154.7	434.3ppt	30 Oct 94
5	EPAF0994	005	0.0	0.000ppt	30 Oct 94
6	EPAF0994	006	0.0	0.000ppt	30 Oct 94
7	Std 1	Hg	0.2	0.0ppt	30 Oct 94
8	Std 2	Hg	0.1	0.0ppt	30 Oct 94
9	Std 3	Hg	34.0	100.0ppt	30 Oct 94
10	Std 4	Hg	32.9	100.0ppt	30 Oct 94
11	Std 5	Hg	83.7	250.0ppt	30 Oct 94
12	Std 6	Hg	78.5	250.0ppt	30 Oct 94
13	Std 7	Hg	140.2	500.0ppt	30 Oct 94
14	Std 8	Hg	156.0	500.0ppt	30 Oct 94
15	EPAF0994	007	0.4	0.923ppt	30 Oct 94
16	EPAF0994	008	0.0	0.000ppt	30 Oct 94
17	EPAF0994	001	8.1	26.74ppt	30 Oct 94
18	EPAF0994	002	7.5	24.81ppt	30 Oct 94
19	EPAF0994	003	13.8	46.27ppt	30 Oct 94
20	EPAF0994	004	13.4	44.85ppt	30 Oct 94
21	EPAF0994	005	9.2	30.65ppt	30 Oct 94
22	EPAF0994	006	7.2	23.66ppt	30 Oct 94
23	EPAF0994	007	9.1	30.30ppt	30 Oct 94
24	EPAF0994	008	9.6	31.75ppt	30 Oct 94
25	EPAF0994	009	6.6	21.83ppt	30 Oct 94
26	EPAF0994	010	7.3	24.12ppt	30 Oct 94
27	EPAF0994	011	148.2	500.1ppt	30 Oct 94
28	EPAF0994	012	152.5	514.6ppt	30 Oct 94
29	EPAF0994	013	0.0	0.000ppt	30 Oct 94
30	EPAF0994	014	0.0	0.000ppt	30 Oct 94
31	EPAF0994	015	62.6	211.0ppt	30 Oct 94
32	EPAF0994	016	60.7	204.4ppt	30 Oct 94
33	EPAF0994	017	17.6	59.06ppt	30 Oct 94
34	EPAF0994	018	19.8	66.21ppt	30 Oct 94
35	EPAF0994	019	35.4	118.9ppt	30 Oct 94
36	EPAF0994	020	35.5	119.4ppt	30 Oct 94
37	EPAF0994	021	28.4	95.41ppt	30 Oct 94
38	EPAF0994	022	29.6	99.63ppt	30 Oct 94
39	EPAF0994	023	101.5	342.4ppt	30 Oct 94
40	EPAF0994	024	101.7	343.0ppt	30 Oct 94
41	EPAF0994	025	125.9	424.9ppt	30 Oct 94
42	EPAF0994	026	135.1	455.9ppt	30 Oct 94
43	EPAF0994	027	0.0	0.000ppt	30 Oct 94
44	EPAF0994	028	0.0	0.000ppt	30 Oct 94
45	EPAF0994	029	23.1	77.66ppt	30 Oct 94
46	EPAF0994	030	22.0	73.75ppt	30 Oct 94
47	EPAF0994	031	32.0	107.7ppt	30 Oct 94
48	EPAF0994	032	31.7	106.6ppt	30 Oct 94
49	EPAF0994	033	20.3	68.24ppt	30 Oct 94
50	EPAF0994	034	20.4	68.49ppt	30 Oct 94
51	EPAF0994	035	45.7	154.0ppt	30 Oct 94
52	EPAF0994	036	48.5	163.4ppt	30 Oct 94
	EPAF0994	037	34.8	117.0ppt	30 Oct 94
	EPAF0994	038	31.9	107.2ppt	30 Oct 94

63	EPAF0994	047					
64	EPAF0994	048					
65	EPAF0994	049					
66	EPAF0994	050					
67	EPAF0994	051	137.5	126.0ppt			
	EPAF0994	052	36.1	121.4ppt			
	EPAF0994	053	149.8	505.6ppt			
70	EPAF0994	054	150.9	509.1ppt			
71	EPAF0994	055	0.0	0.000ppt			
72	EPAF0994	056	0.1	0.000ppt			
73	EPAF0994	057	28.4	95.56ppt			
74	EPAF0994	058	27.0	90.80ppt			
75	EPAF0994	059	20.6	69.12ppt			
76	EPAF0994	060	22.6	75.77ppt			
77	EPAF0994	061	144.3	487.0ppt			
78	EPAF0994	062	146.9	495.8ppt			
79	EPAF0994	063	0.0	0.000ppt			
80	EPAF0994	064	0.0	0.000ppt			

[illegible] Mercury:

spiked amount(pg): 1000   Conc of standard(pg/ul): 5.00

standard	Peak Area
1	27.28
2	28.17
3	38.78
4	34.77
5	34.58
6	32.92
avg.	32.58
std.	3.33

Sample No.	W.S.W (g)	D.S.W (g)	SPIKED (ng/g)	1st.V. (ml)	2nd.V. (ml)	P.A xE+5	CONC (pg/ul)	CONC (ng/g)	avg	std	RECOV (%)	Avg.Rec. (%)	REC.CON (ng/g)	std
A	B	C	D	E	F	G	H	I	J	K	L	M	N	O
900a	4.347	0.060		4	0.8	0	0.00	0.00						
b	4.328	0.059		4	0.8	0	0.00	0.00	0.00	0.00			0.00	0.00
c	4.389	0.060	16.69	4	0.8	21.78	3.34	17.42			104.37			
d	4.29	0.059	17.00	4	0.8	19.19	2.95	15.65			92.04	98.21		
980a	4.384	0.347		4	0.8	6.78	1.04	0.94						
b	4.191	0.334		3.7	0.8	5.46	0.84	0.85	0.89	0.04			1.12	0.05
c	4.219	0.338	2.98	4	0.8	23.38	3.59	3.34			82.17			
d	4.68	0.371	2.70	4	0.8	23.08	3.54	2.99			77.80	79.98		
394a	4.22	0.211		3.6	0.8	0	0.00	0.00						
b	4.651	0.233		4	0.8	0	0.00	0.00	0.00	0.00			0.00	0.00
c	4.352	0.218	4.59	4	0.8	19.87	3.05	4.37			95.30			
d	4.419	0.221	4.52	4	0.8	18.42	2.83	3.99			88.35	91.83		
400a	4.58	0.084		4	0.8	0	0.00	0.00						
b	4.291	0.079		4	0.8	0	0.00	0.00	0.00	0.00			0.00	0.00
c	4.526	0.083	11.99	4	0.6	7.57	1.16	5.80			48.41			
d	4.45	0.082	12.19	4	0.8	14.64	2.25	8.56			70.22	70.22		
415a	4.237	0.157		4	0.8	0	0.00	0.00						
b	4.21	0.156		4	0.8	bpk	0.00	0.00	0.00	0.00			0.00	0.00
c	4.431	0.164	6.10	4	0.8	17.62	2.70	5.15			84.51			
d	4.369	0.162	6.18	4	0.8	9.88	1.52	2.93			47.39	84.51		
416a	4.309	0.127		4	0.8	0	0.00	0.00						
b	4.468	0.132		4	0.8	0	0.00	0.00	0.00	0.00			0.00	0.00
c	4.701	0.139	7.21	4	0.9	17.68	2.71	6.11			84.80			
d	4.522	0.133	7.48	4	0.8	17.45	2.68	6.27			83.70	84.25		
419a	4.467	0.087		4	0.8	0	0.00	0.00						
b	4.424	0.086		4	0.8	0	0.00	0.00	0.00				0.00	0.00
c	4.343	0.085	15.32	4	0.8	18.27	2.80	13.42			87.53			
d	4.938	0.074	13.47	4	0.8	18.21	2.79	11.77			87.34	87.49		
423a	4.389	0.198		4	0.8	0	0.00	0.00						
b	4.359	0.197		4	0.8	0	0.00	0.00	0.00	0.00			0.00	0.00
c	4.426	0.200	5.01	4	0.8	20.91	3.21	5.02			100.29			
d	4.696	0.212	4.72	4	0.8	19.61	3.01	4.44			94.06	97.17		
425a	4.302	0.365		3.8	0.8	0	0.00	0.00						
b	4.327	0.387		4	0.8	0	0.00	0.00	0.00	0.00			0.00	0.00
c	4.389	0.372	2.68	4	0.8	19.84	3.05	2.56			95.16			
d	4.133	0.351	2.85	4	0.8	17.17	2.64	2.35			82.35	88.76		
429a	4.543	0.274		4	0.8	0	0.00	0.00						
b	4.399	0.265		4	0.8	bpk	0.00	0.00	0.00	0.00			0.00	0.00
c	4.27	0.257	3.89	3.8	0.8	18.33	2.97	3.80			97.59			
d	4.201	0.253	3.95	4	0.8	15	2.30	2.84			71.95	84.77		

Methyl Mercury

Spiked amount (pg) 1000 Conc of standard (pg/ul) 5.00

(last step volume ul)
200 CH2Cl2

standard	Peak Area
1	28.42
2	28.24
3	32.35
4	30.21
5	33.00
6	36.01
avg.	31.21
std.	2.96

Samp.No.	W S W (g)	D.S.W (g)	SPIKED (ng/g)	1st V. (ml)	2st V. (ml)	P A xE+5	CONC (pg/ul)	CONC (ng/g)	avg	std	RECOV. (%)	Avg.Rec. (%)	REC.CON (ng/g)	std
A	B	C	D	E	F	G	H	I	J	K	L	M	N	O
900a	4.347	0.080		4	0.8	3.01	0.48	2.53						
b	4.328	0.059		4	0.8	2.54	0.41	2.14	2.34	0.19			2.85	0.23
c	4.369	0.060	16.69	4	0.8	20.58	3.29	17.18			98.95			
d	4.29	0.059	17.00	4	0.8	17.74	2.84	15.10			75.09	82.02		
990a	4.364	0.347		4	0.8	4.59	0.74	0.86						
b	4.191	0.334		3.7	0.8	2.52	0.40	0.41	0.54	0.13			0.68	0.16
c	4.219	0.336	2.98	4	0.8	19.22	3.08	2.97			78.26			
d	4.68	0.371	2.70	4	0.8	19.58	3.13	2.64			78.09	78.17		
394a	4.22	0.211		3.6	0.8	5.25	0.84	1.38						
b	4.651	0.233		4	0.8	6.74	1.08	1.45	1.41	0.03			1.33	0.03
c	4.352	0.218	4.59	4	0.8	28.32	4.54	6.50			110.95			
d	4.419	0.221	4.52	4	0.8	26.6	4.26	4.02			101.27	106.41		
400a	4.56	0.084		4	0.8	2.22	0.36	1.32						
b	4.291	0.079		4	0.8	1.97	0.32	1.25	1.28	0.04			1.54	0.05
c	4.525	0.083	11.99	4	0.8	14.41	2.31	11.53			85.49			
d	4.45	0.082	12.19	4	0.8	18.31	2.93	11.18			81.14	83.31		
413a	4.237	0.137		4	0.8	2.84	0.46	0.91						
b	4.21	0.156		4	0.8	bpk	0.00	0.00	0.91				1.18	
c	4.431	0.164	6.10	4	0.8	18.48	2.96	5.84			77.56			
d	4.369	0.162	6.19	4	0.8	18.16	2.91	5.62			78.27	78.91		
416a	4.309	0.127		4	0.8	0	0.00	0.00						
b	4.468	0.132		4	0.8	0	0.00	0.00	0.00	0.00			0.00	0.00
c	4.701	0.139	7.21	4	0.8	17.65	2.83	6.37			88.38			
d	4.522	0.133	7.49	4	0.8	19.43	3.11	7.29			97.29	92.83		
419a	4.467	0.067		4	0.8	0	0.00	0.00						
b	4.424	0.066		4	0.8	0	0.00	0.00	0.00				0.00	0.00
c	4.343	0.065	15.32	4	0.8	16.25	2.60	12.47			81.37			
d	4.938	0.074	13.47	4	0.8	16.83	2.70	11.35			84.27	82.82		
423a	4.389	0.198		4	0.8	2.7	0.43	0.68						
b	4.359	0.197		4	0.8	4.22	0.68	1.07	0.88	0.20			0.91	0.20
c	4.428	0.200	5.01	4	0.8	22.61	3.62	5.67			95.67			
d	4.696	0.212	4.72	4	0.8	22.98	3.68	5.43			98.45	96.06		
425a	4.302	0.366		3.9	0.8	0	0.00	0.00						
b	4.327	0.367		4	0.8	0	0.00	0.00	0.00	0.00			0.00	0.00
c	4.389	0.372	2.68	4	0.8	20.06	3.21	2.70			100.44			
d	4.133	0.351	2.85	4	0.8	21.53	3.45	3.07			107.81	104.12		
429a	4.543	0.274		4	0.8	5.7	0.91	1.04						
b	4.399	0.265		4	0.8	bpk	0.00	0.00	1.04				1.32	
c	4.27	0.257	3.89	3.8	0.8	22.03	3.53	4.52			99.29			
d	4.201	0.253	3.95	4	0.8	19.08	3.05	3.77			69.04	79.17		

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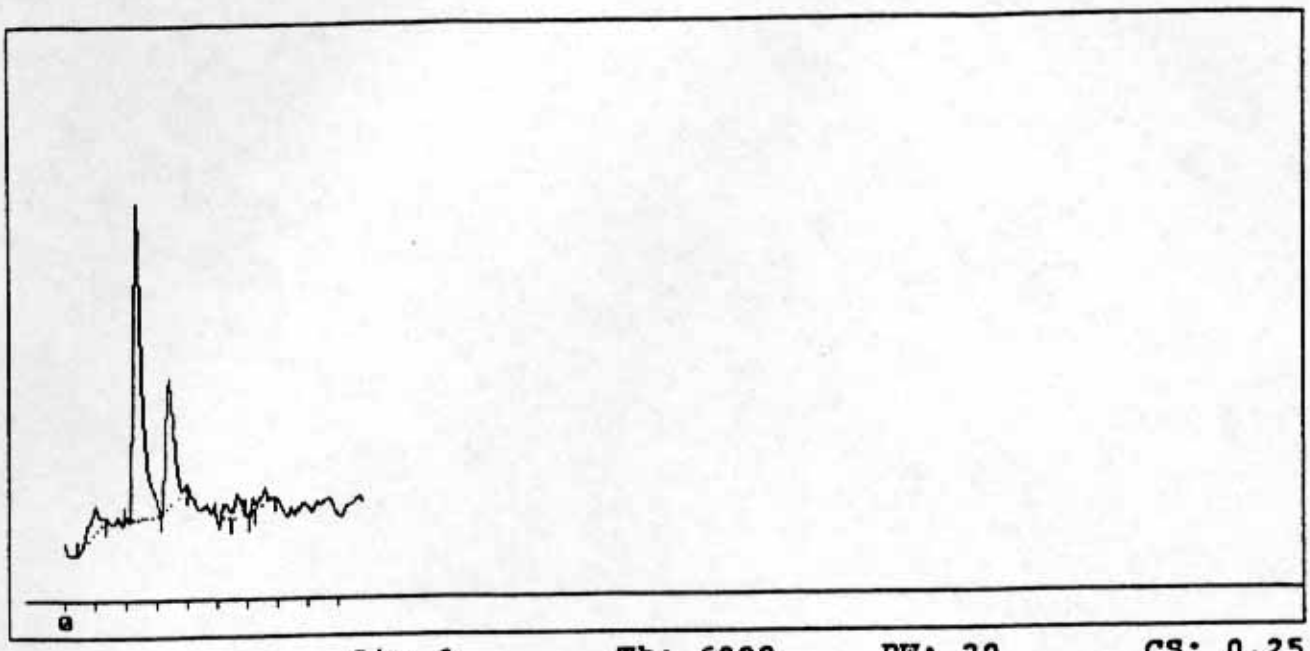
Date 1/28/1997



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Peak Report

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Data File: 28JAN16

Method File:

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3	00:03:20	1.665E+06	9.162E+04		
4	00:05:11	1.311E+05	1.252E+04		
5	00:05:38	1.829E+05	1.879E+04		
6	00:06:10	8.433E+04	1.120E+04		
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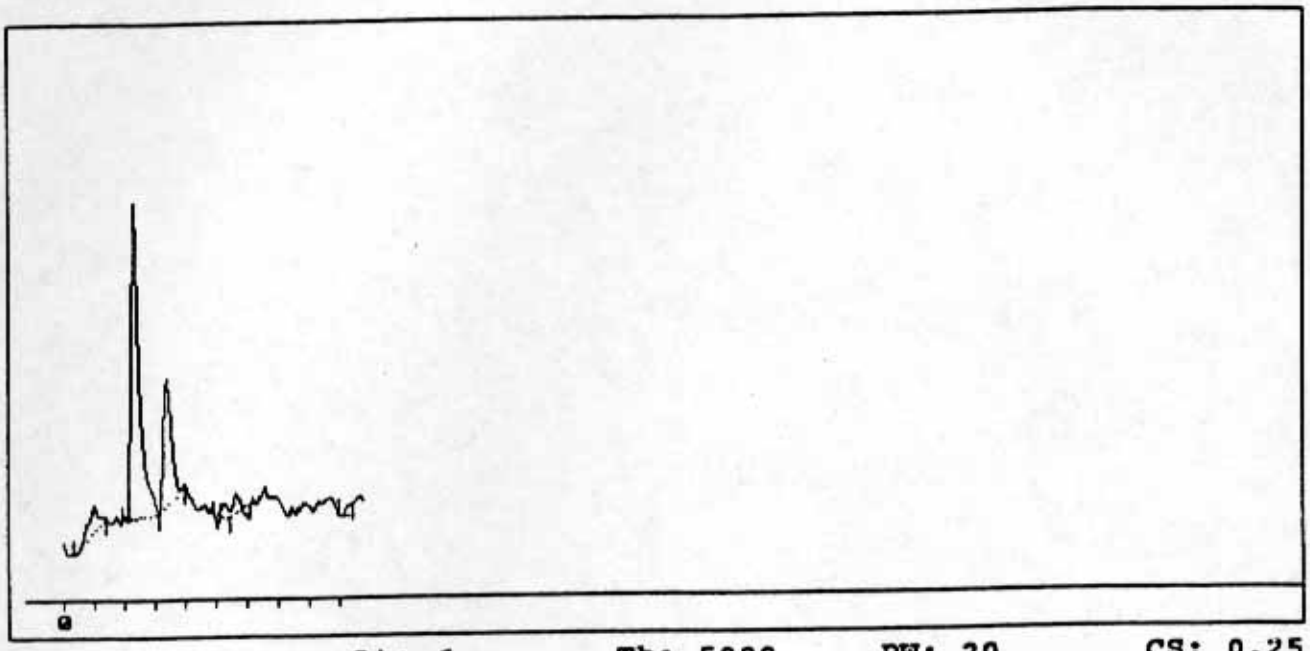
Date 1/28/1997



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Peak Report

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Method File:

Ch 1 Detector:

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1	00:00:56	4.755E+05	2.228E+04		
2	00:02:13	4.067E+06	2.228E+05		
3	00:03:20	1.665E+06	9.162E+04		
4	00:05:11	1.278E+05	1.227E+04		
5	00:05:38	4.531E+04	1.680E+04		
6	00:09:24	1.637E+05	9.975E+03		

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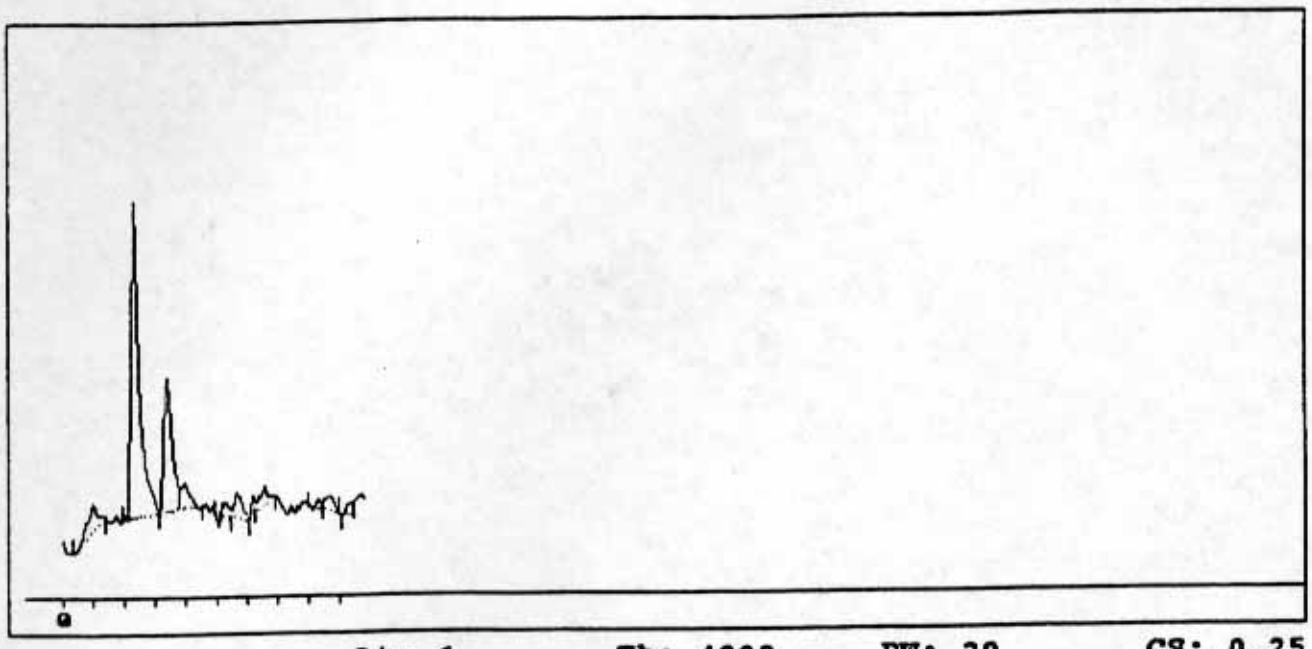
Data File : 28JAN16
Method File :

Date 1/28/1997



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Peak Report

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Data File: 28JAN16

Method File:

Ch 1 Detector:

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2	00:02:13	4.067E+06	2.228E+05		
3	00:03:20	1.833E+06	9.514E+04		
4	00:03:59	3.034E+05	1.884E+04		
5	00:05:11	1.142E+05	1.227E+04		
6	00:05:38	3.317E+05	2.026E+04		
7	00:06:10	1.241E+05	1.695E+04		
8	00:06:35	2.323E+05	1.505E+04		
9	00:08:17	8.190E+04	9.030E+03		
10	00:08:43	1.994E+05	1.244E+04		
11	00:09:24	1.612E+05	1.013E+04		

Analysis of Organomercury Compounds in Sediments by Capillary GC with Atomic Fluorescence Detection

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Key Words:

Organomercury compounds
Capillary GC
Atomic fluorescence detection

Summary

Analysis of methyl- and ethylmercury (MM and EM) halides in biological and environmental samples is generally performed by gas chromatography with electron capture detection. Tedious sample work-up protocols and poor chromatographic response (using packed columns) have, however, shown the need for the development of new methods in this field.

This paper reports a sensitive method, free from these deficiencies, for the determination of methyl- and ethylmercury. The organomercury compounds (MM and EM) are first released from the sample matrix, by the combined action of acidic potassium bromide and cupric ions, and then extracted into dichloromethane. The initial extracts are subjected to thiosulfate clean-up and the organomercury species are isolated as their chloride derivatives by addition of cupric chloride, and subsequent extraction into a small volume of organic solvent. Capillary GC coupled with atomic fluorescence detection provided excellent separation efficiencies for methyl- and ethylmercury and proved to be a very selective and sensitive technique. The absolute detection limit for both MM and EM was found to be 0.2 pg.

1 Introduction

Mercury is a widely distributed environmental pollutant. Its organic compounds, particularly methylmercury, are far more toxic than elemental mercury or its inorganic salts [1,2]. Such widespread hazard and toxicological concern have stimulated a great demand for reliable, precise, and sensitive methods for the determination of organomercury (MM and EM) compounds in water, sediments, fish, and other biological samples.

The analytical technique most commonly used for determination of organomercury compounds is gas chromatography using electron capture detection (GC-ECD) [1–11], with or without prior derivatization of the mercury compounds. Other techniques involve derivatization by butylation [12,13], aqueous phase ethylation [14–16], or hydridization [17,18], coupled with microwave-induced plasma (MIP) [10,12,13,19–22], atomic absorption [14,16,23–27], or atomic fluorescence [15,28,29] detection. Most of the chromatographic methods reported have used packed columns [5,7,8,10,11,16]. A wide variety of stationary phases have been recommended for use in organic mercury analysis, but many of these have shown one or more deficiencies [1,7,11–13]:

(a) moderate to severe peak tailing;

(b) poor column efficiency which often leads to problems with interferences;

(c) poor and often variable response to MM and EM; and

(d) reduced peak areas (or heights) of MM and EM from sediment or fish extracts, despite good responses from standards prior to injection of sample extracts.

O'Reilly [11] investigated this undesirable behavior in detail and proposed 'passivation' of the packed column, prior to analytical measurements, by use of a concentrated solution of mercury(II) chloride in benzene (later replaced by toluene) [12]. The passivation procedure must, unfortunately, be repeated frequently as the benefits of the conditioning gradually diminish, and the onset of ion-exchange and adsorption processes prevents satisfactory elution of the mercury species [1,11,13]. The injection of large amounts of mercury(II) chloride has several other drawbacks [1]:

(a) gradual and irreversible contamination of electron capture detectors;

(b) rapid deterioration of the performance of the column; and

(c) long periods during which no analysis can be performed.

This report describes a procedure for the analysis of organic mercury which successfully addresses the above chromatographic drawbacks. The method, a modification of the procedure developed by *Cappon* and *Smith* [5], employs a non-polar capillary column for higher separation efficiency. The initial extracts are subjected to sodium thiosulfate clean-up prior to GC analysis with a highly sensitive atomic fluorescence detector system.

2 Experimental

2.1 Sample Preparation

Sediment samples (from a freshwater marsh, Everglades National Park, Florida, USA) were collected in non-sterile, wide-mouth specimen cups (125 mL, Fisher Scientific) and those not analyzed immediately were frozen at –20 °C to preserve the samples' chemical integrity. Each sample was homogenized with distilled water (30–50 mL) for 3 min at high speed, using a blender (Osterizer), to a uniform consistency.

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[2] Department of Chemistry.
[3] Drinking Water Research Center.
[4] Department of Biological Sciences.

2.2 Procedure

A portion (1.0–5.0 g) of the homogenized sample was placed in a 20 mL borosilicate glass scintillation vial (Kimble #74511). Distilled water (5 mL), followed by copper sulfate (1.0 M; 3.0 mL) and acidic potassium bromide solution (3.0 mL) were added and the mixture was shaken for 1 h at 330 rpm (Gyratory Shaker Model G2). Dichloromethane (5 mL) was added and the mixture shaken for 12 h at 330 rpm and then centrifuged for 10 min at 5000 g in a Sorvall Model RC-5 refrigerated centrifuge (Dupont). An exactly known volume of the dichloromethane layer (3.5–4.0 mL) was transferred to a 7.0 mL borosilicate glass scintillation vial (Fisher Scientific #0333726) and sodium thiosulfate solution (0.01 M; 1.0 mL) was added. The mixture was shaken for 20 min at 330 rpm and centrifuged at high speed in a IEC clinical centrifuge. The aqueous layer (0.9 mL) was placed in a 2.0 mL microcentrifuge tube (Fisherbrand, Fisher Scientific), and copper chloride (0.5 M; 0.3 mL) and dichloromethane (0.3 mL) were added. The contents were mixed for 1 min on a Vortex Genie mixer and centrifuged for 2 min at high speed in a Hermle centrifuge. The dichloromethane was transferred to a 2.0 mL glass sampling vial containing a few crystals of anhydrous sodium sulfate and submitted for GC analysis using 5.0 µL injections. Samples spiked with known concentrations of methyl- and ethylmercury were extracted to evaluate the recovery factor used for quantification.

2.3 Analysis and Instrumentation

A schematic diagram of the GC-AFS system used in this work is presented in **Figure 1**. Chromatography was performed with a Hewlett-Packard (Model 5890 Series II) gas chromatograph coupled with an HP (Model 7673) automatic sampler and fitted with a 15 m × 0.53 mm i.d. (Megabore) fused silica column coated with a 1 µm film of the non-polar bonded phase DB-1 (J&W Scientific). Splitless injection was employed; the injector temperature was 250 °C. The column oven temperature was held at 40 °C for 1 min after injection, programmed at 60 °/min to 140 °C, which was held for 3 min, then programmed at 50 °/min to 200 °C which was held for 10 min. The column and make-up flows were 4.0 and 60 mL/min, respectively.

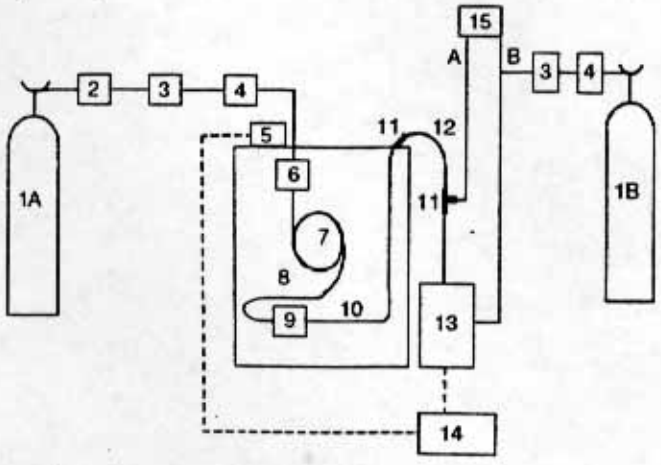

Figure 1
Gas chromatographic – atomic fluorescence spectrometric system: 1A, helium; 1B, argon; 2, oxygen trap; 3, mercury trap; 4, moisture trap; 5, automatic sampler; 6, injector; 7, column; 8, press-fit union; 9, pyrolyzer; 10, deactivated 0.53 mm i.d. fused silica; 11, PTFE unions; 12, 0.5 mm i.d. PTFE transfer line; 13, atomic fluorescence detector; 14, E-Lab chromatographic control and data acquisition system; 15, mass flow controller Channel A make-up, Channel B sheath gas.

The column eluate was led through a pyrolyzer (P.S. Analytical Ltd., UK) positioned inside the GC oven, *via* a 65 cm long deactivated fused silica tubing (0.53 mm i.d., J&W Scientific), which was connected to the column with a glass 'press-fit' union (J&W Scientific); the pyrolyzer temperature was 800 °C. The mercury atoms formed in the pyrolysis unit were transferred, *via* 0.5 mm i.d. tubing (FEP, Alltech Associates), from the outlet of the deactivated fused silica tubing to the fluorescence detector. The transfer line was passed through a small hole in the top of the GC oven to the detector and the connections were made with PTFE unions.

A Merlin Mercury Fluorescence Detector System (AFS), Model 10.023, (P.S. Analytical) was used. In operation the sample was fed to the detector as a gas which was channelled through a chimney past a light source and a photomultiplier tube at right angles to each other. The optical assembly was sheathed in a flow of argon (300 mL/min). Efficient isolation of the required excitation and emission wavelengths was achieved by means of a specific high intensity mercury lamp source (Cathodeon, Cambridge, UK) and a fixed 254 nm filter.

The integrate time was 0.25 s, the calibration range 1000 (most sensitive), the fine gain 10 (maximum), the recorder output voltage 1 V, and the damping switch was on (for signal smoothing). A real-time chromatographic control and data acquisition system (E-Lab, Version 4.10 R, OMS Tech) was interfaced with the GC and AFS detector system. In this work, the detection limit was defined as the amount of mercury giving a peak area equal to three times the standard deviation of the background signal.

2.4 Gases

All gases were supplied by Liquid Carbonic Speciality Gases and were of zero grade quality. The carrier gas was helium (99.995 %), purified by passage through, first, an oxygen trap, then a mercury trap (gold-activated carbon) and a moisture trap. Argon (99.998 %), employed as make-up gas and sheath gas for the AFS, was also passed through both a moisture trap and a mercury trap before use. Its flow was regulated by a mass flow controller (Omega) equipped with two channels, channel A (make-up flow) and channel B (sheath gas flow, Figure 1). The mercury trap was used as a means of avoiding deterioration of the detector's signal-to-noise ratio as a result of possible contamination of the carrier and sheath gases.

2.5 Reagents

Distilled, deionized water produced by a Barnstead B-Pure system was used in all solutions. Certified ACS grade potassium bromide, copper(II) sulfate, copper(II) chloride, and sodium thiosulfate (Fisher Scientific) were used throughout this work. The acidic potassium bromide solution was prepared by dissolving 180 g in 200 mL water. Trace metal grade concentrated sulfuric acid (50 mL, Fisher Scientific) was added to 100 mL of water. After cooling to room temperature the solutions were mixed and made up to 1 L with water. Copper sulfate (1.0 M), copper chloride (0.5 M), and sodium thiosulfate (0.01 M) solutions were prepared by dissolving appropriate amounts of the salts in water. All solutions were extracted with dichloromethane prior to use.

2.6 Standards

All mercury standards were purchased from Ultra Scientific. Stock standard solutions of dimethylmercury, methyl-, and ethylmercury chloride (DMM, MMC, and EMC) were prepared by dissolving appropriate amounts of the standards in optima grade methanol (Fisher Scientific). These solutions were stored in dark brown bottles at –10 °C and diluted with dichloromethane to give working

standards of the desired concentrations when required. Under such condition, these stock solutions were stable for several months.

3 Results and Discussion

3.1 Chromatography of Organomercury Compounds

In the analysis of organomercury compounds, the derivatization techniques involved can be time-consuming and the derivatized products may not necessarily reflect the actual concentration of the various organic mercury species native to the sample. In the aqueous phase ethylation technique, both inorganic mercury (Hg^{2+}) and EM are derivatized to diethylmercury and thus the quantification of inorganic mercury and EM inherent in the sample can become difficult. Another disadvantage is the use of the ECD for detection. Evidence for the partial on-column decomposition of MMC has been reported [1,11]. This would result in the loss of the electron-capturing moiety which would render the ECD an unsuitable detector. In addition, mercuric chloride conditioning of the GC column is associated with severe drawbacks [1] and this 'passivation' technique is a major limitation of the analytical method. As evidenced by these disadvantages, there is need for more straightforward methods in the analysis of organomercury compounds.

The objectives of this work were to develop a method for the direct solvent extraction of organomercury compounds from sediments (with dichloromethane) followed by GC analysis. This, however, resulted in irreversible GC column problems if no further sample clean-up steps were performed. A typical chromatogram of the pure mixed mercury standards (DMM, MMC, and EMC) is shown in **Figure 2A**. Clearly, the chromatogram is indicative of excellent chromatographic behavior.

The first injection of a sediment extract, (*i.e.* from sediment acidified with acidic potassium bromide and extracted with dichloromethane after addition of copper sulfate), also resulted in excellent chromatographic behavior for MM and EM present in the sample. A consecutive injection of the same sample extract resulted, however, in no detector signal for these compounds and chromatograms obtained from the sediment samples showed, furthermore, broad bands eluting at elevated column temperatures (200–250 °C), indicative of the elution of high molecular weight polar compounds.

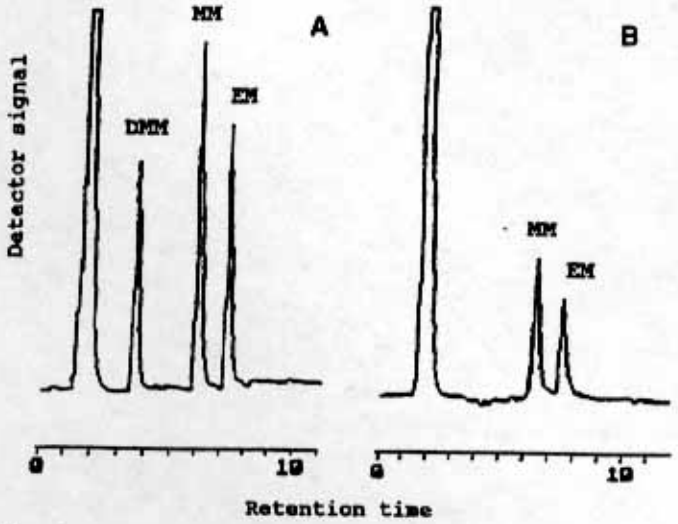

Figure 2
(A) Chromatogram of pure organic mercury standards on new column: DMM, 2.00 pg Hg; MMC, 3.79 pg Hg; EMC, 2.50 pg Hg. (B) Chromatogram of sediment sample after thiosulfate clean-up (MM, 1945.7 pg Hg/g; EM, 1236.6 pg Hg/g).

Interestingly, after such column deterioration, injection of the three standard compounds (DMM, MMC, and EMC) showed that the non-polar compound, DMM, chromatographed without noticeable interferences, whereas MMC and EMC were not detected. Column bake-out and removal of a short length of its inlet end did not improve its efficiency. Once a new column (same stationary phase, DB-1) had been installed, however, with the glass liner, deactivated fused silica, and transfer line unchanged, excellent separation efficiency was again obtained for the organic mercury standards. This clearly indicates that the poor chromatographic response previously observed was associated with column deterioration and not with the injection or detector ends of the GC-AFS system. It seems likely that there is strong interaction of the polar organomercury compounds with high molecular weight compounds (possibly containing sulfur) which have interacted with the stationary phase. As will be shown below, these interferences could be removed from the sample matrix by use of a thiosulfate clean-up step.

Sodium thiosulfate has a high complexing affinity for organic mercury [5,8,33] and can provide rapid clean-up of the initial extracts. **Figure 2B** shows a typical chromatogram from a sediment extract subjected to sodium thiosulfate clean-up. Note that both MM and EM were efficiently separated. DMM cannot be adequately analyzed by methods relying on low pH extraction/preservation, since strongly acidic conditions convert DMM to MM. This explains the absence of DMM in Figure 2B; it is unrelated to the thiosulfate clean-up procedure. With this sample pretreatment, the column was used routinely for several months with no apparent loss in efficiency. The column efficiency and detection limits for dimethylmercury, methyl-, and ethylmercury species under the optimized GC-AFS conditions are shown in **Table 1**.

Table 1
Comparison of column efficiencies and detection limits for DMM, MMC, and EMC determined by GC-AFS.

Analyte	t_r[a] ± *SD* [min]	W_h[b] [min]	n/L[c] [m^{-1}]	*D.L.*[d] [pg]
DMM	3.38 ± 0.0	0.13	271	0.3
MMC	5.82 ± 0.04	0.25	200	0.2
EMC	7.32 ± 0.03	0.23	391	0.2

[a] Retention time.
[b] Peak width at half maximum height.
[c] Number of theoretical plates per unit length, $n = 5.545(t_r/W_h)^2$.
[d] Detection limit.

Using the thiosulfate clean-up procedure, no mercuric chloride conditioning is necessary and thus the limitations imposed by this process are avoided. In addition, the employment of mercury fluorescence detection overcomes the inefficient detection of mercury, characteristic of the ECD, which results from partial on-column decomposition of methylmercury chloride. With this analytical method the organic mercury species (commonly MM and EM) present in a sample can be accurately determined and quantified. This is a major advantage over the ethylation technique where the quantification of EM becomes difficult.

3.2 Quantification and Analysis

Quantitative data were obtained using calibration curves (peak area against concentration of organomercury chloride in picograms of mercury (pg Hg) per 5 µL injection) generated daily using standard solutions of methyl- and ethylmercury chlorides. The relative stand-

ard deviation of the signal for a 3.16 pg Hg/5 µL standard was 1.5 % for peak area measurements ($n = 3$). The calibration curves generated were linear between at least 0 and 4 pg Hg/5 µL. All sediment samples analyzed in this study fell within this linear range. Linear correlation coefficients (r^2) for the calibration curves were 0.998 and 0.999 for MMC and EMC respectively.

Quality control was established by determination of percentage recoveries for each sample using surrogates as described below. A recovery factor (R) which varied between 45 and 65 % for methylmercury and 50 and 80 % for ethylmercury was used in the calculations to compensate for losses of the analytes during sample preparation. R was determined for each sample by analyzing an equivalent amount of the same sample spiked with a known amount of methyl- and ethylmercury and determining the fraction of each compound recovered. It is necessary to determine an R value for each sample since this is influenced by differences in sample matrices which affect the partitioning of organomercury compounds [18,32,34].

4 Conclusion

This chromatographic technique for the analysis of organomercury compounds involves no prederivatization of the analytes, and organic mercury species present in the samples can be accurately determined. In addition, the absolute detection limits were 0.3 pg for dimethylmercury and 0.2 pg for methyl- and ethylmercury; these figures are *ca* four times better than those obtained with existing methods. Direct analysis, *i.e.* without sample clean-up, of dichloromethane extracts of sediment samples from Everglades National Park confirmed previous reports [1,7,11] of severe deterioration of the GC column; this resulted in complete loss of efficiency for organic mercury chlorides after only a few injections. Use of the thiosulfate back-extraction step described above, however, successfully removed matrix interferences, enabling routine analysis of organomercury compounds with no detectable loss of column efficiency.

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DETERMINATION OF METHYLMERCURY IN NATURAL WATERS AT THE SUB-NANOGRAMS PER LITRE LEVEL BY CAPILLARY GAS CHROMATOGRAPHY AFTER ADSORBENT PRECONCENTRATION

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SUMMARY

Methylmercury was preconcentrated from water on to a sulph-hydryl cotton fibre adsorbent, using the column technique or the batch-column two-stage technique. A small volume of 2 M HCl was used to elute methylmercury and to separate it from inorganic mercury; 0.4–0.6 ml of benzene was used to extract methylmercury from the eluate. Analysis was performed by capillary gas chromatography with electron-capture detection. The detection limit for methylmercury was < 0.05 ng l^{-1} in a 4-l water sample. Four surface waters were analysed to test the agreement of methylmercury concentration between the two preconcentration methods, and to test the interference of humic substances on the filtered and unfiltered surface water. The methylmercury concentrations found in different surface water samples ranged from 0.08 to 0.48 ng l^{-1}.

In recent years, elevated mercury levels, mercury being present mainly as methylmercury (MeHg), have been observed in fish in remote oligotrophic lakes. Increased acidification of the lake water combined with high humic content have been shown to increase the MeHg content further in fish, zooplankton and algae [1–8].

Therefore, there has been a rekindling of interest in mercury, to establish why these fish have elevated Hg concentrations and to understand how Hg species are transported/transformed in the biogeochemical cycle. To be able to study the dynamic behaviour of MeHg in the biogeochemical cycle, the ability to measure it in natural water at the ng l^{-1} and sub-ng l^{-1} level is crucial. The difficulty in measuring MeHg in natural waters in remote areas lies in concentrating it from solution. Most previous analytical methods involve many steps, suffer from poor extraction efficiencies and require large sample volumes.

Most of the previous procedures have generally used a selective preconcentration treatment followed by an adequately sensitive analytical technique [9–19]. Extraction with an organic solvent (benzene or toluene) is the most pop-

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ular preconcentration method. However, because the partition coefficient fo MeHg between benzene and water is low, (ca. 5–10 [9]), this method is no adequate for concentrations $\lesssim 0.5$ ng l^{-1}. Several kinds of ion exchange/che lating resins for concentrating MeHg from natural water have also been re ported. Their main functional group is the sulphur donor atom, which favou the bonding of heavy metal ions. Owing to their low capacities, relatively large amounts of resin are required, and because of their strong bonding to MeHg large volumes of strong eluents such as a strong acid or a chelating agent (thi ourea) are commonly used, often followed by a further concentration using an extraction method. In addition, pretreatment of the resin before use is gene ally required [9,16,18], thereby prolonging the time for sample analysis.

The sulphydryl cotton fibre (SCF) adsorbent, produced by introducing th sulphydryl functional group into natural cotton fibres, is very effective for con centrating trace amounts of MeHg [14,20,21]. Previous work [11,22] dem onstrated that the use of a small amount of SCF adsorbent for concentratin MeHg followed by gas chromatography with electron-capture detection (GC ECD) permits the measurement of sub-ng l^{-1} levels of MeHg in natural waters Recently, by the application of capillary GC together with a modified column concentration treatment with SCF adsorbent, significant improvements were made in the sensitivity of the method. For humic-rich water samples a two stage preconcentration procedure consisting of batch concentration as the first and column concentration as the second stage has also been developed and evaluated. These two concentration procedures are compared with respect to the detection limit and interferences in this paper.

EXPERIMENTAL

Methylmercury is preconcentrated from water on to a SCF adsorbent, then the adsorbed MeHg is eluted using a small volume of 2 M HCl and thus separated from inorganic mercury. Benzene is used to extract methylmercury chloride (MeHgCl) from the eluate and the MeHgCl in the benzene extract is determined by GC–ECD.

Handling of the water sample

Sample collection and handling were done carefully to avoid contamination. All glassware used was manufactured of borosilicate or Pyrex glass. New glassware was treated with aqua regia. Sample bottles, glass separating funnels and PTFE and silicone-rubber tubing were leached in 2–4% HCl for at least 24 h. Screw-capped sample vials with PTFE-coated septa (2 and 3.5 ml), used for benzene extraction, were treated with 10% HCl and thoroughly rinsed and leached for several days in deionized water, and before use were rinsed with ethanol and acetone and dried at 180°C. The fresh water sample (1–4 l) was stored in a cold (6°C), dark room after sampling, and analysis was carried out

as soon as possible (within 1 month). The preliminary results showed that ca. 15% MeHg was lost in one fresh water sample containing humic substances when stored for 2 months. Determination of the loss of MeHg in different field samples during storage is currently being studied.

Reagents and solutions

Stock solutions of MeHgCl were prepared by dissolution in benzene or deionized water. The working aqueous solutions of MeHgCl used to spike the artificial and field water samples (ca. 5–30 ng ml^{-1}) were prepared weekly by appropriate dilution from the stock solutions before use and were calibrated using a helium d.c. plasma atomic emission spectrometric (plasma-AES)/sodium tetrahydroborate method [23]. The eluent (2 M) HCl was cleaned by several extractions with benzene.

Synthesis of SCF adsorbent

The SCF adsorbent was synthesized according to the procedure of Liu et al. [24]. A mixture was first prepared by adding the following in sequence to a Pyrex bottle with a wide ground-glass stopper: 100 ml of thioglycolic acid, 60 ml of acetic anhydride, 40 ml of acetic acid (36%) and 0.30 ml of concentrated sulphuric acid. The mixture was allowed to cool to ca. 45°C, then 30 g of cotton wool/60 g of cotton gauze (previously cut into pieces of 10×14 cm) were added and allowed to soak thoroughly in the mixture.

The reaction bottle was placed in an oven for 3–4 days at 40°C, then the product was placed in a filter-funnel with suction filtration and washed thoroughly (ca. 20 times) with deionized water to remove the last traces of thioglycolic acid. The SCF obtained was dried at 40°C for about 24 h and stored in a screw-capped brown bottle in a cold room. No significant changes were observed within 2–3 months after preparation. The SCF wool has good elastic and porous properties, similar to those of the original cotton wool used. The sulphur content of this SCF adsorbent was about 1%.

Column preconcentration

The method described previously [11] was used for column preconcentration. The SCF column used in this study was made of a disposable microlitre pipette tip (Finntips) containing 0.06 g of SCF wool. Before packing, dense clumps of SCF wool should be made less dense by pulling them apart with disposable forceps. The SCF wool should then be packed loosely and as evenly as possible in the tip. Two such SCF columns were filled with deionized water and connected in series. The top of the column was connected to the sample reservoir containing 1–2 l of sample water (Fig. 1). After adjustment of the pH to ca. 4.0 the sample was passed through the column at a flow-rate of about 2–5 ml min^{-1}, controlled by nitrogen pressure, then 1.8 ml of 2 M HCl were pipetted on to the surface of the adsorbent to elute the MeHgCl from the col-

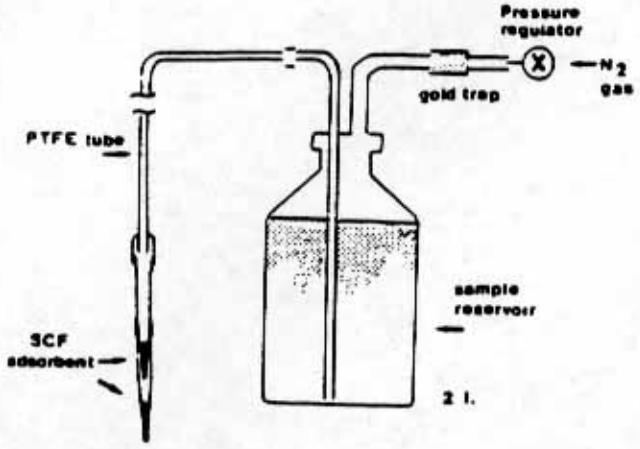

Fig. 1. Schematic diagram of the column concentration apparatus.

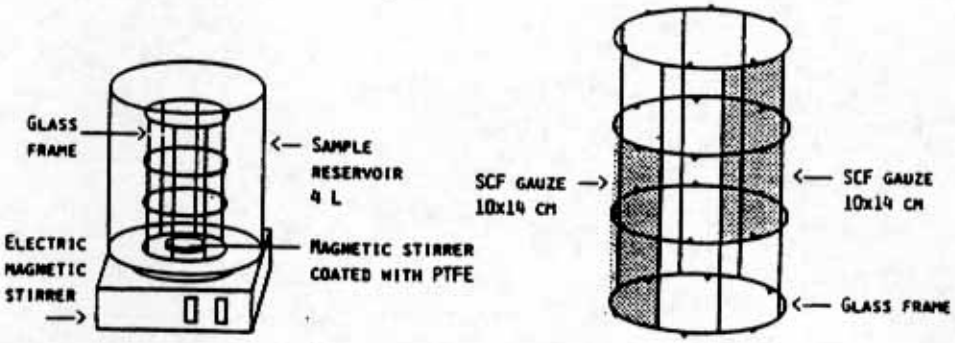

Fig. 2. Schematic diagram of the batch concentration apparatus.

umn. The eluate was collected in a 3.5-ml screw-capped sample vial with a PTFE-coated septum and shaken vigorously with 0.6 ml of benzene for 10 min. The benzene extract was then analysed by GC–ECD.

Two-stage concentration (batch-column procedure)

About 2–4 l of the sample (MeHg concentration > 0.05 ng l^{-1}) were transferred into a Pyrex-glass vessel. After pH adjustment, two pieces of SCF gauze were fixed on a cylindrical glass frame and then placed in the middle of the glass vessel. The water was stirred for 1.5 h using a magnetic stirrer coated with PTFE (Fig. 2).

For desorption of MeHg, the two pieces of SCF gauze were removed from the glass frame, rinsed with deionized water to wash off any adsorbed humic substances and packed into a small funnel. After squeezing water from the adsorbent with a glass rod, 9 ml of 2 M HCl were used to elute MeHg, then the adsorbent was rinsed with ca. 30 ml of deionized water. The eluate and the

solution obtained after rinsing were collected in a 50-ml separating funnel, which was connected to one column packed with 0.03 g of SCF wool. After the preconcentration the MeHg was 200–400 times more concentrated than in the original sample.

The second preconcentration treatment with the column technique was carried out by first neutralizing the acidic eluate to ca. pH 4 with 6 ml of 3 M NaOH and 2 ml of 3 M sodium acetate and then allowing it to pass through the column. MeHgCl was desorbed from the SCF adsorbent by using 1.2 ml of 2 M HCl and the eluate was collected in a 2.0-ml screw-capped sample vial with a PTFE-coated septum and shaken vigorously with 0.4 ml of benzene for 10 min.

Gas chromatographic analysis

A Hewlett-Packard 5710A gas chromatograph equipped with a ^{63}Ni electron-capture detector, a wide-bore capillary column and a Spectra-Physics 4270 integrator was used to determine MeHgCl. Splitless injections of 2–6 μl were made on to a silanized glass liner connected directly to the capillary column by means of a restrictor (J+W Scientific, Riverside, CA). The operating parameters were as follows: column, 15 m×0.52 mm i.d. OV-1701, bonded fused silica, 1-μm film thickness (J+W Scientific); column temperature, 120°C; injector temperature, 200°C; detector temperature, 300°C; carrier gas, helium at 5 ml min^{-1}; and make-up gas, 5% methane in argon at 25 ml min^{-1}.

Quantification was based on peak heights using the external standard method. A 4–5-point calibration graph was established, for concentrations ranging from 0.2 to 2.5 ng ml^{-1}, each day that samples were analysed.

RESULTS AND DISCUSSION

Extraction efficiency

As a single extraction step was used to extract MeHg eluted from the SCF adsorbent, the extraction was not complete. The extraction efficiency was determined experimentally using 1.8 or 1.2 ml of eluent (2 M HCl) containing known MeHg concentrations extracted with 0.6 or 0.4 ml benzene, respectively. The MeHg concentration range (0.2–3.0 ng ml^{-1}) chosen for the determination of extraction efficiency corresponded to that found in the eluate after preconcentration from fresh water samples.

The amount of MeHg in the eluate was calculated using the equation

$$MeHgCl_{eluate} = (MeHgCl_b V_b)/EV_{H_2O} \quad (1)$$

where $MeHgCl_{eluate}$ denotes the concentration of MeHgCl in the eluate before extraction, $MeHgCl_b$ that in the benzene phase and V_b and V_{H_2O} are the vol-

umes of benzene and eluate, respectively. The extraction efficiency (E) obtained from this experiment when $V_{H_2O}/V_b=3$ was 0.65 ± 0.02 ($n=25$). Inserting $E=0.65$ in the equation

$$E=K/(K+V_{H_2O}/V_b)$$

the partition coefficient ($K=\mathrm{MeHgCl_b}/\mathrm{MeHgCl_{H_2O}}$) was 5.6, which is very close to the value of 6 obtained by Fujita and Iwashima [9] for a solution containing 1.7 M HCl and 30% NaCl.

GC analysis

GC analyses were performed at the highest available detector sensitivity detector (attenuation $\times 1$). Temperature programming was impossible owing to disturbances to the baseline at this low attenuation. Because the highest sensitivity of the detector is used, impurities in the solvents and reagents used and/or those introduced during sample preparation can easily mask the MeHgCl peak. Previously, a packed column had been used [11]. However, the separation from the solvent peak and nearby interfering peaks was very poor. Baseline separation from the solvent peak and improved separation from interfering peaks was obtained by using a wide-bore capillary column (see Experimental and Fig. 3a,b). The sensitivity was also improved.

MeHgCl is easily decomposed when exposed to active sites on glass and metal surfaces at elevated temperatures. Therefore, the injection liner was silanized twice using 10% dichlorodimethylsilane in toluene. Decomposition of MeHgCl

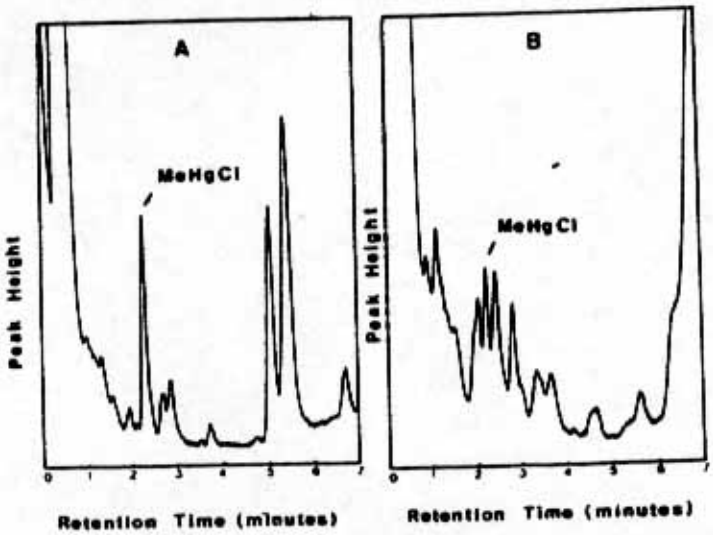

Fig. 3. Gas chromatograms: (A) 3 μl of standard benzene solution, 1.6 ng ml^{-1} MeHgCl; (B) 5 μl of benzene extract after 6820-fold preconcentration from field surface water containing 0.15 ng l^{-1} MeHg.

was even observed on the capillary column. The column could be regenerated by successive rinses with hexane, toluene and methanol.

The chromatographic separation is shown in Fig. 3. The interfering peaks on either side of MeHgCl were suspected to be derived from the HCl used as eluent in the preconcentration steps. Attempts were made to remove these interfering compounds from the HCl, and the best results were obtained by extracting freshly prepared 2 M HCl several times with benzene.

Each sample analysis required about 30 min to elute all the peaks that could interfere with a subsequent injection. The ECD response to MeHgCl was linear over the range required for these analyses (1.5–20 pg) and the correlation coefficient of the linear regression for the calibration graph was generally better than 0.990. The detection limit was ca. 0.5–1.0 pg.

Analysis of artificial and field water samples

In order to measure and control the quality of the analytical results with the methods developed in this study, MeHg was determined in artificial water samples, blanks and field water samples spiked with known concentrations of MeHg.

To evaluate the two-stage preconcentration method and to compare it with the column method, several experiments were done to assess the recovery of methylmercury in artificial waters using both methods. The concentration ranges chosen corresponded to those found in fresh water samples. The results in Table 1 show that MeHg was quantitatively adsorbed on the absorbent using the column concentration method. The recovery of the spiked MeHg varied from 81 to 108% with a standard deviation of 9.3% and a mean value of 95.8%. The interval estimate of the mean value was ±8.3% (95% confidence level). The recovery of spiked MeHg from the two-stage method varied from 48 to 75% and increased with increasing MeHg concentration. Figure 4 shows that

TABLE 1

Recovery of spiked methylmercury in artificial water

Concentration method	Volume (l)	Methylmercury concentration (ng l^{-1} as Hg)	Recovery (%) (Mean ± S.D.)
Column	0.1–4	0.1–30	95.8 ± 9.3 (n=8)
Two-stage	4	0.05	55.5 ± 4.9 (n=2)
		0.1	48.3 ± 6.0 (n=3)
		0.2	61.5 ± 4.9 (n=2)
		0.3	61.5 ± 7.8 (n=2)
		0.4	71.0 ± 5.5 (n=2)
		0.5	75.0 ± 5.6 (n=2)

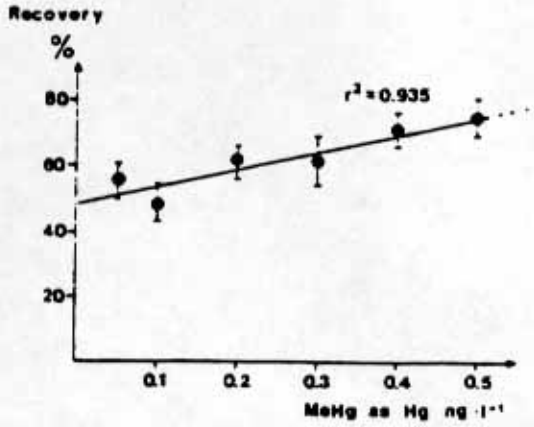

Fig. 4. Linear regression between recovery of spiked MeHg and MeHg concentration in artificial water samples. The error bars correspond to standard deviations of measured MeHg concentrations shown in Table 1.

the correlation coefficient, $r^2 = 0.935$, is high. The lower recovery compared with that of the column method was mainly due to losses of MeHg in the batch concentration step.

The blank was also checked for contributions of MeHg from the reagents and the method used. No MeHg was detected in the blank for either method.

For field samples, 2–4 l of water were usually used for the determination of MeHg (>0.05 ng l^{-1}). The column method was generally applied for samples with a low content of humic substances (dissolved organic carbon <5 mg l^{-1} or colour <20 mg Pt l^{-1}). The column method was not used for humic-rich sample waters, as humic substances may interact on the surface of mineral particles occurring in the colloidal state, resulting in partial or complete blockage of the column, and they may also produce emulsions during the extraction step, making it difficult to separate the small volume (0.6 ml) of benzene extract from the aqueous phase. These two problems could be avoided by using the two-stage method.

Table 2 shows that the MeHg concentrations in two surface waters determined by use of the two preconcentration methods were within 15% of the mean.

As a check on the difference among MeHg determinations in filtered and unfiltered surface waters, two lake waters were examined. Table 3 shows that the MeHg concentrations in the filtered and unfiltered waters were within 15% of the mean and the recoveries of spiked MeHg were very close to those obtained with the artificial waters (see Fig. 4).

Compared with the column concentration method, the two-stage concentration treatment had the disadvantages of a larger number of chemical steps and lower yields. However, the two-stage method had less problem with the for-

TABLE 2

Determination of methylmercury in two surface waters with the column concentration and two-stage concentration methods

Sampling location	Sampling date	Methylmercury concentration (ng l^{-1} as Hg)	
		Column method	Two-stage method
Runoff F1 catchment in Lake Gärdsjön watershed	15.10.87	0.18	0.17
Lake Loppesjön[a]	22.9.87	0.09	0.08

[a]The total concentration of mercury was 1.35 ng l^{-1} (results obtained by Y.H. Lee and Å. Iverfeldt).

TABLE 3

Determination of methylmercury in filtered and unfiltered waters from two lake samples using the two-stage preconcentration method

Sampling location	Sampling date	Colour (mg Pt l^{-1})	Methylmercury concentration (ng l^{-1} as Hg)	
			Unfiltered	Filtered
Lake St. Skärsjön[a]	24.8.88	110	0.48	0.48
Lake Löjesjön[b]	24.8.88	98	0.30	0.34

[a]Recovery of spiked MeHg was 69% and the total concentration of mercury was 7.73 ng l^{-1} (results obtained by Y.H. Lee and Å. Iverfeldt). [b]Recovery of spiked MeHg was 47% and the total concentration of mercury was 8.02 ng l^{-1} (results obtained by Y.H. Lee and Å. Iverfeldt).

mation of emulsions during the extraction step. Therefore, the latter method is suitable for field samples with a high content of humic substances.

The detection limit with both the column and the two-stage preconcentration treatments was <0.05 ng l^{-1} when starting with a 4-l sample when the GC injection volume was 5 μl. The main sources of error were the uncertainty of the GC analysis, the extraction efficiency and the recovery of spiked MeHgCl in natural water samples.

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APPENDIX C: Data Reviews

EVERGLADES ECOSYSTEM ASSESSMENT
(PHASE II REMAP)

Data Review
May 1999 Sampling

Data Review, May 1999 Sampling

Foreward

The data review documents developed by the US Environmental Protection Agency (EPA) as part of the Investigation of Mercury Contamination in the Florida Everglades Ecosystem and Everglades Ecosystem Assessment (Phase II REMAP) Project are presented in the Data Review May 1999 (M4) and September 1999 Sampling (M5) documents.

The Phase II data review determines whether the Data Quality Objectives (DQO) have been satisfied as outlined in the Quality Assurance Project Plan (QAPP). The M4 and M5 Sampling results were analyzed to determine whether they met the criteria developed during the planning phase and whether the total error within the tolerable decision error ranges as specified in the QAPP to support decisions.

The Data Review, May 1999 Sampling document summarizes the assessments of the critical and non-critical parameters. Ten percent of the samples were randomly selected during the validation process to characterize the quality of the data set. Five of the eleven critical parameters are qualified with a “J”. Parameters associated with this qualifier should be considered an estimate for a number of quality control variances. The results for total phosphorus, total nitrogen, total organic carbon in surface water and methylmercury and bulk density in soil should be considered an estimate based on findings. A table summarizing the critical and non-critical parameters is enclosed in the Data Review document along with the detailed calculations and criteria for each selected sample and parameter.

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CRITICAL QA/QC SUMMARIES

EPA SESD South Florida Phase II Dry Season Sampling: May 1999
Summarized Qualifiers for 10% of the Critical Parameters

	Surface Water					Soil/ Sediment				Fish	
	SERC Laboratory				Battelle Lab	SERC Laboratory				SERC Laboratory	
Station ID	Total Phosphorus	Total Nitrogen	TOC	Total Mercury	Methyl Mercury	Methyl Mercury	Total Phosphorus	AFDW	Bulk Density	Total Mercury	Length/Weight
M4-500											
M4-501	"**"		"H"			"DQO"		"DQO"	"DQO (NR)"	No Sample	No Sample
M4-508	"M","**"	"**","M"	"H"			No Sample		"DQO"	"DQO (NR)"		
M4-533	"M","**"	"**"	"H"			"M"			"DQO (NR)"		
M4-538	"M","**"	"**"	"H"			No Sample			"DQO (NR)"		
M4-548	No Sample	No Sample	No Sample	"M"		"DQO"			"DQO (NR)"		
M4-556	"M","**"	"**"	"H"			"DQO", "*", "M"			"DQO (NR)"		
M4-566	"M","**"	"**","M"	"H"			"DQO", "M"			"DQO (NR)"		
M4-568	"M","**"	"**"	"H"	"M (NR)"		"DQO"			"DQO (NR)"		
M4-576	"M","**"	"**"	"H"						"DQO (NR)"		
M4-586	"M","**"	"**"	"H"	"M (NR)"		"*", "M"		"DQO"	"DQO (NR)"	No Sample	No Sample
M4-594	"M","**"	"**"	"H"			"DQO"	"DQO"		"DQO (NR)"		
M4-599	"M","**"	"**"	"H"	"M"		"DQO"			"DQO (NR)"		
M4-809	"M","**"	"**","M"	"H"			"*","M"			"DQO (NR)"		
M4-872	"M","**"	"**","M"	"H"			"DQO (NR)"			"DQO (NR)"		
Qualifier Notation	**"J"**	**"J"**	**"J"**			**"J"**			**"J", "***"**		

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.
"B" Analyte concentration in the associated blank was >3 times the MDL.
"H" Analysis digestion performed after holding times have expired.
"NR" Data was unavailable for review.
"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

"*" The matrix recoveries were below QAPP QA limits. MeHg matrix spikes are run with every sample and the sample is adjusted based on matrix affects.
"**" Blanks were reported above the MDL. Lab water was used as the blank water but not in the sample digestion process.
"***" Phase II soil/sediment samples were prepped with a high speed blender which may alter comparability with the phase I bulk density values.

Comments:
The coefficient of variance (R2) values for methyl mercury were not reviewed.

EPA SESD South Florida Phase II Dry Season Sampling: May 1999
Summarized Findings for 10% of the Critical Parameters Reviewed
Total Mercury in Surface Water Analyzed By SERC

Sample ID by QC Batch	Matrix Spike Not Reviewed for Batch	Data Entry Check Not Noted	Holding Time Exceeded	CCV Not Reviewed	Blank Result >3 MDL	Corr. Coef. Not Reviewed	Cannot Reproduce Results Calcs.	Precision Criteria Not Met	No Dups/Reps Reviewed	No Blanks Reviewed in Batch	Potential Matrix Effect (Data User Note)
M4-508-SWF M4-809-SWF											
M4-533-SWF M4-538-SWF											
M4-548-SWF											X
M4-556-SWF M4-872-SWF M4-501-SWF											
M4-566-SWF											
M4-568-SWF M4-586-SWF	X X										
M4-576-SWF M4-594-SWF											
M4-599-SWF											X

Comments:
1. Samples from 61 stations were analyzed.
2. Results are an average of 3 separate runs.
3. No matrix spikes were reviewed for batches that included samples 568 and 586.
4. Potential matrix effects are indicated for samples 548 and 599 based on matrix spike recoveries.

Footnotes:
" X " Indicates this situation did occur.

EPA SESD South Florida Phase II Dry Season Sampling: May 1999
Summarized Findings for 10% of the Critical Parameters Reviewed
Total Mercury in Fish Analyzed By SERC

Sample ID by QC Batch	Matrix Spike Not Reviewed for Batch	Data Entry Check Not Noted	Holding Time Exceeded	CCV Not Reviewed	Blank Result >3 MDL	Corr. Coef. Not Reviewed	Cannot Reproduce Results Calcs.	Precision Criteria Not Met	No Dups/Reps Reviewed	No Blanks Reviewed in Batch	Potential Matrix Effect (Data User Note)
M4-501-FIF	NO SAMPLE										
M4-508-FIF		*									
M4-533-FIF		*									
M4-538-FIF		*									
M4-548-FIF		*									
M4-556-FIF		*									
M4-566-FIF		*	X**								
M4-568-FIF		*	X**								
M4-576-FIF			X**								
M4-586-FIF	NO SAMPLE										
M4-594-FIF			X**								
M4-599-FIF			X**								
M4-809-FIF			X**								
M4-872-FIF			X**								

Comments:

1. Documentation of data entry checked was not reviewed, with the exceptions of 576, 594, 599, 809, and 872 where documentation of data entry checked was found.
2. QAPP holding times were exceeded for samples 566, 568, 576, 594, 599, 809, and 872.

Footnotes:

" * " Refer to the Standard Operating Procedures.

" ** " The fish samples were frozen prior to analysis. SERC references a study demonstrating no significant loss of analyte for frozen samples exceeding holding times.The study can be provided by FIU upon request.

" X " Indicates this situation did occur.

EPA SESD South Florida Phase II Dry Season Sampling: May 1999
Summarized Findings for 10% of the Critical Parameters Reviewed
Total Phosphorus in Surface Water Analyzed By SERC

Sample ID by QC Batch	Matrix Spike Not Reviewed for Batch	Data Entry Check Not Noted	Holding Time Exceeded	CCV Not Reviewed	Blank Result >3 MDL	Corr. Coef. Not Reviewed	Cannot Reproduce Results Calcs.	Precision Criteria Not Met	No Dups/Reps Reviewed	No Blanks Reviewed in Batch	Potential Matrix Effect (Data User Note)
M4-501-SWF		*	**		X***						
M4-508-SWF		*			X***						X
M4-533-SWF		*	**		X***						
M4-538-SWF		*			X***						X
M4-548-SWF	NO SAMPLE										
M4-556-SWF		*			X***						X
M4-566-SWF		*	**		X***						X
M4-568-SWF		*			X***						X
M4-576-SWF		*			X***						X
M4-586-SWF		*			X***						X
M4-594-SWF		*			X***						X
M4-599-SWF		*			X***						X
M4-809-SWF		*			X***						X
M4-872-SWF		*			X***						X

Comments:

1. Blank results included with this batch were >3 times the MDL.
2. The blank results were significantly different (0.021 ppm vs. 0.0081 ppm) in this batch.
3. Holding times were exceeded for samples 501, 533, and 566.
4. Documentation of data entry check was not reviewed, with the exception of 501 where documentation of data entry check was verified.

Footnotes:

" * " Refer to the Standard Operating Procedures.

" ** " Holding time goal only.

" *** " Blanks were reported above the MDL. Lab water was used as the blank water but not in the sample digestion process. Procedures have been modified to correct this.

" X " Indicates this situation did occur.

EPA SESD South Florida Phase II Dry Season Sampling: May 1999
Summarized Findings for 10% of the Critical Parameters Reviewed
Total Nitrogen in Surface Water Analyzed By SERC

Sample ID by QC Batch	Matrix Spike Not Reviewed for Batch	Data Entry Check Not Noted	Holding Time Exceeded	CCV Not Reviewed	Blank Result >3 MDL	Corr. Coef. Not Reviewed	Cannot Reproduce Results Calcs.	Precision Criteria Not Met	No Dups/Reps Reviewed	No Blanks Reviewed in Batch	Potential Matrix Effect (Data User Note)
M4-501-SWF	X	*	**	X		NA			X		
M4-508-SWF	X	*	**	X	X***	NA			X		
M4-533-SWF		*	**		X***	NA					
M4-538-SWF		*	**		X***	NA					
M4-548-SWF	NO SAMPLE										
M4-556-SWF		*	**		X***	NA					
M4-568-SWF		*	**		X***	NA		X			
M4-576-SWF		*	**		X***	NA		X			
M4-586-SWF		*	**		X***	NA		X			
M4-594-SWF		*	**		X***	NA		X			
M4-599-SWF		*	**		X***	NA		X			
M4-566-SWF		*	**		X***	NA					X
M4-809-SWF		*	**		X***	NA					X
M4-872-SWF		*	**		X***	NA					X

Comments:

1. No matrix spikes, dups/reps, or CCVs were reviewed for batches containing samples 501 and 508.
2. Documentation of data entry check was not reviewed for any samples.
3. QAPP holding time goals were exceeded for all samples analyzed.
4. Blank results for all sample batches, except the batch containing sample 501, were > 3 times the MDL a follows: 1/1 blanks included in sample 508 batch; 1/3 blanks included in the sample 533 batch; 3/3 blanks included in the sample 568 batch and 1/3 blanks included in the sample 566 batch.
5. Correlation coefficients is not applicable is this analysis (blank and one other point curve).
6. A blank correction was used for sample 501. Sample was diluted with the lab water.
7. Potential matrix effects are indicated for samples 566, 809, and 872 based on matrix spike recoveries.

Footnotes:

" * " Refer to the Standard Operating Procedures.
" ** " Holding time goal only.
" *** " Blanks were reported above the MDL. Lab water was used as the blank water but not in the sample digestion process. Procedures have been modified to correct this.
" X " Indicates this situation did occur.

EPA SESD South Florida Phase II Dry Season Sampling: May 1999
Summarized Findings for 10% of the Critical Parameters Reviewed
Total Organic Carbon in Surface Water Analyzed By SERC

Sample ID by QC Batch	Matrix Spike Not Reviewed for Batch	Data Entry Check Not Noted	Holding Time Exceeded	CCV Not Reviewed	Blank Result >3 MDL	Corr. Coef. Not Reviewed	Cannot Reproduce Results Calcs.	Precision Criteria Not Met	No Dups/Reps Reviewed	No Blanks Reviewed in Batch	Potential Matrix Effect (Data User Note)
M4-501-SWF		*	X								
M4-508-SWF		*	X								
M4-533-SWF		*	X								
M4-538-SWF		X	X								
M4-548-SWF	NO SAMPLE										
M4-556-SWF		*	X								
M4-566-SWF		*	X								
M4-568-SWF		*	X								
M4-576-SWF		*	X								
M4-586-SWF		*	X								
M4-594-SWF		*	X								
M4-599-SWF		*	X								
M4-809-SWF		*	X								
M4-872-SWF		*	X								

Comments:

1. Holding times were exceeded for all samples.
2. Based on the Sept. 99 technical review of the SERC laboratory, the QA/QC check standards that were being used were made in 1998. Standards should be prepared at least every 2 months.
3. Based on the Sept. 99 technical review of the SERC laboratory, it was noted that sample injection volumes were slightly less than was indicated by the setting of the pipette system (~4%).
4. The laboratory comparisons that were performed did show a bias between the two laboratories. It is unknown why the laboratory results don't compare, but each laboratory is using a different method and instrument.

Footnotes:

" * " Refer to the Standard Operating Procedures.

" X " Indicates this situation did occur.

EPA SESD South Florida Phase II Dry Season Sampling: May 1999
Summarized Findings for 10% of the Critical Parameters Reviewed
Total Phosphorus in Soil Analyzed By SERC

Sample ID by QC Batch	Preservation Not Documented	Matrix Spike Not Reviewed for Batch	Data Entry Check Not Noted	Holding Time Exceeded	CCV Not Reviewed	Blank Result >3 MDL	Corr. Coef. Not Reviewed	Cannot Reproduce Results Calcs.	Precision Criteria Not Met	No Dups/Reps Reviewed	No Blanks Reviewed in Batch	Potential Matrix Effect (Data User Note)
M4-508-SFF				*							X	
M4-533-SFF				*							X	
M4-538-SFF				*							X	
M4-594-SFF				*								
M4-501-SFF				*								
M4-548-SFF				*								
M4-556-SFF				*								
M4-566-SFF				*								
M4-568-SFF				*								
M4-576-SFF				*								
M4-586-SFF				*								
M4-599-SFF				*								
M4-809-SFF				*								
M4-872-SFF				*								

Comments:

1. No blank results were reviewed for sample 508 batch.
2. QAPP holding time goals were exceeded for all samples analyzed.
3. 1/4 dups analyzed in the sample 594 batch exceeded RPD DQOs.
4. 1/9 stds analyzed in the sample 594 batch exceeded %R DQOs.

Footnotes:

" * " Holding time goal only.

" X " Indicates this situation did occur.

EPA SESD South Florida Phase II Dry Season Sampling: May 1999
Summarized Findings for 10% of the Critical Parameters Reviewed
Soil Ash-Free Dry Weight Analyzed By SERC

Sample ID by QC Batch	Preservation Not Documented	Matrix Spike Not Reviewed for Batch	Data Entry Check Not Noted	Holding Time Exceeded	CCV Not Reviewed	Blank Result >3 MDL	Corr. Coef. Not Reviewed	Cannot Reproduce Results Calcs.	Precision Criteria Not Met	No Dups/Reps Reviewed	No Blanks Reviewed in Batch	Potential Matrix Effect (Data User Note)
M4-501-SFF M4-508-SFF										X X		
M4-533-SFF M4-538-SFF												
M4-548-SFF												
M4-556-SFF												
M4-566-SFF M4-568-SFF												
M4-576-SFF												
M4-586-SFF									X			
M4-594-SFF M4-599-SFF												
M4-809-SFF												
M4-872-SFF												

Comments:

1. No dups/ reps were reviewed that would be associated with sample 501 and sample 508.
2. The RPD for duplicates associated with sample 586 exceeded project DQOs.

Footnotes:

" X " Indicates this situation did occur.

EPA SESD South Florida Phase II Dry Season Sampling: May 1999
Summarized Findings for 10% of the Critical Parameters Reviewed
Soil Bulk Density Analyzed By SERC

Sample ID by QC Batch	Preservation Not Documented	Matrix Spike Not Reviewed for Batch	Data Entry Check Not Noted	Holding Time Exceeded	CCV Not Reviewed	Blank Result >3 MDL	Corr. Coef. Not Reviewed	Cannot Reproduce Results Calcs.	Precision Criteria Not Met	No Dups/Reps Reviewed	No Blanks Reviewed in Batch	Potential Matrix Effect (Data User Note)
M4-508-SFF										X		
M4-533-SFF										X		
M4-538-SFF										X		
M4-594-SFF										X		
M4-501-SFF										X		
M4-548-SFF										X		
M4-556-SFF										X		
M4-566-SFF										X		
M4-568-SFF										X		
M4-576-SFF										X		
M4-586-SFF										X		
M4-599-SFF										X		
M4-809-SFF										X		
M4-872-SFF										X		

Comments:

1. All project samples for this parameter were analyzed in the same analytical batch.
2. No dups/reps were reviewed for this analytical batch.

Footnotes:

" X " Indicates this situation did occur.

EPA SESD South Florida Phase II Dry Season Sampling: May 1999
Summarized Findings for 10% of the Critical Parameters Reviewed
Methylmercury in Surface Water Analyzed By Battelle Laboratory

Sample ID by QC Batch	Matrix Spike Not Reviewed for Batch	Data Entry Check Not Noted	Holding Time Exceeded	CCV Not Reviewed	Blank Result >3 MDL	Corr. Coef. Not Reviewed	Cannot Reproduce Results Calcs.	Precision Criteria Not Met	No Dups/Reps Reviewed	No Blanks Reviewed in Batch	Potential Matrix Effect (Data User Note)
M4-500-SWB		*								**	
M4-538-SWB		*								**	
M4-556-SWB		*								**	
M4-566-SWB		*								**	
M4-576-SWB		*								**	
M4-809-SWB		*								**	
M4-901-SWB		*								**	

Comments:

1. Instrument blanks were not reviewed for any samples.
2. Documentation of data entry check was not reviewed for any samples.

Footnotes:

" * " Refer to the Standard Operating Procedures.
" ** " Method blanks were reported only.
" X " Indicates this situation did occur.

NON-CRITICAL QA/QC SUMMARIES

Non-Critical Parameters Analyzed by SERC
Review of the May, 1999 (M4) Data Set

	Supporting Documentation												
			Laboratory Records										
Analysis	Approved Project QA Plan	Comprehensive QA Plan (Lab	Calibration Verification	Analysis/Digestion Date/Time	Parameters Analyzed	Spikes Utilized	Duplicates Utilized	Instrument Maintenance Logs	Digestion Logs	Bench Sheets	Run Logs	Supplies/Consumables	Performance Evaluations
NH4	X	X	X	X	X	X	X	X	X	X	X	X	X
NO2	X	X	X	X	X	X	X	X	X	X	X	X	X
NO3	X	X	X	X	X	X	X	X	X	X	X	X	X
PO4	X	X	X	X	X	X	X	X	X	X	X	X	NR
CH4	X	X	X	X	X	NR	X	X	X	X	X	NR	NR
CO2	X	X	X	X	X	NR	X	X	X	X	X	NR	NR
APA	X	X	NR	X	X	NR	X	X	X	X	X	NR	NR
Mineral Content	X	X	NA	X	X	NA	X	X	X	X	X	X	NR
Diatoms	X	X	X	X	X	NR	NR	NR	NR	NR	NR	NR	NR
Pigments	**	**	**	**	**	**	**	**	**	**	**	**	**
Chlorophyll a	**	**	**	**	**	**	**	**	**	**	**	**	**
Ethyl Mercury	**	**	**	**	**	**	**	**	**	**	**	**	**

Footnotes:

" * " Analyses are in the process of being analyzed.

" ** " No analyses required.

" NR " Not Reviewed

" NA " Not Applicable

" X " Indicates this situation did occur.

Non-Critical Parameters Analyzed by Battelle Laboratories
Review of the May, 1999 (M4) Data Set

	Supporting Documentation												
			Laboratory Records										
Analysis	Approved Project QA Plan	Comprehensive QA Plan (Lab	Calibration Verification	Analysis/Digestion Date/Time	Parameters Analyzed	Spikes Utilized	Duplicates Utilized	Instrument Maintenance Logs	Digestion Logs	Bench Sheets	Run Logs	Supplies/Consumables	Performance Evaluations
Total Mercury (water)	X	X	X	X	X	X	X	X	X	X	X	NR	NR
Methylmercury (soil)	X	X	X	X	X	X	X	X	X	X	X	NR	NR
Methylmercury (floc)	X	X	X	X	X	X	X	X	X	X	X	NR	NR
Methylmercury (periphyton)	X	X	X	X	X	X	X	X	X	X	X	NR	NR

Footnotes:
"NR" Not Reviewed
" X " Indicates this situation did occur.

CRITICAL QA/QC REVIEW

Water - SERC

Recalculated Results for 10% of the Total Phosphorus in Surface Water Sample Set
Analyzed by Florida International University (SERC) for the May 1999 Dry Season (M4)

Entered by gtc 11/30/99, njs 12/3/99 Checked by jtm 1/6/00

Sample	QC Batch	Qualifier Note	Dilution	Rep um	Base Line Factor um	Peak - Base Line um	MW g	Corrected Peak * MW	Sample Results ppm	Detection Limit ppm	
M4-508-SWF	08-06-99 Channel 2		1	1.20	-0.09	1.29	31	39.99	0.040	0.0006	
M4-538-SWF	"		1	1.08	-0.09	1.17	31	36.27	0.036	0.0006	
M4-548-SWF	"	NO SAMPLE									No sample was collected
M4-556-SWF	"		1	0.26	-0.09	0.35	31	10.85	0.011	0.0006	
M4-566-SWF	"		1	0.19	-0.09	0.28	31	8.68	0.009	0.0006	
M4-568-SWF	"		1	0.31	-0.09	0.4	31	12.4	0.012	0.0006	
M4-576-SWF	"		1	0.17	-0.09	0.26	31	8.06	0.008	0.0006	
M4-586-SWF	"		1	0.53	-0.09	0.624	31	19.344	0.019	0.0006	
M4-594-SWF	"		1	0.49	-0.09	0.577	31	17.887	0.018	0.0006	
M4-599-SWF	"		1	0.47	-0.09	0.556	31	17.236	0.017	0.0006	
M4-809-SWF	"		1	0.31	-0.09	0.402	31	12.462	0.012	0.0006	
M4-872-SWF	"		1	0.30	-0.09	0.39	31	12.09	0.012	0.0006	

Sample		Qualifier Note	Dilution	Rep um	Base Line Factor um	Peak - Base Line um	MW g	Corrected Peak * MW	Sample Results ppm	Detection Limit*3 ppm	SPK CONC (ppm)	R%	RPD
UMS	"		1	0.138422	-0.09	0.228	31	7.08	0.00708	0.0018			
UMS	"		1	0.133859	-0.09	0.224	31	6.94	0.00694	0.0018			2.018
MS	"	"M"	1	1.02088	-0.09	1.111	31	34.44	0.03444	0.0018	0.0401	68.6	
MS	"	"M"	1	1.092386	-0.09	1.182	31	36.65	0.03665	0.0018	0.0401	74.1	-6.236
CCV	"		1	1.493456	-0.09	1.583	31	49.09	0.04909	0.0018	0.05	98.17	
CCV	"		1	1.48841	-0.09	1.578	31	48.93	0.04893	0.0018	0.05	97.86	
CCV	"		1	1.520315	-0.09	1.610	31	49.92	0.04992	0.0018	0.05	99.84	
CCV	"		1	1.520315	-0.09	1.610	31	49.92	0.04992	0.0018	0.05	99.84	
CCV	"		1	1.497409	-0.09	1.587	31	49.21	0.04921	0.0018	0.05	98.42	
CCV	"		1	1.513901	-0.09	1.604	31	49.72	0.04972	0.0018	0.05	99.44	
CCV	"		1	1.483366	-0.09	1.573	31	48.77	0.04877	0.0018	0.05	97.55	
M537	"		1	0.764442	-0.09	0.854	31	26.49	0.02649	0.0018			
M537D	"		1	0.693524	-0.09	0.784	31	24.29	0.02429	0.0018			8.659
M561	"		1	1.877519	-0.09	1.968	31	60.99	0.06099	0.0018			
M561D	"		1	1.649798	-0.09	1.740	31	53.93	0.05393	0.0018			12.285
M574	"		1	0.269442	-0.09	0.359	31	11.14	0.01114	0.0018			
M574D	"		1	0.267805	-0.09	0.358	31	11.09	0.01109	0.0018			0.456
M599	"		1	0.466677	-0.09	0.557	31	17.26	0.01726	0.0018			
M599D	"		1	0.475123	-0.09	0.565	31	17.52	0.01752	0.0018			-1.506
Digestion Blk	"	"**"	1	0.586559	-0.09	0.677	31	20.97	0.02097	0.0018			
Digestion Blk	"	"**"	1	0.169684	-0.09	0.260	31	8.05	0.00805	0.0018			

"**" Blanks were reported above the MDL. Lab water was used as the blank water but not in the sample digestion process. Procedures have been modified to correct this.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

Recalculated Results for 10% of the Total Phosphorus in Surface Water Sample Set
Analyzed by Florida International University (SERC) for the May 1999 Dry Season (M4)

Entered by njs 03-06-00 Checked by

Sample		Qualifier Note	Dilution	Rep um	Base Line Factor um	Peak - Base Line um	MW g	Corrected Peak * MW	Sample Results ppm	Detection Limit ppm			
M4-501-SWF	11-09-99 Channel 1	"H"	1	2.8344317	-0.45	3.284	31	101.817383	0.102	0.0006	Sample was reanalyzed on 11-09-99.		
Sample		**Qualifier Note**	**Dilution**	**Rep um**	**Base Line Factor um**	**Peak - Base Line um**	**MW g**	**Corrected Peak * MW**	**Sample Results ppm**	**Detection Limit*3 ppm**	**SPK CONC (ppm)**	**R%**	**RPD**
UMS	11-09-99 Channel 1		1	0.642113	-0.45	1.092	31	33.86	0.03386	0.0018			
UMS	"		1	0.611171	-0.45	1.061	31	32.90	0.03290	0.0018			2.874
MS	"		1	1.885198	-0.45	2.335	31	72.39	0.07239	0.0018	0.0401	98.5	
MS	"		1	1.922837	-0.45	2.373	31	73.56	0.07356	0.0018	0.0401	101.4	-1.599
CCV	"		1	7.547741	-0.45	7.998	31	247.93	0.24793	0.0018	0.25	99.17	
CCV	"		1	7.94943	-0.45	8.399	31	260.38	0.26038	0.0018	0.25	104.15	
CCV	"		1	7.713398	-0.45	8.163	31	253.07	0.25307	0.0018	0.25	101.23	
CCV	"		1	7.920441	-0.45	8.370	31	259.48	0.25948	0.0018	0.25	103.79	
CCV	"		1	7.810336	-0.45	8.260	31	256.07	0.25607	0.0018	0.25	102.43	
Digestion Blk	"	"**"	1	0.467208	-0.45	0.917	31	28.43	0.02843	0.0018			
Digestion Blk Dup	"	"**"	1	0.324104	-0.45	0.774	31	24.00	0.02400	0.0018			

Sample		Qualifier Note	Dilution	Rep um	Base Line Factor um	Peak - Base Line um	MW g	Corrected Peak * MW	Sample Results ppm	Detection Limit ppm			
M4-533-SWF	11-08-99 Channel 2	"H"	1	4.15	0	4.150	31	128.638282	0.129	0.0006	Sample was reanalyzed on 11-09-99.		
Sample		**Qualifier Note**	**Dilution**	**Rep um**	**Base Line Factor um**	**Peak - Base Line um**	**MW g**	**Corrected Peak * MW**	**Sample Results ppm**	**Detection Limit*3 ppm**	**SPK CONC (ppm)**	**R%**	**RPD**
UMS	11-08-99 Channel 2		1	0.282074	0	0.282	31	8.74	0.00874	0.0018			
UMS	"		1	0.309724	0	0.310	31	9.60	0.00960	0.0018			-9.344
MS	"	"M" (NR)	1	0.473718	0	0.474	31	14.69	0.01469	0.0018	No spike added		
MS	"	"M" (NR)	1	0.581571	0	0.582	31	18.03	0.01803	0.0018	No spike added		-20.440
CCV	"		1	1.458868	0	1.459	31	45.22	0.04522	0.0018	0.05	90.45	
CCV	"		1	1.50171	0	1.502	31	46.55	0.04655	0.0018	0.05	93.11	
CCV	"		1	1.533685	0	1.534	31	47.54	0.04754	0.0018	0.05	95.09	
CCV	"		1	1.541847	0	1.542	31	47.80	0.04780	0.0018	0.05	95.59	
CCV	"		1	1.55805	0	1.558	31	48.30	0.04830	0.0018	0.05	96.60	
002b	"		1	1.208445	0	1.208	31	37.46	0.03746	0.0018			
002b-Duplicate	"		1	1.21742	0	1.217	31	37.74	0.03774	0.0018			-0.740
Digestion Blk	"	"**"	1	0.586559	0	0.587	31	18.18	0.01818	0.0018			
Digestion Blk	"	"**"	1	0.169684	0	0.170	31	5.26	0.00526	0.0018			

"**" Blanks were reported above the MDL. Lab water was used as the blank water but not in the sample digestion process. Procedures have been modified to correct this.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"NR" Data was unavaliable for review.

"H" Analysis digestion performed after holding times have expired.

Recalculated Results for 10% of the Total Nitrogen Sample Set

Analyzed by Florida International University (SERC) for the May 1999 Dry Season (M4)

Entered by gtc 11/30/99, njs 12/3/99 Checked by jtm 1/6/00

Sample	Qualifier Note	Dilution	Rep 1 uM	Rep 2 uM	Rep 3 uM	Rep 4 uM	Rep 5 uM	Rep 6 uM	Average Replicates uM	Blank Correction uM	Corrected Peak uM	Slope	Average Replicate x Slope	Dilution Correction	Sample Results ppm	Detection Limit /3 times (ppm)	SPK CONC (ppm)	R%	RPD
M4-501-SWF		1:10	172485	171475	151729				165230	46842	118388	0.0000029	0.34	10	3.43	0.03/0.09			
Method Blk			48612	38487	28525				38541	46842	-8301	0.0000029	-0.02	1	-0.02	0.03/0.09			
CCV	"NR"		Information was not Provided																
QC CHECK			684703	695739	704954				695132	46842	648290	0.0000029	1.88	1	1.88	0.03/0.09	2	94.00	
M4-508-SWF		1	535115	545591	473656				518121			0.0000029	1.50	1	1.50	0.03/0.09			
Method Blk	"**"		46700	37807	37454				40654			0.0000029	0.12	1	0.12	0.03/0.09			
CCV			714826	692031	704188	721796	702294	707545	707113			0.0000029	2.05	1	2.05	0.03/0.09	2	102.63	
QC CHECK	"NR"		Information was not Provided																
M4-533-SWF		1	532258	571333	577420				560337			0.000002958	1.66	1	1.66	0.03/0.09			
M4-538-SWF		1	707980	740035	712903				720306			0.000002958	2.13	1	2.13	0.03/0.09			
M4-548-SWF	No Sample																		
M4-556-SWF		1	640134	650338	635815				642096			0.000002958	1.90	1	1.90	0.03/0.09			
Method Blk			25908	29682	24182				26591			0.000002958	0.08	1	0.08	0.03/0.09			
Method Blk	"**"		79060	50585	43646				57764			0.000002958	0.17	1	0.17	0.03/0.09			
Method Blk			25672	21609	15354				20878			0.000002958	0.06	1	0.06	0.03/0.09			
QC CHECK			655188	679795	712524				682502			0.000002958	2.02	1	2.02	0.03/0.09	2	100.9	
CCV			667998	662450	630057	635203	639381	674006	653502			0.000002958	1.93	1	1.93	0.03/0.09	2	96.65	
CCV			694053	680057	681458				685189			0.000002958	2.03	1	2.03	0.03/0.09	2	101.3	
UMS			163178	168819	152700				161566			0.000002958	0.48	1	0.48	0.03/0.09	0		
MS			531790	535324	565577				544230			0.000002958	1.61	1	1.61	0.03/0.09	1	113.2	
MSD			517371	515613	525167				519384			0.000002958	1.54	1	1.54	0.03/0.09	1	105.8	4.672
M4-568-SWF		1	562075	544621	540689				549128			0.00000335	1.84	1	1.84	0.03/0.09			
M4-576-SWF		1	492921	471658	509015				491198			0.00000335	1.65	1	1.65	0.03/0.09			
M4-586-SWF		1	796009	823680	798954				806214			0.00000335	2.70	1	2.70	0.03/0.09			
M4-594-SWF		1	781529	821147	789371				797349			0.00000335	2.67	1	2.67	0.03/0.09			
M4-599-SWF		1	712788	700149	706622	727782	689657	733358	711726			0.00000335	2.39	1	2.39	0.03/0.09			
QC-599	"DQO"	1	712788	700149	706622				706520			0.00000335	2.37	1	2.37	0.03/0.09			
QC-599-DUP	"DQO"	1	727782	689657	73358				496932			0.00000335	1.67	1	1.67	0.03/0.09			34.831
Method Blk	"**"		32428	35506	27407				31780			0.00000335	0.11	1	0.11	0.03/0.09			
Method Blk	"**"		38913	28302	41033				36083			0.00000335	0.12	1	0.12	0.03/0.09			
Method Blk	"**"		49809	35709	34505				40008			0.00000335	0.13	1	0.13	0.03/0.09			
QC_CHECK			499954	580180	556717				545617			0.00000335	1.83	1	1.83	0.03/0.09	2	91.5	
QC_CHECK			690152	631733	647739				656541			0.00000335	2.20	1	2.20	0.03/0.09	2	110.1	
CCV			709855	633784	625810	650619	621389	651475	648822			0.00000335	2.18	1	2.18	0.03/0.09	2	108.8	
UMS			335909	400566	357801				364759			0.00000335	1.22	1	1.22	0.03/0.09	0		
MS			618182	643383	596345				619303			0.00000335	2.08	1	2.08	0.03/0.09	1	85.4	
MSD			590507	615465	616454				607475			0.00000335	2.04	1	2.04	0.03/0.09	1	81.4	1.928
M4-566-SWF		1	337987	369404	336643				348011			0.00000344	1.20	1	1.20	0.03/0.09			
M4-809-SWF		1	379303	355621	331947				355624			0.00000344	1.22	1	1.22	0.03/0.09			
M4-872-SWF		1	406587	392109	411261				403319			0.00000344	1.39	1	1.39	0.03/0.09			
Method Blk			31327	21013	28646				26995			0.00000344	0.09	1	0.09	0.03/0.09			
Method Blk	"**"		35654	33785	24202				31214			0.00000344	0.11	1	0.11	0.03/0.09			
Method Blk			27287	22116	15431				21611			0.00000344	0.07	1	0.07	0.03/0.09			
QC_CHECK			522155	532227	497563				517315			0.00000344	1.78	1	1.78	0.03/0.09	2	88.9	
QC_CHECK			643389	578379	638163				619977			0.00000344	2.13	1	2.13	0.03/0.09	2	106.6	
CCV			631208	603723	590386	610675	588524	587068	601931			0.00000344	2.07	1	2.07	0.03/0.09	2	103.5	
UMS			437051	404652	415914				419206			0.00000344	1.44	1	1.44	0.03/0.09	0		
MS	"M"		590787	591598	622457				601614			0.00000344	2.07	1	2.07	0.03/0.09	1	62.7	
MSD	"M"		646740	625091	619866				630566			0.00000344	2.17	1	2.17	0.03/0.09	1	72.7	-4.699

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

"NR" Data was unavailable for review.

"**" Blanks were reported above the MDL. Lab water was used as the blank water but not in the sample digestion process. Procedures have been modified to correct this.

Recalculated Results for 10% of the Total Organic Carbon in Surface Water Sample Set Analyzed by Florida International University (SERC) for the May 1999 Dry Season (M4)

Entered by gtc 11/30/99, njs 12/3/99 Checked by jtm 1/6/00

Sample	Qualifier Note	Dilution	Instrument Result ppm	Blank Factor ppm	Sample Results ppm	Detection Limit ppm
M4-501-SWF	"H"	1	40.02	0	40.02	0.12
M4-508-SWF	"H"	1	22.54	0	22.54	0.12
M4-533-SWF	"H"	1	33.56	0	33.56	0.12
M4-538-SWF	"H"	1	40.72	0	40.72	0.12
M4-548-SWF	NO SAMPLE				NA	
M4-556-SWF	"H"	1	30.59	0	30.59	0.12
M4-566-SWF	"H"	1	21.29	0	21.29	0.12
M4-568-SWF	"H"	1	22.21	0	22.21	0.12
M4-576-SWF	"H"	1	22.73	0	22.73	0.12
M4-586-SWF	"H"	1	30.78	0	30.78	0.12
M4-594-SWF	"H"	1	26.91	0	26.91	0.12
M4-599-SWF	"H"	1	35.08	0	35.08	0.12
M4-809-SWF	"H"	1	22.47	0	22.47	0.12
M4-872-SWF	"H"	1	22.56	0	22.56	0.12

Sample	Qualifier Note	Dilution	Instrument Result ppm	Blank Factor ppm	Sample Results ppm	Detection Limit*3 ppm	Spike Conc (ppm)	R%	RPD
Method Blank		1	1.05	1.061	-0.01	0.36	0		
Method Blank		1	1.116	1.061	0.06	0.36	0		
Method Blank		1	1.231	1.061	0.17	0.36	0		
Method Blank		1	0.993	1.061	-0.07	0.36	0		
Method Blank		1	1.083	1.061	0.02	0.36	0		
Method Blank		1	0.893	1.061	-0.17	0.36	0		
CCV 10		1	10.82	1.061	9.76	0.36	10	97.59	
CCV 5		1	6.077	1.061	5.02	0.36	5	100.32	
CCV 10		1	10.67	1.061	9.61	0.36	10	96.09	
CCV 5		1	6.149	1.061	5.09	0.36	5	101.76	
CCV 10		1	10.74	1.061	9.68	0.36	10	96.79	
UMS		1	7.865	1.061	6.80	0.36			
UMS		1	7.789	1.061	6.73	0.36			1.123
MS		1	13.95	1.061	12.89	0.36	8	76.06	
MS		1	13.96	1.061	12.90	0.36	8	77.14	-0.078
537	*	1				0.36	0		
537D	*	1				0.36	0		
561	*	1				0.36	0		
561D	*	1				0.36	0		
574	*	1				0.36	0		
574D	*	1				0.36	0		
599		1	34.63	0	34.63	0.36	0		
599D		1	35.52	0	35.52	0.36	0		-2.537
894		1	27.81	0	27.81	0.36	0		
894D		1	28.83	0	28.83	0.36	0		-3.602

" * " Unable to verify

"H" Analysis digestion performed after holding times have expired.

10 % Recalculated Results for Total Mercury in Surface Water
Analyzed by Florida International University (SERC) for the May 1999 Dry Season (M4)

Entered by gtc 12/1/99, njs 12/3/99 Checked by jtm 1/6/00

Sample	QC Batch	Qualifier Note	Dilution	Instrument Reading Peak Height Rep 1,2,3	Method Reagant Blank Peak Hieght	Corrected Reading Peak Height Rep 1,2,3	Y ratio	Hg Concentration ppt	Averaged Result ppt	DETECTION LIMIT/*3 ppt	SPK CONC (ppm)	R%	Standard Deviation	Relative Standard Deviation
M4-508-SWF-1	BK13EF1		1	120.10	4.5	115.60	18.6	6.39		0.3/0.9				
	"		1	120.70	4.5	116.20	18.6	6.43		0.3/0.9				
	"		1	115.90	4.5	111.40	18.6	6.16	6.33	0.3/0.9			0.14	2.3
M4-508-SWF-2	"		1	116.90	4.5	112.40	18.6	6.22		0.3/0.9				
	"		1	117.60	4.5	113.10	18.6	6.26		0.3/0.9				
	"		1	119.20	4.5	114.70	18.6	6.34	6.27	0.3/0.9			0.07	1.0
M4-508-SWF-3	"		1	121.20	4.5	116.70	18.6	6.45		0.3/0.9				
	"		1	118.00	4.5	113.50	18.6	6.28		0.3/0.9				
	"		1	122.90	4.5	118.40	18.6	6.55	6.34	0.3/0.9			0.13	2.0
M4-809-SWF-1	"		1	61.10	4.5	56.60	18.6	3.13		0.3/0.9				
	"		1	61.80	4.5	57.30	18.6	3.17		0.3/0.9				
	"		1	60.40	4.5	55.90	18.6	3.09	3.13	0.3/0.9			0.04	1.2
M4-809-SWF-2	"		1	59.60	4.5	55.10	18.6	3.05		0.3/0.9				
	"		1	60.90	4.5	56.40	18.6	3.12		0.3/0.9				
	"		1	60.20	4.5	55.70	18.6	3.08	3.08	0.3/0.9			0.04	1.2
M4-809-SWF-3	"		1	55.10	4.5	50.60	18.6	2.80		0.3/0.9				
	"		1	55.70	4.5	51.20	18.6	2.83		0.3/0.9				
	"		1	57.30	4.5	52.80	18.6	2.92	3.02	0.3/0.9			0.14	4.5
QA-511-SWF-1	"		1	66.10	4.5	61.60	18.6	3.41		0.3/0.9				
	"		1	68.5	4.5	64.00	18.6	3.54		0.3/0.9				
	"		1	67.5	4.5	63.00	18.6	3.48	3.40	0.3/0.9			0.07	2.0
QA-511-SWF-2	"		1	65.90	4.5	61.40	18.6	3.40		0.3/0.9				
	"		1	67.3	4.5	62.80	18.6	3.47		0.3/0.9				
	"		1	66.7	4.5	62.20	18.6	3.44	3.20	0.3/0.9			0.04	1.2
QA-511-SWF-3	"		1	64.70	4.5	60.20	18.6	3.33		0.3/0.9				
	"		1	63.8	4.5	59.30	18.6	3.28		0.3/0.9				
	"		1	65	4.5	60.50	18.6	3.35	3.41	0.3/0.9			0.08	2.4
QA-511-SWF-Matrix Spike	"		1	81.40	4.5	76.90	18.6	4.25		0.3/0.9	1			
	"		1	81	4.5	76.50	18.6	4.23		0.3/0.9	1			
	"		1	79.5	4.5	75.00	18.6	4.15	4.21	0.3/0.9	1	80.02	0.06	1.3
Instrument Blank	"		1	2.59		2.59	18.6	0.14		0.3/0.9				
CCV-1	"		1	89.10	4.5	84.60	18.6	4.68		0.3/0.9	5	93.59		
CCV-2	"		1	90.60	4.5	86.10	18.6	4.76		0.3/0.9	5	95.25		
CCV-3	"		1	88.60	4.5	84.10	18.6	4.65		0.3/0.9	5	93.03		
CCV-4	"		1	89.60	4.5	85.10	18.6	4.71		0.3/0.9	5	94.14		
CCV-5	"		1	86.50	4.5	82.00	18.6	4.54		0.3/0.9	5	90.71		
CCV-6	"		1	91.00	4.5	86.50	18.6	4.78		0.3/0.9	5	95.69		
CCV-7	"		1	88.60	4.5	84.10	18.6	4.65		0.3/0.9	5	93.03		
CCV-8	"		1	85.80	4.5	81.30	18.6	4.50		0.3/0.9	5	89.94		
CCV-9	"		1	90.80	4.5	86.30	18.6	4.77		0.3/0.9	5	95.47		
CCV-10	"		1	89.70	4.5	85.20	18.6	4.71		0.3/0.9	5	94.25		
CCV-11	"		1	84.70	4.5	80.20	18.6	4.44		0.3/0.9	5	88.72		
CCV-12	"		1	89.00	4.5	84.50	18.6	4.67		0.3/0.9	5	93.48		
CCV-13	"		1	84.60	4.5	80.10	18.6	4.43		0.3/0.9	5	88.61		
CCV-14	"		1	88.90	4.5	84.40	18.6	4.67		0.3/0.9	5	93.37		
CCV-15	"		1	96.70	4.5	92.20	18.6	5.10		0.3/0.9	5	102.00		
CCV-16	"		1	87.60	4.5	83.10	18.6	4.60	4.67	0.3/0.9	5	91.93	0.20	4.2

10 % Recalculated Results for Total Mercury in Surface Water
Analyzed by Florida International University (SERC) for the May 1999 Dry Season (M4)

Entered by gtc 12/1/99, njs 12/3/99 Checked by jtm 1/6/00

Sample	QC Batch	Qualifier Note	Dilution	Instrument Reading Peak Height Rep 1,2,3	Method Reagant Blank Peak Hieght	Corrected Reading Peak Height Rep 1,2,3	Y ratio	Hg Concentration ppt	Averaged Result ppt	DETECTION LIMIT/*3 ppt	SPK CONC (ppm)	R%	Standard Deviation	Relative Standard Deviation
M4-533-SWF-1	HG12EF1		1	39.10	9.39	29.71	15.7	1.95		0.3/0.9				
	"		1	41.90	9.39	32.51	15.7	2.13		0.3/0.9				
	"		1	40.30	9.39	30.91	15.7	2.03	2.03	0.3/0.9			0.09	4.5
M4-533-SWF-2	"		1	42.70	9.39	33.31	15.7	2.18		0.3/0.9				
	"		1	40.20	9.39	30.81	15.7	2.02		0.3/0.9				
	"		1	41.30	9.39	31.91	15.7	2.09	2.10	0.3/0.9			0.08	3.9
M4-533-SWF-3	"		1	37.20	9.39	27.81	15.7	1.82		0.3/0.9				
	"		1	39.20	9.39	29.81	15.7	1.95		0.3/0.9				
	"		1	38.20	9.39	28.81	15.7	1.89	2.01	0.3/0.9			0.12	5.8
M4-538-SWF-1	"		1	57.30	9.39	47.91	15.7	3.14		0.3/0.9				
	"		1	58.10	9.39	48.71	15.7	3.19		0.3/0.9				
	"		1	59.80	9.39	50.41	15.7	3.30	3.21	0.3/0.9			0.08	2.6
M4-538-SWF-2	"		1	55.80	9.39	46.41	15.7	3.04		0.3/0.9				
	"		1	54.30	9.39	44.91	15.7	2.94		0.3/0.9				
	"		1	55.10	9.39	45.71	15.7	3.00	2.99	0.3/0.9			0.05	1.6
M4-538-SWF-3	"		1	59.30	9.39	49.91	15.7	3.27		0.3/0.9				
	"		1	55.70	9.39	46.31	15.7	3.03		0.3/0.9				
	"		1	59.80	9.39	50.41	15.7	3.30	3.14	0.3/0.9			0.14	4.4
QA-532-SWF-1	"		1	58.20	9.39	48.81	15.7	3.20		0.3/0.9				
	"		1	60.4	9.39	51.01	15.7	3.34		0.3/0.9				
	"		1	59	9.39	49.61	15.7	3.25	3.07	0.3/0.9			0.07	2.4
QA-532-SWF-2	"		1	60.10	9.39	50.71	15.7	3.32		0.3/0.9				
	"		1	58.8	9.39	49.41	15.7	3.24		0.3/0.9				
	"		1	62.9	9.39	53.51	15.7	3.51	3.21	0.3/0.9			0.14	4.3
QA-532-SWF-3	"		1	60.20	9.39	50.81	15.7	3.33		0.3/0.9				
	"		1	59.8	9.39	50.41	15.7	3.30		0.3/0.9				
	"		1	60.1	9.39	50.71	15.7	3.32	3.31	0.3/0.9			0.09	2.6
QA-532-SWF-Matrix Spike	"		1	77.00	9.39	67.61	15.7	4.43		0.3/0.9	1			
	"		1	76.5	9.39	67.11	15.7	4.40		0.3/0.9	1			
	"		1	78.3	9.39	68.91	15.7	4.52	4.45	0.3/0.9	1	113.51	0.06	1.4
Instrument Blank	"		1	3.07		3.07	15.7	0.20		0.3/0.9				
CCV-1	"		1	72.80	9.39	63.41	15.7	4.16		0.3/0.9	5	83.10		
CCV-2	"		1	73.00	9.39	63.61	15.7	4.17		0.3/0.9	5	83.37		
CCV-3	"		1	72.90	9.39	63.51	15.7	4.16		0.3/0.9	5	83.23		
CCV-4	"		1	74.20	9.39	64.81	15.7	4.25		0.3/0.9	5	84.94		
CCV-5	"		1	68.30	9.39	58.91	15.7	3.86		0.3/0.9	5	77.21		
CCV-6	"		1	70.60	9.39	61.21	15.7	4.01		0.3/0.9	5	80.22		
CCV-7	"		1	78.20	9.39	68.81	15.7	4.51		0.3/0.9	5	90.18		
CCV-8	"		1	72.20	9.39	62.81	15.7	4.12		0.3/0.9	5	82.32		
CCV-9	"		1	72.80	9.39	63.41	15.7	4.16		0.3/0.9	5	83.10		
CCV-10	"		1	69.60	9.39	60.21	15.7	3.95		0.3/0.9	5	78.91		
CCV-11	"		1	77.30	9.39	67.91	15.7	4.45		0.3/0.9	5	89.00		
CCV-12	"		1	75.50	9.39	66.11	15.7	4.33		0.3/0.9	5	86.64		
CCV-13	"		1	80.00	9.39	70.61	15.7	4.63		0.3/0.9	5	92.54		
CCV-14	"		1	76.50	9.39	67.11	15.7	4.40		0.3/0.9	5	87.95		
CCV-15	"		1	76.10	9.39	66.71	15.7	4.37		0.3/0.9	5	87.43		
CCV-16	"		1	74.90	9.39	65.51	15.7	4.29	4.24	0.3/0.9	5	85.86	0.20	4.8

10 % Recalculated Results for Total Mercury in Surface Water
Analyzed by Florida International University (SERC) for the May 1999 Dry Season (M4)

Entered by gtc 12/1/99, njs 12/3/99 Checked by jtm 1/6/00

Sample	QC Batch	Qualifier Note	Dilution	Instrument Reading Peak Height Rep 1,2,3	Method Reagant Blank Peak Hieght	Corrected Reading Peak Height Rep 1,2,3	Y ratio	Hg Concentration ppt	Averaged Result ppt	DETECTION LIMIT/*3 ppt	SPK CONC (ppm)	R%	Standard Deviation	Relative Standard Deviation
M4-548-SWF-1	BK25EF1		1	173.70	3.57	170.13	15.2	11.52		0.3/0.9				
	"		1	177.50	3.57	173.93	15.2	11.77		0.3/0.9				
	"		1	181.00	3.57	177.43	15.2	12.01	11.77	0.3/0.9			0.25	2.1
M4-548-SWF-2	"		1	171.70	3.57	168.13	15.2	11.38		0.3/0.9				
	"		1	171.60	3.57	168.03	15.2	11.37		0.3/0.9				
	"		1	164.90	3.57	161.33	15.2	10.92	11.22	0.3/0.9			0.26	2.4
M4-548-SWF-3	"		1	165.80	3.57	162.23	15.2	10.98		0.3/0.9				
	"		1	162.80	3.57	159.23	15.2	10.78		0.3/0.9				
	"		1	162.40	3.57	158.83	15.2	10.75	11.28	0.3/0.9			0.45	4.0
QA-858-SWF-1	HG24EFI	*	1	31.40	3.57	27.83	11.4	2.51		0.3/0.9				
	"	*	1	31.00	3.57	27.43	11.4	2.48		0.3/0.9				
	"	*	1	31.00	3.57	27.43	11.4	2.48	2.49	0.3/0.9			0.02	0.8
QA-858-SWF-2	"	*	1	32.60	3.57	29.03	11.4	2.62		0.3/0.9				
	"	*	1	33.00	3.57	29.43	11.4	2.66		0.3/0.9				
	"	*	1	34.40	3.57	30.83	11.4	2.78	2.69	0.3/0.9			0.09	3.2
QA-858-SWF-3	"	*	1	32.30	3.57	28.73	11.4	2.59		0.3/0.9				
	"	*	1	33.00	3.57	29.43	11.4	2.66		0.3/0.9				
	"	*	1	34.30	3.57	30.73	11.4	2.77	2.62	0.3/0.9			0.12	4.4
QA-858-SWF-Matrix Spike	"	* "M"	1	107.90	3.57	104.33	11.4	9.42		0.3/0.9	5			
	"	* "M"	1	106.7	3.57	103.13	11.4	9.31		0.3/0.9	5			
	"	* "M"	1	108.6	3.57	105.03	11.4	9.48	9.40	0.3/0.9	5	135.69	0.09	0.9
Instrument Blank	BK25EF1		1	1.37		1.37	15.2	0.09		0.3/0.9				
CCV-1	"		1	73.40	3.57	69.83	15.2	4.73		0.3/0.9	5	94.53		
CCV-2	"		1	74.15	3.57	70.58	15.2	4.78		0.3/0.9	5	95.54		
CCV-3	"		1	73.35	3.57	69.78	15.2	4.72		0.3/0.9	5	94.46		
CCV-4	"		1	70.60	3.57	67.03	15.2	4.54		0.3/0.9	5	90.74		
CCV-5	"		1	72.60	3.57	69.03	15.2	4.67	4.69	0.3/0.9	5	93.44	0.09	2.0

"*" A single digestion batch prepared on 5-34-99 was split into two runs. The matrix spike is reported in File HG24EFI and the sample under review is reported in File BK25EFI. The digestion batch demonstrates the digestion process was performed under certain criteria

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 70 to 130% range.

10 % Recalculated Results for Total Mercury in Surface Water
Analyzed by Florida International University (SERC) for the May 1999 Dry Season (M4)

Entered by gtc 12/1/99, njs 12/3/99 Checked by jtm 1/6/00

Sample	QC Batch	Qualifier Note	Dilution	Instrument Reading Peak Height Rep 1,2,3	Method Reagant Blank Peak Hieght	Corrected Reading Peak Height Rep 1,2,3	Y ratio	Hg Concentration ppt	Averaged Result ppt	DETECTION LIMIT/*3 ppt	SPK CONC (ppm)	R%	Standard Deviation	Relative Standard Deviation
M4-556-SWF-1	HG21EF2		1	51.70	7.76	43.94	19.8	2.28		0.3/0.9				
	"		1	53.60	7.76	45.84	19.8	2.38		0.3/0.9				
	"		1	55.70	7.76	47.94	19.8	2.49	2.39	0.3/0.9			0.10	4.4
M4-556-SWF-2	"		1	53.60	7.76	45.84	19.8	2.38		0.3/0.9				
	"		1	55.80	7.76	48.04	19.8	2.50		0.3/0.9				
	"		1	54.00	7.76	46.24	19.8	2.40	2.43	0.3/0.9			0.06	2.5
M4-556-SWF-3	"		1	53.50	7.76	45.74	19.8	2.38		0.3/0.9				
	"		1	52.40	7.76	44.64	19.8	2.32		0.3/0.9				
	"		1	50.30	7.76	42.54	19.8	2.21	2.37	0.3/0.9			0.09	3.9
M4-556-SWF-Matrix Spike	"		1	143.90	7.76	136.14	19.8	7.07		0.3/0.9	5			
	"		1	150.90	7.76	143.14	19.8	7.44		0.3/0.9	5			
	"		1	145.40	7.76	137.64	19.8	7.15	7.22	0.3/0.9	5	96.99	0.19	2.7
M4-872-SWF-1	"		1	39.00	7.76	31.24	19.8	1.62		0.3/0.9				
	"		1	41.70	7.76	33.94	19.8	1.76		0.3/0.9				
	"		1	38.80	7.76	31.04	19.8	1.61	1.67	0.3/0.9			0.08	5.0
M4-872-SWF-2	"		1	35.60	7.76	27.84	19.8	1.45		0.3/0.9				
	"		1	36.00	7.76	28.24	19.8	1.47		0.3/0.9				
	"		1	34.40	7.76	26.64	19.8	1.38	1.43	0.3/0.9			0.04	3.0
M4-872-SWF-3	"		1	36.40	7.76	28.64	19.8	1.49		0.3/0.9				
	"		1	38.50	7.76	30.74	19.8	1.60		0.3/0.9				
	"		1	37.60	7.76	29.84	19.8	1.55	1.55	0.3/0.9			0.12	7.4
M4-501-SWF-1	"		1	300.70	7.76	292.94	19.8	15.22		0.3/0.9				
	"		1	292.50	7.76	284.74	19.8	14.79		0.3/0.9				
	"		1	290.40	7.76	282.64	19.8	14.69	14.90	0.3/0.9			0.28	1.9
M4-501-SWF-2	"		1	293.40	7.76	285.64	19.8	14.84		0.3/0.9				
	"		1	290.00	7.76	282.24	19.8	14.67		0.3/0.9				
	"		1	297.40	7.76	289.64	19.8	15.05	14.85	0.3/0.9			0.19	1.3
M4-501-SWF-3	"		1	296.60	7.76	288.84	19.8	15.01		0.3/0.9				
	"		1	296.30	7.76	288.54	19.8	14.99		0.3/0.9				
	"		1	296.10	7.76	288.34	19.8	14.98	14.92	0.3/0.9			0.18	1.2
Instrument Blank	"		1	2.46		2.46	19.8	0.13		0.3/0.9				
CCV-1	"		1	106.50	7.76	98.74	19.8	5.13		0.3/0.9	5	102.61		
CCV-2	"		1	106.60	7.76	98.84	19.8	5.14		0.3/0.9	5	102.71		
CCV-3	"		1	109.80	7.76	102.04	19.8	5.30		0.3/0.9	5	106.04		
CCV-4	"		1	111.10	7.76	103.34	19.8	5.37		0.3/0.9	5	107.39		
CCV-5	"		1	105.30	7.76	97.54	19.8	5.07		0.3/0.9	5	101.36		
CCV-6	"		1	105.70	7.76	97.94	19.8	5.09		0.3/0.9	5	101.78		
CCV-7	"		1	108.10	7.76	100.34	19.8	5.21		0.3/0.9	5	104.27		
CCV-8	"		1	103.20	7.76	95.44	19.8	4.96		0.3/0.9	5	99.18		
CCV-9	"		1	110.30	7.76	102.54	19.8	5.33		0.3/0.9	5	106.56		
CCV-10	"		1	106.60	7.76	98.84	19.8	5.14		0.3/0.9	5	102.71		
CCV-11	"		1	103.00	7.76	95.24	19.8	4.95		0.3/0.9	5	98.97		
CCV-12	"		1	106.80	7.76	99.04	19.8	5.15		0.3/0.9	5	102.92		
CCV-13	"		1	107.60	7.76	99.84	19.8	5.19		0.3/0.9	5	103.75		
CCV-14	"		1	102.90	7.76	95.14	19.8	4.94	5.14	0.3/0.9	5	98.87	0.14	2.6

10 % Recalculated Results for Total Mercury in Surface Water

Analyzed by Florida International University (SERC) for the May 1999 Dry Season (M4)

Entered by gtc 12/1/99, njs 12/3/99 Checked by jtm 1/6/00

Sample	QC Batch	Qualifier Note	Dilution	Instrument Reading Peak Height Rep 1,2,3	Method Reagant Blank Peak Hieght	Corrected Reading Peak Height Rep 1,2,3	Y ratio	Hg Concentration ppt	Averaged Result ppt	DETECTION LIMIT/*3 ppt	SPK CONC (ppm)	R%	Standard Deviation	Relative Standard Deviation
M4-566-SWF-1	HG20EF1		1	22.60	2.28	20.32	11.7	1.79		0.3/0.9				
	"		1	24.90	2.28	22.62	11.7	1.99		0.3/0.9				
	"		1	24.40	2.28	22.12	11.7	1.95	1.91	0.3/0.9			0.11	5.6
M4-566-SWF-2	"		1	23.00	2.28	20.72	11.7	1.82		0.3/0.9				
	"		1	22.80	2.28	20.52	11.7	1.80		0.3/0.9				
	"		1	22.50	2.28	20.22	11.7	1.78	1.80	0.3/0.9			0.02	1.2
M4-566-SWF-3	"		1	23.80	2.28	21.52	11.7	1.89		0.3/0.9				
	"		1	23.20	2.28	20.92	11.7	1.84		0.3/0.9				
	"		1	23.70	2.28	21.42	11.7	1.88	1.86	0.3/0.9			0.07	3.9
M4-566-SFW-Matrix Spike	"		1	90.90	2.28	88.62	11.7	7.79		0.3/0.9	5			
	"		1	97.40	2.28	95.12	11.7	8.36		0.3/0.9	5			
	"		1	97.80	2.28	95.52	11.7	8.40	8.19	0.3/0.9	5	126.50	0.34	4.2
Instrument Blank	"		1	3.35		3.35	11.7	0.29		0.3/0.9				
CCV-1	"		1	68.60	2.28	66.32	11.7	5.83		0.3/0.9	5	116.63		
CCV-2	"		1	69.40	2.28	67.12	11.7	5.90		0.3/0.9	5	118.04		
CCV-3	"		1	63.60	2.28	61.32	11.7	5.39		0.3/0.9	5	107.84		
CCV-4	"		1	65.00	2.28	62.72	11.7	5.52		0.3/0.9	5	110.30		
CCV-5	"		1	63.60	2.28	61.32	11.7	5.39		0.3/0.9	5	107.84		
CCV-6	"		1	65.00	2.28	62.72	11.7	5.52		0.3/0.9	5	110.30		
CCV-7	"		1	65.80	2.28	63.52	11.7	5.59		0.3/0.9	5	111.71		
CCV-8	"		1	63.20	2.28	60.92	11.7	5.36		0.3/0.9	5	107.14		
CCV-9	"		1	64.80	2.28	62.52	11.7	5.50		0.3/0.9	5	109.95		
CCV-10	"		1	64.40	2.28	62.12	11.7	5.46	5.54	0.3/0.9	5	110.90	0.18	3.3

10 % Recalculated Results for Total Mercury in Surface Water
Analyzed by Florida International University (SERC) for the May 1999 Dry Season (M4)

Entered by gtc 12/1/99, njs 12/3/99 Checked by jtm 1/6/00

Sample	QC Batch	Qualifier Note	Dilution	Instrument Reading Peak Height Rep 1,2,3	Method Reagant Blank Peak Hieght	Corrected Reading Peak Height Rep 1,2,3	Y ratio	Hg Concentration ppt	Averaged Result ppt	DETECTION LIMIT/*3 ppt	SPK CONC (ppm)	R%	Standard Deviation	Relative Standard Deviation
M4-568-SWF-1	HG11EF2		1	36.4	4.51	31.89	15.6	2.10		0.3/0.9				
	"		1	35.9	4.51	31.39	15.6	2.07		0.3/0.9				
	"		1	35.7	4.51	31.19	15.6	2.06	2.08	0.3/0.9			0.02	1.1
M4-568-SWF-2	"		1	32.7	4.51	28.19	15.6	1.86		0.3/0.9				
	"		1	33.2	4.51	28.69	15.6	1.89		0.3/0.9				
	"		1	29.9	4.51	25.39	15.6	1.67	1.81	0.3/0.9			0.12	6.5
M4-568-SWF-3	"		1	33.2	4.51	28.69	15.6	1.89		0.3/0.9				
	"		1	33	4.51	28.49	15.6	1.88		0.3/0.9				
	"		1	33.5	4.51	28.99	15.6	1.91	1.93	0.3/0.9			0.13	6.9
M4-586-SWF-1	"		1	182.30	4.51	177.79	15.6	11.73		0.3/0.9				
	"		1	182.90	4.51	178.39	15.6	11.76		0.3/0.9				
	"		1	183.40	4.51	178.89	15.6	11.80	11.76	0.3/0.9			0.04	0.3
M4-586-SWF-2	"		1	186.00	4.51	181.49	15.6	11.97		0.3/0.9				
	"		1	185.70	4.51	181.19	15.6	11.95		0.3/0.9				
	"		1	185.90	4.51	181.39	15.6	11.96	11.96	0.3/0.9			0.01	0.1
M4-586-SWF-3	"		1	184.70	4.51	180.19	15.6	11.88		0.3/0.9				
	"		1	180.80	4.51	176.29	15.6	11.63		0.3/0.9				
	"		1	184.10	4.51	179.59	15.6	11.84	11.84	0.3/0.9			0.12	1.0
No Matrix Spike with Batch	"	"M" (NR), "*"												
Instrument Blank	"		1	3.35		3.35	15.6	0.22		0.3/0.9				
CCV-1	"		1	79.4	4.51	74.89	15.6	4.94		0.3/0.9	5	98.78		
CCV-2	"		1	79.5	4.51	74.99	15.6	4.95		0.3/0.9	5	98.91		
CCV-3	"		1	81.7	4.51	77.19	15.6	5.09		0.3/0.9	5	101.81		
CCV-4	"		1	78.7	4.51	74.19	15.6	4.89		0.3/0.9	5	97.85		
CCV-5	"		1	76.7	4.51	72.19	15.6	4.76		0.3/0.9	5	95.22		
CCV-6	"		1	79	4.51	74.49	15.6	4.91		0.3/0.9	5	98.25		
CCV-7	"		1	77.3	4.51	72.79	15.6	4.80		0.3/0.9	5	96.01		
CCV-8	"		1	76.7	4.51	72.19	15.6	4.76		0.3/0.9	5	95.22		
CCV-9	"		1	74.4	4.51	69.89	15.6	4.61		0.3/0.9	5	92.18		
CCV-10	"		1	77.1	4.51	72.59	15.6	4.79		0.3/0.9	5	95.74		
CCV-11	"		1	80.3	4.51	75.79	15.6	5.00		0.3/0.9	5	99.97		
CCV-12	"		1	81.6	4.51	77.09	15.6	5.08		0.3/0.9	5	101.68		
CCV-13	"		1	78.7	4.51	74.19	15.6	4.89		0.3/0.9	5	97.85		
CCV-14	"		1	77.2	4.51	72.69	15.6	4.79		0.3/0.9	5	95.88		
CCV-15	"		1	73.6	4.51	69.09	15.6	4.56		0.3/0.9	5	91.13		
CCV-16	"		1	74.7	4.51	70.19	15.6	4.63	4.85	0.3/0.9	5	92.58	0.16	3.3

" * " Analyst Error, Sample was not spiked by mistake.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 70 to 130% range.

"NR" Data was unavaliable for review.

10 % Recalculated Results for Total Mercury in Surface Water

Analyzed by Florida International University (SERC) for the May 1999 Dry Season (M4)

Entered by gtc 12/1/99, njs 12/3/99 Checked by jtm 1/6/00

Sample	QC Batch	Qualifier Note	Dilution	Instrument Reading Peak Height Rep 1,2,3	Method Reagant Blank Peak Hieght	Corrected Reading Peak Height Rep 1,2,3	Y ratio	Hg Concentration ppt	Averaged Result ppt	DETECTION LIMIT/*3 ppt	SPK CONC (ppm)	R%	Standard Deviation	Relative Standard Deviation
M4-576-SWF-1	HG07EFI		1	54.40	6.78	47.62	17.4	2.82		0.3/0.9				
	"		1	52.1	6.78	45.32	17.4	2.68		0.3/0.9				
	"		1	54.8	6.78	48.02	17.4	2.84	4.14	0.3/0.9			0.09	2.1
M4-576-SWF-2	"		1	46.10	6.78	39.32	17.4	2.32		0.3/0.9				
	"		1	45.5	6.78	38.72	17.4	2.29		0.3/0.9				
	"		1	46.7	6.78	39.92	17.4	2.36	3.25	0.3/0.9			0.04	1.1
M4-576-SWF-3	"		1	65.60	6.78	58.82	17.4	3.48		0.3/0.9				
	"		1	64.3	6.78	57.52	17.4	3.40		0.3/0.9				
	"		1	63.9	6.78	57.12	17.4	3.38	2.84	0.3/0.9			0.48	16.9
M4-576-SWF-Matrix Spike	"		1	75.80	6.78	69.02	17.4	4.08		0.3/0.9	1			
	"		1	75	6.78	68.22	17.4	4.03		0.3/0.9	1			
	"		1	74.2	6.78	67.42	17.4	3.99	4.03	0.3/0.9	1	119.30	0.05	1.2
M4-594-SWF-1	"		1	58.30	6.78	51.52	17.4	3.05		0.3/0.9				
	"		1	60.5	6.78	53.72	17.4	3.18		0.3/0.9				
	"		1	61.6	6.78	54.82	17.4	3.24	3.54	0.3/0.9			0.10	2.8
M4-594-SWF-2	"		1	58.10	6.78	51.32	17.4	3.03		0.3/0.9				
	"		1	57.2	6.78	50.42	17.4	2.98		0.3/0.9				
	"		1	57.1	6.78	50.32	17.4	2.98	3.40	0.3/0.9			0.03	1.0
M4-594-SWF-3	"		1	55.90	6.78	49.12	17.4	2.90		0.3/0.9				
	"		1	55.2	6.78	48.42	17.4	2.86		0.3/0.9				
	"		1	58	6.78	51.22	17.4	3.03	3.03	0.3/0.9			0.12	4.0
Instrument Blank	"		1	0.60		0.60	17.4	0.04		0.3/0.9				
CCV-1	"		1	85.60	6.78	78.82	17.4	4.66		0.3/0.9	5	93.21		
CCV-2	"		1	83.40	6.78	76.62	17.4	4.53		0.3/0.9	5	90.61		
CCV-3	"		1	92.10	6.78	85.32	17.4	5.04		0.3/0.9	5	100.89		
CCV-4	"		1	90.40	6.78	83.62	17.4	4.94		0.3/0.9	5	98.88		
CCV-5	"		1	84.90	6.78	78.12	17.4	4.62		0.3/0.9	5	92.38		
CCV-6	"		1	85.90	6.78	79.12	17.4	4.68		0.3/0.9	5	93.56		
CCV-7	"		1	89.40	6.78	82.62	17.4	4.89		0.3/0.9	5	97.70		
CCV-8	"		1	87.60	6.78	80.82	17.4	4.78		0.3/0.9	5	95.57		
CCV-9	"		1	84.90	6.78	78.12	17.4	4.62		0.3/0.9	5	92.38		
CCV-10	"		1	86.20	6.78	79.42	17.4	4.70	4.75	0.3/0.9	5	93.92	0.16	3.5

10 % Recalculated Results for Total Mercury in Surface Water
Analyzed by Florida International University (SERC) for the May 1999 Dry Season (M4)

Entered by gtc 12/1/99, njs 12/3/99 Checked by jtm 1/6/00

Sample	QC Batch	Qualifier Note	Dilution	Instrument Reading Peak Height Rep 1,2,3	Method Reagant Blank Peak Hieght	Corrected Reading Peak Height Rep 1,2,3	Y ratio	Hg Concentration ppt	Averaged Result ppt	DETECTION LIMIT/*3 ppt	SPK CONC (ppm)	R%	Standard Deviation	Relative Standard Deviation
M4-599-SWF-1	HG10EF1		1	44.00	5.28	38.72	15.0	2.66		0.3/0.9				
	"		1	43.50	5.28	38.22	15.0	2.62		0.3/0.9				
	"		1	42.80	5.28	37.52	15.0	2.57	4.01	0.3/0.9			0.04	1.0
M4-599-SWF-2	"		1	43.80	5.28	38.52	15.0	2.64		0.3/0.9				
	"		1	40.00	5.28	34.72	15.0	2.38		0.3/0.9				
	"		1	44.40	5.28	39.12	15.0	2.68	3.29	0.3/0.9			0.16	5.0
M4-599-SWF-3	"		1	45.20	5.28	39.92	15.0	2.74		0.3/0.9				
	"		1	42.80	5.28	37.52	15.0	2.57		0.3/0.9				
	"		1	43.60	5.28	38.32	15.0	2.63	2.61	0.3/0.9			0.08	3.2
M4-599-SWF-Matrix Spike	"	"M"	1	61.20	5.28	55.92	15.0	3.84		0.3/0.9	1			
	"	"M"	1	63.6	5.28	58.32	15.0	4.00		0.3/0.9	1			
	"	"M"	1	64.1	5.28	58.82	15.0	4.03	3.96	0.3/0.9	1	134.58	0.11	2.7
Instrument Blank	"		1	4.44		4.44	15.0	0.30		0.3/0.9				
CCV-1	"		1	81.50	5.28	76.22	15.0	5.23		0.3/0.9	5	104.55		
CCV-2	"		1	80.90	5.28	75.62	15.0	5.19		0.3/0.9	5	103.73		
CCV-3	"		1	83.50	5.28	78.22	15.0	5.36		0.3/0.9	5	107.30		
CCV-4	"		1	83.70	5.28	78.42	15.0	5.38		0.3/0.9	5	107.57		
CCV-5	"		1	82.80	5.28	77.52	15.0	5.32		0.3/0.9	5	106.34		
CCV-6	"		1	80.10	5.28	74.82	15.0	5.13		0.3/0.9	5	102.63		
CCV-7	"		1	75.10	5.28	69.82	15.0	4.79		0.3/0.9	5	95.77		
CCV-8	"		1	72.50	5.28	67.22	15.0	4.61		0.3/0.9	5	92.21		
CCV-9	"		1	81.60	5.28	76.32	15.0	5.23		0.3/0.9	5	104.69		
CCV-10	"		1	78.10	5.28	72.82	15.0	4.99		0.3/0.9	5	99.89		
CCV-11	"		1	79.50	5.28	74.22	15.0	5.09		0.3/0.9	5	101.81		
CCV-12	"		1	79.50	5.28	74.22	15.0	5.09		0.3/0.9	5	101.81		
CCV-13	"		1	80.90	5.28	75.62	15.0	5.19		0.3/0.9	5	103.73		
CCV-14	"		1	79.90	5.28	74.62	15.0	5.12	5.12	0.3/0.9	5	102.36	0.21	4.1

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 70 to 130% range.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M4-501-SWF Surface Water

Laboratory Records	**Total P**	**Total N**	**TOC**	**Total Hg**	**MeHg**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	5-11-99/No Time	5-11-99/No Time	5-11-99/No Time	5-11-99/No Time	5-11-99/No Time
Digestion Date	5-26-99/1130	8-30-99/1646	No Digestion	05/20/99	
Analysis Date	11/09/99	9-2-99/1623	06/13/99	05/21/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle Lab
Total Volume	10ml	Injection Vial	4 ml	1000 ml	is Main Lab
Sample Volume Analyzed	sample aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1:10	1	1	
Results	0.102	3.43	40.02	14.95	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	Raw Data Initialed	Raw Data Initialed	Raw Data Initialed	JL	JL
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	X	No	No	Yes (FLD)	Yes
All Calculation Checked	X	X	X	X	
Holding Time Met	Yes	Past Holding Time Goal	Past HT	Yes	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	By Run Date/ID	By Run Date/ID	By Run Date/ID	File ID	File ID
Method Blanks	**	-0.02 ppm	No Digestion	X	
Instrument Blanks	NR	NR	Good After Correction	0.128 ppt <MDL	
Duplicates (RPD)	0.718	1 RPD (NR)	-0.634,-0.9 RPD	All Dups <20% RPD	
Matrix Spike Recoveries (75-125)	100%	95% R (NR)	77% R	96.99	
Blank Spike/CCV Recoveries	95%	94%	96-102% R	Single level of std-all good	
Detection Range	0.0006-0.2 ppm	0.03 and >	0.12 ppm and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.997	NA	0.999	0.9988	
QC Report (Verified)					
Data Entry Checked by Another	NA	No	No	Yes	
All Calculation Checked	"	Yes	Yes	Yes	
QC Limits Met	**		"H"		SESD is main lab

Notes

For TOC analysis, only 2 of 5 duplicate samples run were recorded, due to inability to read the instrument printout.

Total Nitrogen analysis: Reported RPD data and Matrix Spike Recoveries could not be reviewed (NR).

SESD Laboratory was switched to be the main laboratory for MeHg. FIU is responsible for 10% of the MeHg.

** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

Station ID M4-501-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	X
Matrix	X	X	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	In Hg Lab Area
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	NA	NA	NA	X	
Sample Run Logs	"	X	X	X	
Instrument Raw Data	"	X	X	X	
Bench Sheets	"	With Raw Printout	With Raw Printout	Notes on Printouts	Notes on Printouts
Sample Preparation Logs	"	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	SFW=SWF	
All Calculation Checked	NA	X	X	X	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	**		"H"		

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

" B " Analyte concentration in the associated blank was >3 times the MDL.

" H " Analysis digestion performed after holding times have expired.

" NR " Data was unavailable for review.

" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

" ** " The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M4-508-SWF Surface Water

Laboratory Records	**Total P**	**Total N**	**TOC**	**Total Hg**	**MeHg**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	5-11-99/No Time	5-11-99/No Time	5-11-99/No Time	5-11-99/No Time	5-11-99/No Time
Digestion Date	5-26-99/1130	5-26, 8-27-99/1220	No Digestion	5-12-99/1800	
Analysis Date	08/06/99	8-30-99/0957	06/13/99	05/13/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle Lab
Total Volume	10ml	Injection Vial	4 ml	1000 ml	is Main Lab
Sample Volume Analyzed	sample aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1	1	1	
Results	0.04	1.504	22.53	6.35	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	Raw Data Initialed	Raw Data Initialed	Raw Data Initialed	JL	JL
Method Detection limit	0.0006ppm	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	No	No	No	Yes (FLD)	Yes
All Calculation Checked	X	X	X	X	
Holding Time Met	Yes	Past Holding Time Goal	Past HT	Yes	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	By Run Date/ID	By Run Date/ID	By Run Date/ID	File ID	File ID
Method Blanks	**	0.12 >MDL	No Digestion	X	
Instrument Blanks	Recalibration Blks Good	NR	Good After Correction	0.14 ppt <MDL	
Duplicates (RPD)	Sample Dups-Good	2 RPD (NR)	-0.634,-0.9 RPD	All Dups <20% RPD	
Matrix Spike Recoveries (75-125)	74%	65% (NR)	77% R	80.02%	
Blank Spike/CCV Recoveries	CCV %R Good	102.1 % R	96-102% R	Single level of std-all good	
Detection Range	0.0006-0.2 ppm	0.03 and >	0.12 ppm and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.9958	NA	0.999	0.9998	
QC Report (Verified)					
Data Entry Checked by Another	No	No	No	Yes	
All Calculation Checked	Yes	Not All Info Given	Yes	Yes	
QC Limits Met	"**", "M"	"**", "M"	"H"		

Notes

For TOC analysis, only 2 of 5 duplicate samples run were recorded, due to inability to read the instrument printout.

Total Nitrogen analysis: Reported RPD data and Matrix Spike Recoveries could not be reviewed (NR).

SESD Laboratory was switched to be the main laboratory for MeHg. FIU is responsible for 10% of the MeHg.

** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

Station ID **M4-508-SWF** **Surface Water**

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	X
Matrix	X	X	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	In Hg Lab Area
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	NA	NA	NA	X	
Sample Run Logs	X	X	X	X	
Instrument Raw Data	X	X	X	X	
Bench Sheets	With Raw Printout	With Raw Printout	With Raw Printout	Notes on Printouts	Notes on Printouts
Sample Preparation Logs	X	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	SFW=SWF	
All Calculation Checked	X	X	X	X	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	"**", "M"	"**", "M"	"H"		

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

" B " Analyte concentration in the associated blank was >3 times the MDL.

" H " Analysis digestion performed after holding times have expired.

" NR " Data was unavailable for review.

" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

" ** " The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M4-533-SWF Surface Water

Laboratory Records	**Total P**	**Total N**	**TOC**	**Total Hg**	**MeHg**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	5-10-99/No Time	5-10-99/No Time	5-10-99/No Time	5-10-99/No Time	5-10-99/No Time
Digestion Date	5-26-99/1130	5-26,8-27-99/1220	No Digestion	05/12/99	
Analysis Date	11/09/99	8-30-99/1338	06/13/99	05/13/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle Lab
Total Volume	10ml	Injection Vial	4 ml	1000 ml	is Main Lab
Sample Volume Analyzed	sample aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1	1	1	
Results	0.129	1.657	33.56	2.01	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	Raw Data Initialed	Raw Data Initialed	Raw Data Initialed	JL	JL
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	No	No	No	Yes (FLD)	Yes
All Calculation Checked	X	X	X	X	
Holding Time Met	Yes	Past Holding Time Goal	Past HT	Yes	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	By Run Date/ID	By Run Date/ID	By Run Date/ID	File ID	File ID
Method Blanks	**	1 of 3 >MDL	No Digestion	X	
Instrument Blanks	Recalibration Blks Good	NR	Good After Correction	0.2 ppt <MDL	
Duplicates (RPD)	Sample Dups-Good	1.7 RPD	-0.634,-0.9 RPD	All Dups <20% RPD	
Matrix Spike Recoveries (75-125)	NR	109.5% Average	77% R	113.51%	
Blank Spike/CCV Recoveries	CCV %R Good	100.9 %R	96-102% R	Single level of std-all good	
Detection Range	0.0006-0.2 ppm	0.03 and >	0.12 ppm and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.998	NA	0.999	0.9998	
QC Report (Verified)					
Data Entry Checked by Another	No	No	No	Yes	
All Calculation Checked	Yes	Yes	Yes	Yes	
QC Limits Met	"**", "M"	"**"	"H"		

Notes

For TOC analysis, only 2 of 5 duplicate samples run were recorded, due to inability to read the instrument printout.

Total Nitrogen analysis: Reported RPD data and Matrix Spike Recoveries could not be reviewed (NR).

SESD Laboratory was switched to be the main laboratory for MeHg. FIU is responsible for 10% of the MeHg.

** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

Station ID M4-533-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	X
Matrix	X	X	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	In Hg Lab Area
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	NA	NA	NA	X	
Sample Run Logs	X	X	X	X	
Instrument Raw Data	X	X	X	X	
Bench Sheets	With Raw Printout	With Raw Printout	With Raw Printout	Notes on Printouts	Notes on Printouts
Sample Preparation Logs	X	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	SFW=SWF	
All Calculation Checked	X	X	X	X	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	"**", "M"	"**"	"H"		

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

" B " Analyte concentration in the associated blank was >3 times the MDL.

" H " Analysis digestion performed after holding times have expired.

" NR " Data was unavailable for review.

" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

" ** " The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M4-538-SWF Surface Water

Laboratory Records	**Total P**	**Total N**	**TOC**	**Total Hg**	**MeHg**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	5-10-99/No Time	5-10-99/No Time	5-10-99/No Time	5-10-99/No Time	5-10-99/No Time
Digestion Date	5-26-99/1130	5-26, 8-27-99/1220	No Digestion	05/12/99	
Analysis Date	08/06/99	8-30-99/1338	06/13/99	05/13/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle Lab
Total Volume	10ml	Injection Vial	4 ml	1000 ml	is Main Lab
Sample Volume Analyzed	aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1	1	1	
Results	0.036	2.131	40.72	3.14	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	Raw Data Initialed	Raw Data Initialed	Raw Data Initialed	JL	JL
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	No	No	No	Yes (FLD)	Yes
All Calculation Checked	X	X	X	X	
Holding Time Met	Yes	Past Holding Time Goal	Past HT	Yes	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	By Run Date/ID	By Run Date/ID	By Run Date/ID	File ID	File ID
Method Blanks	**	1 of 3 >MDL	No Digestion	X	
Instrument Blanks	Recalibration Blks Good	NR	Good After Correction	0.2 ppt <MDL	
Duplicates (RPD)	Sample Dups-Good	1.7 RPD	-0.634,-0.9 RPD	All Dups <20% RPD	
Matrix Spike Recoveries (75-125)	Reported 74% (NR)	109.5% Average	77% R	113.51%	
Blank Spike/CCV Recoveries	CCV %R Good	100.9 %R	96-102% R	Single level of std-all good	
Detection Range	0.0006-0.2 ppm	0.03 and >	0.12 ppm and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.9958	NA	0.999	0.9998	
QC Report (Verified)					
Data Entry Checked by Another	No	No	No	Yes	
All Calculation Checked	Yes	Yes	Yes	Yes	
QC Limits Met	"**", "M"	"**"	"H"		

Notes

For TOC analysis, only 2 of 5 duplicate samples run were recorded, due to inability to read the instrument printout.

Total Nitrogen analysis: Reported RPD data and Matrix Spike Recoveries could not be reviewed (NR).

SESD Laboratory was switched to be the main laboratory for MeHg. FIU is responsible for 10% of the MeHg.

** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

Station ID M4-538-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	X
Matrix	X	X	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	In Hg Lab Area
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	NA	NA	NA	X	
Sample Run Logs	X	X	X	X	
Instrument Raw Data	X	X	X	X	
Bench Sheets	With Raw Printout	With Raw Printout	With Raw Printout	Notes on Printouts	Notes on Printouts
Sample Preparation Logs	X	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	SFW=SWF	
All Calculation Checked	X	X	X	X	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	"**", "M"	"**"	"H"		

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

" B " Analyte concentration in the associated blank was >3 times the MDL.

" H " Analysis digestion performed after holding times have expired.

" NR " Data was unavailable for review.

" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

" ** " The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M4-548-SWF Surface Water

Laboratory Records	**Total P**	**Total N**	**TOC**	**Total Hg**	**MeHg**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	5-9-99/No Time	5-9-99/No Time	5-9-99/No Time	5-9-99/No Time	5-9-99/No Time
Digestion Date	Sample Lost	Sample Lost	Sample Lost	5-23-99/1800	
Analysis Date	Sample Lost	Sample Lost	Sample Lost	05/25/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume				1000 ml	Battelle Lab
Total Volume				1000 ml	is Main Lab
Sample Volume Analyzed				sample aliquot	
Dilution				1	
Results				11.27	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	Raw Data Initialed	Raw Data Initialed	Raw Data Initialed	JL	JL
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another				Yes (FLD)	Yes
All Calculation Checked				X	
Holding Time Met				Yes	28 days
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	By Run Date/ID	By Run Date/ID	By Run Date/ID	File ID	File ID
Method Blanks				X	
Instrument Blanks				0.093 ppt <MDL	
Duplicates (RPD)				All Dups <20% RPD	
Matrix Spike Recoveries (75-125)				135.69%	
Blank Spike/CCV Recoveries				Single level of std-all good	
Detection Range				0.3 ppt and >	
Correlation Coefficient (>0.995)				0.9998	
QC Report (Verified)					
Data Entry Checked by Another				Yes	
All Calculation Checked				Yes	
QC Limits Met				"M"	

Notes

Sample container or containers for total phosphorus, total nitrogen and TOC were misplaced and therefore analysis could not be performed.

Total Hg analysis: No method blanks and no blank spike recoveries were reported.

SESD Laboratory was switched to be the main laboratory for MeHg. FIU is responsible for 10% of the MeHg.

MeHg results from FIU are pending.

Station ID M4-548-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	X
Matrix	X	X	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	In Hg Lab Area
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	NA	NA	NA	X	
Sample Run Logs				X	
Instrument Raw Data				X	
Bench Sheets				Notes on Printouts	Notes on Printouts
Sample Preparation Logs				X	
Raw Data (Verified)					
Sample ID Transferred				SFW=SWF	
All Calculation Checked				X	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers				"M"	

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

" B " Analyte concentration in the associated blank was >3 times the MDL.

" H " Analysis digestion performed after holding times have expired.

" NR " Data was unavailable for review.

" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

" ** " The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M4-556-SWF Surface Water

Laboratory Records

	Total P	Total N	TOC	Total Hg	MeHg
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	5-8-99/No Time	5-8-99/No Time	5-8-99/No Time	5-8-99/No Time	5-8-99/No Time
Digestion Date	5-26-99/1130	5-26, 8-27-99/1220	No Digestion	05/20/99	
Analysis Date	08/06/99	8-30-99/1338	06/13/99	05/21/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle Lab
Total Volume	10ml	Injection Vial	4 ml	1000 ml	is Main Lab
Sample Volume Analyzed	aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1	1	1	
Results	0.011	1.90	30.59	2.38	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	Raw Data Initialed	Raw Data Initialed	Raw Data Initialed	JL	JL
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	No	No	No	Yes (FLD)	Yes
All Calculation Checked	X	X	X	X	
Holding Time Met	Yes	Past Holding Time Goal	Past HT	Yes	28 days
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	By Run Date/ID	By Run Date/ID	By Run Date/ID	File ID	File ID
Method Blanks	**	1 of 3 >MDL	No Digestion	X	
Instrument Blanks	Recalibration Blks Good	NR	Good After Correction	.128 ppt <MDL	
Duplicates (RPD)	Sample Dups-Good	1.7 RPD	-0.634,-0.9 RPD	All Dups <20% RPD	
Matrix Spike Recoveries (75-125)	74%	109.5% Average	77% R	96.99%	
Blank Spike/CCV Recoveries	CCV %R Good	100.9 %R	96-102% R	Single level of std-all good	
Detection Range	0.0006-0.2 ppm	0.03 and >	0.12 ppm and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.9958	NA	0.999	0.9988	
QC Report (Verified)					
Data Entry Checked by Another	No	No	No	Yes	
All Calculation Checked	Yes	Yes	Yes	Yes	
QC Limits Met	"**", "M"	"**"	"H"		

Notes

For TOC analysis, only 2 of 5 duplicate samples run were recorded, due to inability to read the instrument printout.

Total Nitrogen analysis: Reported RPD data and Matrix Spike Recoveries could not be reviewed (NR).

SESD Laboratory was switched to be the main laboratory for MeHg. FIU is responsible for 10% of the MeHg.

** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

Station ID M4-556-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	X
Matrix	X	X	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	In Hg Lab Area
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	NA	NA	NA	X	
Sample Run Logs	X	X	X	X	
Instrument Raw Data	X	X	X	X	
Bench Sheets	With Raw Printout	With Raw Printout	With Raw Printout	Notes on Printouts	Notes on Printouts
Sample Preparation Logs	X	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	SFW=SWF	
All Calculation Checked	X	X	X	X	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	"**", "M"	"**"	"H"		

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

" B " Analyte concentration in the associated blank was >3 times the MDL.

" H " Analysis digestion performed after holding times have expired.

" NR " Data was unavailable for review.

" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

" ** " The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M4-566-SWF Surface Water

Laboratory Records	**Total P**	**Total N**	**TOC**	**Total Hg**	**MeHg**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	5-7-99/No Time	5-7-99/No Time	5-7-99/No Time	5-7-99/No Time	5-7-99/No Time
Digestion Date	5-26-99/1130	5-26-99/1030,Date Not Noted	No Digestion	05/19/99	
Analysis Date	08/06/99	8-4-99/1507	06/13/99	05/20/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle Lab
Total Volume	10ml	Injection Vial	4 ml	1000 ml	is Main Lab
Sample Volume Analyzed	sample aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1	1	1	
Results	0.009	1.197	21.29	1.86	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	Raw Data Initialed	Raw Data Initialed	Raw Data Initialed	JL	JL
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	No	No	No	Yes (FLD)	Yes
All Calculation Checked	X	X	X	X	
Holding Time Met	Yes	Past Holding Time Goal	Past HT	Yes	28 days
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	By Run Date/ID	By Run Date/ID	By Run Date/ID	File ID	File ID
Method Blanks	**	1 of 3 >MDL	No Digestion	X	
Instrument Blanks	Recalibration Blks Good	NR	Good After Correction	0.295 ppt <MDL	
Duplicates (RPD)	Sample Dups-Good	-3.68 RPD	-0.634,-0.9 RPD	All Dups <20% RPD	
Matrix Spike Recoveries (75-125)	74%	62.7, 72.7% R	77% R	126.50%	
Blank Spike/CCV Recoveries	CCV %R Good	88.9%, 106.6%R	96-102% R	Single level of std-all good	
Detection Range	0.0006-0.2 ppm	0.03 and >	0.12 ppm and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.9958	NA	0.999	0.996	
QC Report (Verified)					
Data Entry Checked by Another	No	No	No	Yes	
All Calculation Checked	Yes	Yes	Yes	Yes	
QC Limits Met	"**", "M"	"**", "M"	"H"		

Notes

For TOC analysis, only 2 of 5 duplicate samples run were recorded, due to inability to read the instrument printout.

For Total Nitrogen Analysis, 1 of 3 instrument blanks were above the 3 times MDL limit.

Total Nitrogen analysis: Reported RPD data and Matrix Spike Recoveries could not be reviewed (NR).

SESD Laboratory was switched to be the main laboratory for MeHg. FIU is responsible for 10% of the MeHg.

** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

Station ID M4-566-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	X
Matrix	X	X	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	In Hg Lab Area
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	NA	NA	NA	X	
Sample Run Logs	X	X	X	X	
Instrument Raw Data	X	X	X	X	
Bench Sheets	With Raw Printout	With Raw Printout	With Raw Printout	Notes on Printouts	Notes on Printouts
Sample Preparation Logs	X	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	SFW=SWF	
All Calculation Checked	X	X	X	X	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	"**", "M"	"**", "M"	"H"		

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

" B " Analyte concentration in the associated blank was >3 times the MDL.

" H " Analysis digestion performed after holding times have expired.

" NR " Data was unavailable for review.

" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

" ** " The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M4-568-SWF Surface Water

Laboratory Records	**Total P**	**Total N**	**TOC**	**Total Hg**	**MeHg**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	5-7-99/No Time	5-7-99/No Time	5-7-99/No Time	5-7-99/No Time	5-7-99/No Time
Digestion Date	5-26-99/1130	5-26-99/1030	No Digestion	5-10-99/1800	
Analysis Date	08/06/99	8-4-99/1155	06/13/99	05/11/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle Lab
Total Volume	10ml	Injection Vial	4 ml	1000 ml	is Main Lab
Sample Volume Analyzed	sample aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1	1	1	
Results	0.012	1.842	22.21	1.93	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	Raw Data Initialed	Raw Data Initialed	Raw Data Initialed	JL	JL
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	No	No	No	Yes (FLD)	Yes
All Calculation Checked	X	X	X	X	
Holding Time Met	Yes	Past Holding Time Goal	Past HT	Yes	28 days
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	By Run Date/ID	By Run Date/ID	By Run Date/ID	File ID	File ID
Method Blanks	**	3 of 3 >MDL	No Digestion	X	
Instrument Blanks	Recalibration Blks Good	NR	Good After Correction	0.221 ppt <MDL	
Duplicates (RPD)	Sample Dups-Good	1.2 RPD	-0.634,-0.9 RPD	All Dups <20% RPD	
Matrix Spike Recoveries (75-125)	74%	83.4%R	77% R	No Matrix Spike	
Blank Spike/CCV Recoveries	CCV %R Good	100.8 - 108 %R	96-102% R	Single level of std-all good	
Detection Range	0.0006-0.2 ppm	0.03 and >	0.12 ppm and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.9958	NA	0.999	0.9995	
QC Report (Verified)					
Data Entry Checked by Another	No	No	No	Yes	
All Calculation Checked	Yes	Yes	Yes	Yes	
QC Limits Met	"**", "M"	"**"	"H"	"M (NR)"	

Notes

For TOC analysis, only 2 of 5 duplicate samples run were recorded, due to inability to read the instrument printout.

Total Nitrogen analysis: Reported RPD data and Matrix Spike Recoveries could not be reviewed (NR).

SESD Laboratory was switched to be the main laboratory for MeHg. FIU is responsible for 10% of the MeHg.

** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

Station ID M4-568-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	X
Matrix	X	X	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	In Hg Lab Area
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	NA	NA	NA	X	
Sample Run Logs	X	X	X	X	
Instrument Raw Data	X	X	X	X	
Bench Sheets	With Raw Printout	With Raw Printout	With Raw Printout	Notes on Printouts	Notes on Printouts
Sample Preparation Logs	X	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	SFW=SWF	
All Calculation Checked	X	X	X	X	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	"**", "M"	"**"	"H"	"M (NR)"	

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

" B " Analyte concentration in the associated blank was >3 times the MDL.

" H " Analysis digestion performed after holding times have expired.

" NR " Data was unavailable for review.

" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

" ** " The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M4-576-SWF Surface Water

Laboratory Records

	Total P	Total N	TOC	Total Hg	MeHg
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	5-4-99/No Time	5-4-99/No Time	5-4-99/No Time	5-4-99/No Time	5-4-99/No Time
Digestion Date	5-26-99/1130	5-26-99/1030	No Digestion	5-6-99/1800	
Analysis Date	08/06/99	8-4-99/1155	06/13/99	05/07/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle Lab
Total Volume	10ml	Injection Vial	4 ml	1000 ml	is Main Lab
Sample Volume Analyzed	sample aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1	1	1	
Results	0.008	1.648	22.73	2.84	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	Raw Data Initialed	Raw Data Initialed	Raw Data Initialed	JL	JL
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	No	No	No	Yes (FLD)	Yes
All Calculation Checked	X	X	X	X	
Holding Time Met	Yes	Past Holding Time Goal	Past HT	Yes	28 days
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	By Run Date/ID	By Run Date/ID	By Run Date/ID	File ID	File ID
Method Blanks	**	3 of 3 >MDL	No Digestion	X	
Instrument Blanks	Recalibration Blks Good	NR	Good After Correction	0.03 ppt <MDL	
Duplicates (RPD)	Sample Dups-Good	1.2 RPD	-0.634,-0.9 RPD	All Dups <20% RPD	
Matrix Spike Recoveries (75-125)	74%	83.4%R	77% R	119.30%	
Blank Spike/CCV Recoveries	CCV %R Good	100.8 - 108 %R	96-102% R	Single level of std-all good	
Detection Range	0.0006-0.2 ppm	0.03 and >	0.12 ppm and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.9958	NA	0.999	0.9994	
QC Report (Verified)					
Data Entry Checked by Another	No	No	No	Yes	
All Calculation Checked	Yes	Yes	Yes	Yes	
QC Limits Met	"**", "M"	"**"	"H"		

Notes

For TOC analysis, only 2 of 5 duplicate samples run were recorded, due to inability to read the instrument printout.

Total Nitrogen analysis: Reported RPD data and Matrix Spike Recoveries could not be reviewed (NR).

SESD Laboratory was switched to be the main laboratory for MeHg. FIU is responsible for 10% of the MeHg.

** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

Station ID M4-576-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	X
Matrix	X	X	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	In Hg Lab Area
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	NA	NA	NA	X	
Sample Run Logs	X	X	X	X	
Instrument Raw Data	X	X	X	X	
Bench Sheets	With Raw Printout	With Raw Printout	With Raw Printout	Notes on Printouts	Notes on Printouts
Sample Preparation Logs	X	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	SFW=SWF	
All Calculation Checked	X	X	X	X	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	"**", "M"	"**"	"H"		

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

" B " Analyte concentration in the associated blank was >3 times the MDL.

" H " Analysis digestion performed after holding times have expired.

" NR " Data was unavailable for review.

" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

" ** " The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M4-586-SWF Surface Water

Laboratory Records	**Total P**	**Total N**	**TOC**	**Total Hg**	**MeHg**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	5-6-99/No Time	5-6-99/No Time	5-6-99/No Time	5-6-99/No Time	5-6-99/No Time
Digestion Date	5-26-99/1130	5-26-99/1030	No Digestion	5-10-99/1800	
Analysis Date	08/06/99	8-4-99/1155	06/13/99	05/11/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle Lab
Total Volume	10ml	Injection Vial	4 ml	1000 ml	is Main Lab
Sample Volume Analyzed	sample aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1	1	1	
Results	0.019	2.705	30.78	11.85	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	Raw Data Initialed	Raw Data Initialed	Raw Data Initialed	JL	JL
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	No	No	No	Yes (FLD)	Yes
All Calculation Checked	X	X	X	X	
Holding Time Met	Yes	Past Holding Time Goal	Past HT	Yes	28 days
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	By Run Date/ID	By Run Date/ID	By Run Date/ID	File ID	File ID
Method Blanks	**	3 of 3 >MDL	No Digestion	X	
Instrument Blanks	Recalibration Blks Good	NR	Good After Correction	0.221 ppt <MDL	
Duplicates (RPD)	Sample Dups-Good	1.2 RPD	-0.634,-0.9 RPD	All Dups <20% RPD	
Matrix Spike Recoveries (75-125)	74%	83.4%R	77% R	No Matrix Spike	
Blank Spike/CCV Recoveries	CCV %R Good	100.8 - 108 %R	96-102% R	Single level of std-all good	
Detection Range	0.0006-0.2 ppm	0.03 and >	0.12 ppm and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.9958	NA	0.999	0.9995	
QC Report (Verified)					
Data Entry Checked by Another	No	No	No	Yes	
All Calculation Checked	Yes	Yes	Yes	Yes	
QC Limits Met	"**", "M"	"**"	"H"	"M (NR)"	

Notes

For TOC analysis, only 2 of 5 duplicate samples run were recorded, due to inability to read the instrument printout.

Total Nitrogen analysis: Reported RPD data and Matrix Spike Recoveries could not be reviewed (NR).

SESD Laboratory was switched to be the main laboratory for MeHg. FIU is responsible for 10% of the MeHg.

** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

Station ID M4-586-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	X
Matrix	X	X	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	In Hg Lab Area
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	NA	NA	NA	X	
Sample Run Logs	X	X	X	X	
Instrument Raw Data	X	X	X	X	
Bench Sheets	With Raw Printout	With Raw Printout	With Raw Printout	Notes on Printouts	Notes on Printouts
Sample Preparation Logs	X	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	SFW=SWF	
All Calculation Checked	X	X	X	X	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	"**", "M"	"**"	"H"	"M (NR)"	

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

" B " Analyte concentration in the associated blank was >3 times the MDL.

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" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

" ** " The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M4-594-SWF Surface Water

Laboratory Records

	Total P	Total N	TOC	Total Hg	MeHg
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	5-5-99/No Time	5-5-99/No Time	5-5-99/No Time	5-5-99/No Time	5-5-99/No Time
Digestion Date	5-26-99/1130	5-26-99/1030	No Digestion	5-6-99/1800	
Analysis Date	08/06/99	8-4-99/1155	06/13/99	05/07/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle Lab
Total Volume	10ml	Injection Vial	4 ml	1000 ml	is Main Lab
Sample Volume Analyzed	sample aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1	1	1	
Results	0.018	2.675	26.91	3.02	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	Raw Data Initialed	Raw Data Initialed	Raw Data Initialed	JL	JL
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	No	No	No	Yes (FLD)	Yes
All Calculation Checked	X	X	X	X	
Holding Time Met	Yes	Past Holding Time Goal	Past HT	Yes	28 days
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	By Run Date/ID	By Run Date/ID	By Run Date/ID	File ID	File ID
Method Blanks	**	3 of 3 >MDL	No Digestion	X	
Instrument Blanks	Recalibration Blks Good	NR	Good After Correction	0.03 ppt <MDL	
Duplicates (RPD)	Sample Dups-Good	1.2 RPD	-0.634,-0.9 RPD	All Dups <20% RPD	
Matrix Spike Recoveries (75-125)	74%	83.4%R	77% R	119%	
Blank Spike/CCV Recoveries	CCV %R Good	100.8 - 108 %R	96-102% R	Single level of std-all good	
Detection Range	0.0006-0.2 ppm	0.03 and >	0.12 ppm and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.9958	NA	0.999	0.9994	
QC Report (Verified)					
Data Entry Checked by Another	No	No	No	Yes	
All Calculation Checked	Yes	Yes	Yes	Yes	
QC Limits Met	"**", "M"	"**"	"H"		

Notes

For TOC analysis, only 2 of 5 duplicate samples run were recorded, due to inability to read the instrument printout.

Total Nitrogen analysis: Reported RPD data and Matrix Spike Recoveries could not be reviewed (NR).

SESD Laboratory was switched to be the main laboratory for MeHg. FIU is responsible for 10% of the MeHg.

** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

Station ID M4-594-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	X
Matrix	X	X	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	In Hg Lab Area
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	NA	NA	NA	X	
Sample Run Logs	X	X	X	X	
Instrument Raw Data	X	X	X	X	
Bench Sheets	With Raw Printout	With Raw Printout	With Raw Printout	Notes on Printouts	Notes on Printouts
Sample Preparation Logs	X	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	SFW=SWF	
All Calculation Checked	X	X	X	X	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	"**", "M"	"**"	"H"		

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

" B " Analyte concentration in the associated blank was >3 times the MDL.

" H " Analysis digestion performed after holding times have expired.

" NR " Data was unavailable for review.

" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

" ** " The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M4-599-SWF Surface Water

Laboratory Records

	Total P	Total N	TOC	Total Hg	MeHg
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	5-6-99/No Time	5-6-99/No Time	5-6-99/No Time	5-6-99/No Time	5-6-99/No Time
Digestion Date	5-26-99/1130	5-26-99/1030	No Digestion	5-9-99/1800	
Analysis Date	08/06/99	8-4-99/1155	06/13/99	05/10/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle Lab
Total Volume	10ml	Injection Vial	4 ml	1000 ml	is Main Lab
Sample Volume Analyzed	sample aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1	1	1	
Results	0.017	2.388	35.08	2.64	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	Raw Data Initialed	Raw Data Initialed	Raw Data Initialed	JL	JL
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	No	No	No	Yes (FLD)	Yes
All Calculation Checked	X	X	X	X	
Holding Time Met	Yes	Past Holding Time Goal	Past HT	Yes	28 days
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	By Run Date/ID	By Run Date/ID	By Run Date/ID	File ID	File ID
Method Blanks	**	3 of 3 >MDL	No Digestion	X	
Instrument Blanks	Recalibration Blks Good	NR	Good After Correction	0.3 ppt <MDL	
Duplicates (RPD)	Sample Dups-Good	1.2 RPD	-0.634,-0.9 RPD	All Dups <20% RPD	
Matrix Spike Recoveries (75-125)	74%	83.4%R	77% R	134.58%	
Blank Spike/CCV Recoveries	CCV %R Good	100.8 - 108 %R	96-102% R	Single level of std-all good	
Detection Range	0.0006-0.2 ppm	0.03 and >	0.12 ppm and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.9958	NA	0.999	0.9998	
QC Report (Verified)					
Data Entry Checked by Another	No	No	No	Yes	
All Calculation Checked	Yes	Yes	Yes	Yes	
QC Limits Met	"**", "M"	"**"	"H"	"M"	

Notes

For TOC analysis, only 2 of 5 duplicate samples run were recorded, due to inability to read the instrument printout.

Total Nitrogen analysis: Reported RPD data and Matrix Spike Recoveries could not be reviewed (NR).

SESD Laboratory was switched to be the main laboratory for MeHg. FIU is responsible for 10% of the MeHg.

** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

Station ID M4-599-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	X
Matrix	X	X	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	In Hg Lab Area
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	NA	NA	NA	X	
Sample Run Logs	X	X	X	X	
Instrument Raw Data	X	X	X	X	
Bench Sheets	With Raw Printout	With Raw Printout	With Raw Printout	Notes on Printouts	Notes on Printouts
Sample Preparation Logs	X	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	SFW=SWF	
All Calculation Checked	X	X	X	X	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	"**", "M"	"**"	"H"	"M"	

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

" B " Analyte concentration in the associated blank was >3 times the MDL.

" H " Analysis digestion performed after holding times have expired.

" NR " Data was unavailable for review.

" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

" ** " The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M4-809-SWF Surface Water

Laboratory Records	**Total P**	**Total N**	**TOC**	**Total Hg**	**MeHg**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	5-11-99/No Time	5-11-99/No Time	5-11-99/No Time	5-11-99/No Time	5-11-99/No Time
Digestion Date	5-26-99/1130	5-26-99/1030,Date Not Noted	No Digestion	05/20/99	
Analysis Date	08/06/99	8-4-99/1507	06/13/99	05/21/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle Lab
Total Volume	10ml	Injection Vial	4 ml	1000 ml	is Main Lab
Sample Volume Analyzed	sample aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1	1	1	
Results	0.012	1.223	22.47	3.02	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	Raw Data Initialed	Raw Data Initialed	Raw Data Initialed	JL	JL
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	No	No	No	Yes (FLD)	Yes
All Calculation Checked	X	X	X	X	
Holding Time Met	Yes	Past Holding Time Goal	Past HT	Yes	28 days
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	By Run Date/ID	By Run Date/ID	By Run Date/ID	File ID	File ID
Method Blanks	**	1 of 3 >MDL	No Digestion	X	
Instrument Blanks	Recalibration Blks Good	NR	Good After Correction	0.14 ppt <MDL	
Duplicates (RPD)	Sample Dups-Good	-3.68 RPD	-0.634,-0.9 RPD	All Dups <20% RPD	
Matrix Spike Recoveries (75-125)	74%	62.7, 72.7% R	77% R	80%	
Blank Spike/CCV Recoveries	CCV %R Good	88.9% - 106.6%R	96-102% R	Single level of std-all good	
Detection Range	0.0006-0.2 ppm	0.03 and >	0.12 ppm and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.9958	NA	0.999	0.9998	
QC Report (Verified)					
Data Entry Checked by Another	No	No	No	Yes	
All Calculation Checked	Yes	Yes	Yes	Yes	
QC Limits Met	"**", "M"	"**", "M"	"H"		

Notes

For TOC analysis, only 2 of 5 duplicate samples run were recorded, due to inability to read the instrument printout.

Total Nitrogen analysis: Reported RPD data and Matrix Spike Recoveries could not be reviewed (NR).

SESD Laboratory was switched to be the main laboratory for MeHg. FIU is responsible for 10% of the MeHg.

** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

Station ID M4-809-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	X
Matrix	X	X	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	In Hg Lab Area
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	NA	NA	NA	X	
Sample Run Logs	X	X	X	X	
Instrument Raw Data	X	X	X	X	
Bench Sheets	With Raw Printout	With Raw Printout	With Raw Printout	Notes on Printouts	Notes on Printouts
Sample Preparation Logs	X	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	SFW=SWF	
All Calculation Checked	X	X	X	X	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	"**", "M"	"**", "M"	"H"		

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

" B " Analyte concentration in the associated blank was >3 times the MDL.

" H " Analysis digestion performed after holding times have expired.

" NR " Data was unavailable for review.

" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

" ** " The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M4-872-SWF Surface Water

Laboratory Records	**Total P**	**Total N**	**TOC**	**Total Hg**	**MeHg**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	5-8-99/No Time	5-8-99/No Time	5-8-99/No Time	5-8-99/No Time	5-8-99/No Time
Digestion Date	5-26-99/1130	5-26-99/1030,Date Not Noted	No Digestion	05/20/99	
Analysis Date	08/06/99	8-4-99/1507	06/13/99	05/21/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle Lab
Total Volume	10ml	Injection Vial	4 ml	1000 ml	is Main Lab
Sample Volume Analyzed	sample aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1	1	1	
Results	0.012	1.387	22.56	1.55	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	Raw Data Initialed	Raw Data Initialed	Raw Data Initialed	JL	JL
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	No	No	No	Yes (FLD)	Yes
All Calculation Checked	X	X	X	X	
Holding Time Met	Yes	Past Holding Time Goal	Past HT	Yes	28 days
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	By Run Date/ID	By Run Date/ID	By Run Date/ID	File ID	File ID
Method Blanks	**	1 of 3 >MDL	No Digestion	X	
Instrument Blanks	Recalibration Blks Good	NR	Good After Correction	.128 ppt <MDL	
Duplicates (RPD)	Sample Dups-Good	-3.68 RPD	-0.634,-0.9 RPD	All Dups <20% RPD	
Matrix Spike Recoveries (75-125)	Reported 74% (NR)	62.7, 72.7% R	77% R	93.10%	
Blank Spike/CCV Recoveries	CCV %R Good	88.9% - 106.6%R	96-102% R	Single level of std-all good	
Detection Range	0.0006-0.2 ppm	0.03 and >	0.12 ppm and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.9958	NA	0.999	0.9988	
QC Report (Verified)					
Data Entry Checked by Another	No	No	No	Yes	
All Calculation Checked	Yes	Yes	Yes	Yes	
QC Limits Met	"**", "M"	"**", "M"	"H"		

Notes

For TOC analysis, only 2 of 5 duplicate samples run were recorded, due to inability to read the instrument printout.

For Total Nitrogen Analysis, 1 of 3 instrument blanks were above the 3 times MDL limit.

Total Nitrogen analysis: Reported RPD data and Matrix Spike Recoveries could not be reviewed (NR).

SESD Laboratory was switched to be the main laboratory for MeHg. FIU is responsible for 10% of the MeHg.

** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

Station ID M4-872-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	X
Matrix	X	X	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	In Hg Lab Area
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	NA	NA	NA	X	
Sample Run Logs	X	X	X	X	
Instrument Raw Data	X	X	X	X	
Bench Sheets	With Raw Printout	With Raw Printout	With Raw Printout	Notes on Printouts	Notes on Printouts
Sample Preparation Logs	X	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	SFW=SWF	
All Calculation Checked	X	X	X	X	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	"**", "M"	"**", "M"	"H"		

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

" B " Analyte concentration in the associated blank was >3 times the MDL.

" H " Analysis digestion performed after holding times have expired.

" NR " Data was unavailable for review.

" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

" ** " The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

10 % Recalculated Results for Methylmercury in Surface Water
Analyzed by Battelle Marine Sciences Laboratory for the May 1999 Dry Season (M4)

Entered by mwb 01/07/00, njs 01/10/00

Sampling Station ID	Battelle MSL Code	Data Qualifier Note	QC Batch	Instrument Peak Height	Distilled Sample Volume(ml)	Volume Analyzed (ml)	Distillation Correction Factor	Y intercept	Slope (factor)	Blank Correction Factor	Hg Concentration ppt	DETECTION LIMIT ppt	ICV/CCV CONC	TRUE CONC	%R	Amount Spiked (ppm)	Amount Recovered (ppm)	%R	Relative Percent Difference
M4-500-SWB	1329-90		13290519	12812	49.727	49.727	0.857	187	54.3	0	5.46	0.0233							
M4-538-SWB	1329-100		"	2455	50.463	50.463	0.857	187	54.3	0	0.966	0.0229							
Method Blank	BLK0518991		"	221	51.378	51.378	0.857	187	54.3	0	0.0142	0.0225							
Instrument Blank		NR	"																
M4-506-SWB (QA)	1329-93		"	1042	50.982	50.982	0.857	187	54.3	0	0.360	0.0227							
M4-506-SWB (QA-DUP)	1329-93		"	1117	51.932	51.932	0.857	187	54.3	0	0.385	0.0223							6.56
M4-508-SWB (QA)	1329-94		"	1081	49.494	49.294	0.857	187	54.3	0	0.390	0.0235							
M4-508-SWB (QA MS)	1329-94MS		"	8490	49.419	49.419	0.857	187	54.3	0	3.610	0.0234				3.35	3.221	96	
Std 134 (QA CCV)	13290519		"										38.40	38.95	98.6				
M4-556-SWB	1329-110		13290520	1960	50.948	50.948	0.946	93	56.1	0	0.690	0.0206							
Method Blank	BLK0518991		"	0	50.503	50.503	0.946	93	56.1	0	-0.035	0.0208							
Instrument Blank		NR	"																
M4-553-SWB (QA)	1329-107		"	1085	50.056	50.056	0.946	93	56.1	0	0.373	0.0209							
M4-553-SWB (QA-DUP)	1329-107		"	1103	50.228	50.228	0.946	93	56.1	0	0.379	0.0209							1.46
M4-555-SWB (QA)	1329-109		"	856	50.670	50.670	0.946	93	56.1	0	0.284	0.0207							
M4-555-SWB (QA-MS)	1329-109MS		"	9617	50.277	50.277	0.946	93	56.1	0	3.569	0.0209				3.29	3.286	100	
Std 134 (QA-CCV)	13290520		"	381									40.37	38.95	103.6				
M4-566-SWB	1329-120		13290525	1026	50.407	50.407	0.904	98	52.4	0.0626	0.326	0.0218							
M4-576-SWB	1329-130		"	410	50.089	50.089	0.904	98	52.4	0.0626	0.069	0.0219							
Method Blank	BLK052499		"	247	50.134	50.134	0.904	98	52.4	0.0626	0.000	0.0219							
Instrument Blank		NR	"																
M4-569-SWB (QA)	1329-123		"	1174	50.181	50.181	0.904	98	52.4	0.0626	0.390	0.0219							
M4-569-SWB (QA-DUP)	1329-123		"	1189	50.254	50.254	0.904	98	52.4	0.0626	0.396	0.0218							1.44
M4-566-SWB (QA)	1329-120		"	1026	50.407	50.407	0.904	98	52.4	0.0626	0.326	0.0218							
M4-566-SWB (QA-MS)	1329-120MS		"	9186	50.325	50.325	0.904	98	52.4	0.0626	3.750	0.0218				3.29	3.424	104	
Std 135 (QA-CCV)			"										167	161	103.7				
M4-809-SWB	1329-140		13290604	4475	49.133	49.133	0.936	339	83.4	0	1.078	0.0216							
Method Blank	BLK060499		"	336	49.951	49.951	0.936	339	83.4	0	-0.001	0.0212							
Instrument Blank		NR	"																
M4-577-SWB (QA)	1329-131		"	2006	50.855	50.855	0.936	339	83.4	0	0.420	0.0208							
M4-577-SWB (QA-DUP)	1329-131		"	1881	50.646	50.646	0.936	339	83.4	0	0.390	0.0209							7.38
M4-559-SWB (QA)	1329-113		"	7458	48.831	48.831	0.936	339	83.4	0	1.868	0.0197							
M4-559-SWB (QA-MS)	1329-113MS		"	19818	49.294	49.294	0.936	339	83.4	0	5.062	0.0195				3.35	3.195	95	
Std 135 (QA-CCV)			"										70.9	81	87.5				
M4-901-SWB	1329-149		13290527	3691	50.225	50.225	1	104	46.2	0	1.55	0.0198							
Method Blank	BLK052699		"	123	50.761	50.761	1	104	46.2	0	0.008	0.0195							
Instrument Blank		NR	"																
M4-870-SWB (QA)	1329-145		"	359	50.344	50.344	1	104	46.2	0	0.11	0.0197							
M4-870-SWB (QA-DUP)	1329-145		"	361	50.589	50.589	1	104	46.2	0	0.11	0.0196							0.296
M4-877-SWB (QA)	1329-147		"	1103	50.596	50.596	1	104	46.2	0	0.43	0.0196							
M4-877-SWB (QA-MS)	1329-147MS		"	8483	50.016	50.016	1	104	46.2	0	3.63	0.0198				3.31	3.199	97	
Std 135 (QA-CCV)			"										83.1	82.7	100.5				

"NR" Not Reviewed

May 1999 Samples for Critical Parameters Analyzed by Battelle
With the 10% Full QA/QC Review
Total Methylmercury in Surface Water

	Sampling Station ID			
Laboratory Records	**M4-500-SWB**	**M4-538-SWB**	**M4-556-SWB**	**M4-566-SWB**
Data Report (Attached)				
Laboratory ID Code	1329-90	1329-100	1329-110	1329-120
Sampling Location ID	M4-500-SWB	M4-538-SWB	M4-556-SWB	M4-566-SWB
Sample Type	surface water = SW	surface water = SW	surface water = SW	surface water = SW
Collection Date	05/11/99	05/10/99	05/08/99	05/07/99
Digestion Date	05/18/99	05/18/99	05/19/99	05/24/99
Analysis Date	05/19/99	05/19/99	05/20/99	05/25/99
QC Batch ID	13290519	13290519	13290520	13290525
Digestion Volume	49.727	50.463	50.948	50.407
Total Volume	49.727	50.463	50.948	50.407
Sample Volume Analyzed	aliquot	aliquot	aliquot	aliquot
Dilution	1	1	1	1
Results	5.46	0.966	0.690	0.326
Measuring Unit	ng/L (ppt)	ng/L (ppt)	ng/L (ppt)	ng/L (ppt)
EPA Method	1631/1630	1631/1630	1631/1630	1631/1630
Analyst	Deuth	Deuth	Deuth	Deuth
Method Detection limit	0.0233	0.0229	0.0206	0.0218
Data Report (Verified)				
Data Entry Checked by Another	NR	NR	NR	NR
All Calculation Checked	LIMS	LIMS	LIMS	LIMS
Holding Time Met	Yes (28 days HT)	Yes (28 days HT)	Yes (28 days HT)	Yes (28 days HT)
QC Report (Attached)				
Laboratory ID Code	1329-90	1329-100	1329-110	1329-120
QC Batch ID	13290519	13290519	13290520	13290525
Method Blanks	0.0142	0.0142	-0.035	0.0626 *
Instrument Blanks	NR	NR	NR	NR
Duplicates (RPD)	6.56	6.56	1.46	1.44
Matrix Spike Recoveries (75-125)	96	96	100	104
Blank Spike/CCV Recoveries	98.6	98.6	104	104
Detection Range	0.0233 and >	0.0229 and >	0.0206 and >	0.0218 and >
Correlation Coefficient (>0.995)	0.99964	0.99964	0.99958	0.99953
QC Report (Verified)				
Data Entry Checked by Another	NR	NR	NR	NR
All Calculation Checked	LIMS	LIMS	LIMS	LIMS
QC Limits Met	Yes	Yes	Yes	Yes

Notes

"*" Method blank is above the true MDL, but less than 3 times the MDL.

Sampling Station ID

	M4-500-SWB	M4-538-SWB	M4-556-SWB	M4-566-SWB
Narrative Description (Attached)				
Total Samples/Matrix	76/Waters	76/Waters	76/Waters	76/Waters
Methods/Parameters	X	X	X	X
Range of Samples analyzed	X	X	X	X
Holding Time Summary	X	X	X	X
Analytical Problems	X	X	X	X
QA/QC Acceptance Limits	X	X	X	X
Integrity of Data Quality	X	X	X	X
Deviations From SOP	X	X	X	X
Observations	X	X	X	X
Sample Management Records				
Sampling Location ID	X	X	X	X
Matrix	X	X	X	X
Preservative (Acid, Temp..)	NR	NR	NR	NR
Collection Data/Time	X	X	X	X
Laboratory ID Code	X	X	X	X
Sample Handling/Storage	X	X	X	X
Log-in Procedures	NR	NR	NR	NR
Raw Data (Attached)				
Sample Work Sheets	X	X	X	X
Sample Run Logs	X	X	X	X
Instrument Raw Data	X	X	X	X
Bench Sheets	NA	NA	NA	NA
Sample Preparation Logs	X	X	X	X
Raw Data (Verified)				
Sample ID Transferred	X	X	X	X
All Calculation Checked	X	X	X	X
Measuring Unit	ng/L	ng/L	ng/L	ng/L
PE Results (Attached)				
Organization	NR	NR	NR	NR
Performance (Pass/Fail)	NR	NR	NR	NR
Validation Criteria				
Applied Qualifiers				

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavaliable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by Battelle
With the 10% Full QA/QC Review
Total Methylmercury in Surface Water

	Sampling Station ID		
Laboratory Records	**M4-576-SWB**	**M4-809-SWB**	**M4-901-SWB**
Data Report (Attached)			
Laboratory ID Code	1329-130	1329-140	1329-149
Sampling Location ID	M4-576-SWB	M4-809-SWB	M4-901-SWB
Sample Type	surface water = SW	surface water = SW	surface water = SW
Collection Date	05/04/99	05/11/99	05/06/99
Digestion Date	05/24/99	06/03/99	05/26/99
Analysis Date	05/25/99	06/04/99	05/27/99
QC Batch ID	13290525	13290604	13290527
Digestion Volume	50.089	49.133	50.225
Total Volume	50.089	49.133	50.225
Sample Volume Analyzed	aliquot	aliquot	aliquot
Dilution	1	1	1
Results	0.0688	1.08	1.55
Measuring Unit	ng/L (ppt)	ng/L (ppt)	ng/L (ppt)
EPA Method	1631/1630	1631/1630	1631/1630
Analyst	Deuth	Deuth	Deuth
Method Detection limit	0.0219	0.0216	0.0198
Data Report (Verified)			
Data Entry Checked by Another	NR	NR	NR
All Calculation Checked	LIMS	LIMS	LIMS
Holding Time Met	Yes (28 days HT)	Yes (28 days HT)	Yes (28 days HT)
QC Report (Attached)			
Laboratory ID Code	1329-130	1329-140	1329-149
QC Batch ID	13290525	13290604	13290527
Method Blanks	0.0626 *	-0.0008	0.00799
Instrument Blanks	NR	NR	NR
Duplicates (RPD)	1.44	7.38	0.296
Matrix Spike Recoveries (75-125)	104	95	97
Blank Spike/CCV Recoveries	104	87.5	100.5
Detection Range	0.0219 and >	0.0216 and >	0.0198 and >
Correlation Coefficient (>0.995)	0.99953	0.9993	0.9991
QC Report (Verified)			
Data Entry Checked by Another	NR	NR	NR
All Calculation Checked	LIMS	LIMS	LIMS
QC Limits Met	Yes	Yes	Yes

Notes

"*" Method blank is above the true MDL, but less than 3 times the MDL.

	Sampling Station ID		
	M4-576-SWB	**M4-809-SWB**	**M4-901-SWB**
Narrative Description (Attached)			
Total Samples/Matrix	76/Waters	76/Waters	76/Waters
Methods/Parameters	X	X	X
Range of Samples analyzed	X	X	X
Holding Time Summary	X	X	X
Analytical Problems	X	X	X
QA/QC Acceptance Limits	X	X	X
Integrity of Data Quality	X	X	X
Deviations From SOP	X	X	X
Observations	X	X	X
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	NR	NR	NR
Collection Data/Time	X	X	X
Laboratory ID Code	X	X	X
Sample Handling/Storage	X	X	X
Log-in Procedures	NR	NR	NR
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	NA	NA	NA
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ng/L	ng/L	ng/L
PE Results (Attached)			
Organization	NR	NR	NR
Performance (Pass/Fail)	NR	NR	NR
Validation Criteria			
Applied Qualifiers			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavaliable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

Soil - SERC

10 % Recalculated Results for Total Phosphorus in Soil/Sediment
Analyzed by Florida International University (SERC) for the May 1999 Dry Season (M4)

Entered by njs 12/13/99 Checked by jtm 1/6/00

Sample	QA/QC Batch	Qualifier Note	Dilution Factor	Weight grams	Peak Height um	Slope um	Sample Results ug/g (ppm)	Detection Limit ug/g (ppm)
M4-548-SFF	09-09-99 C-2		1	0.0252	26247	3089.1016	337.2	0.06
M4-556-SFF	"		1	0.0245	6488	3089.1016	85.7	0.06
M4-566-SFF	"		1	0.0249	16474	3089.1016	214.2	0.06
M4-568-SFF	"		1	0.0254	12716	3089.1016	162.1	0.06
M4-576-SFF	"		1	0.0250	19354	3089.1016	250.6	0.06
M4-586-SFF	"		1	0.0255	4965	3089.1016	63.0	0.06

Sample	QA/QC Batch	Qualifier Note	Dilution Factor	Weight grams	Peak Height um	Slope um	Sample Results ug/g (ppm)	Detection Limit*3 ug/g (ppm)	SPK CONC ug/g (ppm)	R%	RPD
Citrus Leaves	09-09-99 C-2		1	0.0254	94650	3089.1016	1206.3	0.18	1300	92.79	
Citrus Leaves	"		1	0.0253	90102	3089.1016	1152.9	0.18	1300	88.68	4.529
CCV	"		1		64169	3089.1016	20.8	0.18	23.23	89.42	
CCV	"		1		67661	3089.1016	21.9	0.18	23.23	94.29	
CCV	"		1		69660	3089.1016	22.6	0.18	23.23	97.07	
CCV	"		1		69487	3089.1016	22.5	0.18	23.23	96.83	
CCV	"		1		69191	3089.1016	22.4	0.18	23.23	96.42	
CCV	"		1		69102	3089.1016	22.4	0.18	23.23	96.30	
M4-551-SFF	"		1	0.0251	21188	3089.1016	273.3	0.18			
M4-551-SFF-D	"		1	0.0250	20312	3089.1016	263.0	0.18			3.823
M4-560-SFF	"		1	0.0254	12005	3089.1016	153.0	0.18			
M4-560-SFF-D	"		1	0.0250	10963	3089.1016	142.0	0.18			7.489
M4-570-SFF	"		1	0.0250	19409	3089.1016	251.3	0.18			
M4-570-SFF-D	"		1	0.0252	19955	3089.1016	256.3	0.18			-1.977
M4-581-SFF	"		1	0.0250	21100	3089.1016	273.2	0.18			
M4-581-SFF-D	"		1	0.0253	18511	3089.1016	236.9	0.18			14.259
Blanks	"		1		-681	3089.1016	-0.2	0.18			
Blanks	"		1		-417	3089.1016	-0.1	0.18			

10 % Recalculated Results for Total Phosphorus in Soil/Sediment
Analyzed by Florida International University (SERC) for the May 1999 Dry Season (M4)

Entered by njs 12/13/99 Checked by jtm 1/6/00

Sample	QA/QC Batch	Qualifier Note	Dilution Factor	Weight grams	Peak Height um	Slope um	Sample Results ug/g (ppm)	Detection Limit ug/g (ppm)
M4-501-SFF	09-09-99 C-1		1	0.0252	11374	2945.1703	153.3	0.06

Sample	QA/QC Batch	Qualifier Note	Dilution Factor	Weight grams	Peak Height um	Slope um	Sample Results ug/g (ppm)	Detection Limit*3 ug/g (ppm)	SPK CONC ug/g (ppm)	R%	RPD
Citrus Leaves	09-09-99 C-1		1	0.0254	93700	2945.1703	1252.6	0.18	1300	96.35	
Citrus Leaves	"		1	0.0253	93689	2945.1703	1257.4	0.18	1300	96.72	-0.383
CCV	"		1		60533	2945.1703	20.6	0.18	23.23	88.48	
CCV	"		1		62707	2945.1703	21.3	0.18	23.23	91.66	-3.528
M4-507-SFF	"		1	0.0249	55239	2945.1703	753.2	0.18			
M4-507-SFF-D	"		1	0.0250	54625	2945.1703	741.9	0.18			1.519
Blanks	"		1		-787	2945.1703	-0.3	0.18			
Blanks	"		1		-503	2945.1703	-0.2	0.18			

10 % Recalculated Results for Total Phosphorus in Soil/Sediment
Analyzed by Florida International University (SERC) for the May 1999 Dry Season (M4)

Entered by njs 12/13/99 Checked by jtm 1/6/00

Sample	QA/QC Batch	Qualifier Note	Dilution Factor	Weight grams	Peak Height um	Slope um	Sample Results ug/g (ppm)	Detection Limit ug/g (ppm)
M4-508-SFF	10-01-99 C-1		1	0.0130	10673	2814.5767	291.7	0.06
M4-533-SFF	"		1	0.0255	16596	2814.5767	231.2	0.06
M4-538-SFF	"		1	0.0252	7493	2814.5767	105.6	0.06

Sample	QA/QC Batch	Qualifier Note	Dilution Factor	Weight grams	Peak Height um	Slope um	Sample Results ug/g (ppm)	Detection Limit*3 ug/g (ppm)	SPK CONC ug/g (ppm)	R%	RPD
Citrus Leaves	10-01-99 C-1		1	0.0247	91976	2814.5767	1323.0	0.18	1300	101.77	
Citrus Leaves	"		1	0.0250	90070	2814.5767	1280.1	0.18	1300	98.47	3.3
CCV	"		1		62994	2814.5767	22.4	0.18	23.23	96.35	
CCV	"		1		65363	2814.5767	23.2	0.18	23.23	99.97	
CCV	"		1		66430	2814.5767	23.6	0.18	23.23	101.60	
CCV	"		1		64795	2814.5767	23.0	0.18	23.23	99.10	
CCV	"		1		66901	2814.5767	23.8	0.18	23.23	102.32	
M4-516-SFF	"		1	0.0250	26975	2814.5767	383.4	0.18			
M4-516-SFF-D	"		1	0.0250	23637	2814.5767	335.9	0.18			13.2
M4-526-SFF	"		1	0.0247	15757	2814.5767	226.7	0.18			
M4-526-SFF-D	"		1	0.0250	18744	2814.5767	266.4	0.18			-16.1
M4-536-SFF	"	"DQO"	1	0.0248	6409	2814.5767	91.8	0.18			
M4-536-SFF-D	"	"DQO"	1	0.0250	12202	2814.5767	173.4	0.18			-61.5
Blanks	"	"B (NR)"	1		No Sample			0.18			
Blanks	"	"B (NR)"	1		No Sample			0.18			

"B" Analyte concentration in the associated blank was >3 times the MDL.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

10 % Recalculated Results for Total Phosphorus in Soil/Sediment
Analyzed by Florida International University (SERC) for the May 1999 Dry Season (M4)

Entered by njs 12/13/99 Checked by jtm 1/6/00

Sample	QA/QC Batch	Qualifier Note	Dilution Factor	Weight grams	Peak Height um	Slope um	Sample Results ug/g (ppm)	Detection Limit ug/g (ppm)
M4-594-SFF	09-09-99 C-1	*	1	0.03	24063	2877.0205	330.6	0.06

Sample	QA/QC Batch	Qualifier Note	Dilution Factor	Weight grams	Peak Height um	Slope um	Sample Results ug/g (ppm)	Detection Limit*3 ug/g (ppm)	SPK CONC ug/g (ppm)	R%	RPD
Citrus Leaves	09-09-99 C-1		1	0.0254	89928	2877.0205	1230.6	0.18	1300	94.66	
Citrus Leaves	"		1	0.0253	90901	2877.0205	1248.8	0.18	1300	96.06	-1.471
CCV	"		1		64019	2877.0205	22.3	0.18	23.23	95.79	
CCV	"		1		65889	2877.0205	22.9	0.18	23.23	98.59	
CCV	"		1		67199	2877.0205	23.4	0.18	23.23	100.55	
CCV	"		1		62762	2877.0205	21.8	0.18	23.23	93.91	
CCV	"	"DQO"	1		42037	2877.0205	14.6	0.18	23.23	62.90	
CCV	"		1		65202	2877.0205	22.7	0.18	23.23	97.56	
CCV	"		1		59803	2877.0205	20.8	0.18	23.23	89.48	
M4-538-FCF	"	"DQO"	1	0.0248	20884	2877.0205	292.7	0.18			
M4-538-FCF-D	"	"DQO"	1	0.0251	17165	2877.0205	237.7	0.18			20.739
M4-564-FCF	"		1	0.0254	14708	2877.0205	201.3	0.18			
M4-564-FCF-D	"		1	0.0252	14921	2877.0205	205.8	0.18			-2.228
M4-576-FCF	"		1	0.0259	51821	2877.0205	695.4	0.18			
M4-576-FCF-D	"		1	0.0258	52692	2877.0205	709.9	0.18			-2.054
M4-602-FCF	"		1	0.0254	12344	2877.0205	168.9	0.18			
M4-602-FCF-D	"		1	0.0251	12962	2877.0205	179.5	0.18			-6.071
Blanks	"		1		-892	2877.0205	-0.3	0.18			
Blanks	"		1		-1093	2877.0205	-0.4	0.18			

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

* Sample M4-594-SFF was re-run with a set a floc samples and the associated QA/QC. The 10% QA/QC associated with the soil sample met the DQO limits.

10 % Recalculated Results for Total Phosphorus in Soil/Sediment
Analyzed by Florida International University (SERC) for the May 1999 Dry Season (M4)

Entered by njs 12/13/99 Checked by jtm 1/6/00

Sample	QA/QC Batch	Qualifier Note	Dilution Factor	Weight grams	Peak Height um	Slope um	Sample Results ug/g (ppm)	Detection Limit ug/g (ppm)
M4-599-SFF	10-29-99 C1		1	0.0256	12575	2781.3245	176.6	0.06
M4-809-SFF	"		1	0.0249	12430	2781.3245	179.5	0.06
M4-872-SFF	"		1	0.0245	10557	2781.3245	154.9	0.06

Sample	QA/QC Batch	Qualifier Note	Dilution Factor	Weight grams	Peak Height um	Slope um	Sample Results ug/g (ppm)	Detection Limit*3 ug/g (ppm)	SPK CONC ug/g (ppm)	R%	RPD
Citrus Leaves	10-29-99 C1		1	0.0252	78667	2781.3245	1122.4	0.18	1300	86.34	
Citrus Leaves	"		1	0.0254	83527	2781.3245	1182.3	0.18	1300	90.95	-5.203
CCV	"		1		60224	2781.3245	21.7	0.18	23.23	93.21	
CCV	"		1		64529	2781.3245	23.2	0.18	23.23	99.87	
CCV	"		1		63127	2781.3245	22.7	0.18	23.23	97.70	
CCV	"		1		64203	2781.3245	23.1	0.18	23.23	99.37	
CCV	"		1		65533	2781.3245	23.6	0.18	23.23	101.43	
CCV	"		1		64460	2781.3245	23.2	0.18	23.23	99.77	
M4-607-SFF	"		1	0.0254	4084	2781.3245	57.8	0.18			
M4-607-SFF-D	"		1	0.0253	3798	2781.3245	54.0	0.18			6.863
M4-619-SFF	"		1	0.0251	7527	2781.3245	107.8	0.18			
M4-619-SFF-D	"		1	0.0251	8136	2781.3245	116.5	0.18			-7.776
M4-868-SFF	"		1	0.0248	27687	2781.3245	401.4	0.18			
M4-868-SFF-D	"		1	0.0250	27915	2781.3245	401.5	0.18			-0.017
Blanks	"		1		3	2781.3245	0.0	0.18			
Blanks	"		1		-268	2781.3245	-0.1	0.18			

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

10 % Recalculated Results for Ash Free Dry Weight in Soil/Sediment
Analyzed by Florida International University (SERC) for the May 1999 Dry Season (M4)

Entered by njs 12/16/99 Checked by jtm 01/06/00

Sample	Qualifier Note	Cup Wt (g)	Sample Wt (g)	Dried Cup + Ashed Sed	Ashed Sample (g)	Mineral Content Fraction	% Mineral Content	Ash Free Dry Weight	% Ash Free Dry Weight Content	RPD %
M4-501-SFF		7.1272	0.0255	7.1282	0.001	0.039	3.92	0.9608	96.08	
M4-508-SFF		7.1693	0.0244	7.17	0.0007	0.029	2.87	0.9713	97.13	
QA-M4-518-SFF		7.2443	0.0256	7.2481	0.0038	0.148	14.84	0.8516	85.16	
QA-M4-518-SFF-DUP		7.131	0.0255	7.1345	0.0035	0.137	13.73	0.8627	86.27	-1.3
QA-M4-528-SFF		7.1888	0.0252	7.1917	0.0029	0.115	11.51	0.8849	88.49	
QA-M4-528-SFF-DUP		7.1932	0.0255	7.196	0.0028	0.110	10.98	0.8902	89.02	-0.6
M4-533-SFF		7.1744	0.0257	7.1763	0.0019	0.074	7.39	0.9261	92.61	
M4-538-SFF		7.1222	0.0248	7.1355	0.0133	0.536	53.63	0.4637	46.37	
QA-M4-538-SFF-DUP		7.1338	0.0247	7.1462	0.0124	0.502	50.20	0.4980	49.80	-7.1
M4-548-SFF		7.1673	0.0252	7.1697	0.0024	0.095	9.52	0.9048	90.48	
QA-M4-548-SFF-DUP		7.2232	0.025	7.2254	0.0022	0.088	8.80	0.9120	91.20	-0.8
M4-556-SFF		7.1818	0.0254	7.1956	0.0138	0.543	54.33	0.4567	45.67	
QA-M4-558-SFF		7.2129	0.0256	7.2149	0.002	0.078	7.81	0.9219	92.19	
QA-M4-558-SFF-DUP		7.1344	0.025	7.137	0.0026	0.104	10.40	0.8960	89.60	2.8
M4-566-SFF		7.2141	0.0255	7.2176	0.0035	0.137	13.73	0.8627	86.27	
M4-568-SFF		7.1246	0.0254	7.1289	0.0043	0.169	16.93	0.8307	83.07	
QA-M4-568-SFF-DUP		7.1312	0.0254	7.1338	0.0026	0.102	10.24	0.8976	89.76	-7.7
M4-576-SFF		7.1551	0.0251	7.1622	0.0071	0.283	28.29	0.7171	71.71	
QA-M4-578-SFF		7.1189	0.025	7.1365	0.0176	0.704	70.40	0.2960	29.60	
QA-M4-578-SFF-DUP		7.1781	0.0251	7.1954	0.0173	0.689	68.92	0.3108	31.08	-4.9
M4-586-SFF		7.299	0.0254	7.319	0.02	0.787	78.74	0.2126	21.26	
QA-M4-590-SFF	"DQO"	7.3441	0.0256	7.3631	0.019	0.742	74.22	0.2578	25.78	
QA-M4-590-SFF-DUP	"DQO"	7.1493	0.0257	7.1696	0.0203	0.790	78.99	0.2101	21.01	20.4
M4-594-SFF		7.293	0.0255	7.3011	0.0081	0.318	31.76	0.6824	68.24	
M4-599-SFF		7.1615	0.0251	7.1637	0.0022	0.088	8.76	0.9124	91.24	
QA-M4-601-SFF		7.2509	0.0251	7.2544	0.0035	0.139	13.94	0.8606	86.06	
QA-M4-601-SFF-DUP		7.3239	0.0253	7.3283	0.0044	0.174	17.39	0.8261	82.61	4.1
QA-M4-614-SFF		7.1889	0.0254	7.2096	0.0207	0.815	81.50	0.1850	18.50	
QA-M4-614-SFF-DUP		7.1765	0.025	7.1968	0.0203	0.812	81.20	0.1880	18.80	-1.6
M4-809-SFF		7.202	0.0253	7.2029	0.0009	0.036	3.56	0.9644	96.44	
QA-M4-811-SFF		7.2803	0.0252	7.2817	0.0014	0.056	5.56	0.9444	94.44	
QA-M4-811-SFF-DUP		7.264	0.0253	7.2648	0.0008	0.032	3.16	0.9684	96.84	-2.5
M4-872-SFF		7.2362	0.025	7.2455	0.0093	0.372	37.20	0.6280	62.80	
QA-M4-901-SFF		7.2878	0.0253	7.2914	0.0036	0.142	14.23	0.8577	85.77	
QA-M4-901-SFF-DUP		7.2703	0.0256	7.2746	0.0043	0.168	16.80	0.8320	83.20	3.0

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

* Ash Free Dry Weight analyses were reanalyzed if the calculated dry weight had negative weight value. These reanalyses were performed on an individual basis only.

10 % Recalculated Results for Bulk Density in Soil/Sediment
Analyzed by Florida International University (SERC) for the May 1999 Dry Season (M4)

Entered by njs 12/16/99 Checked by jtm 01/06/00

Sample	QA/QC Batch	Qualifier Note	Cup Wt (g)	Sample Volume (mL)	Cup + Wet Sediment (g)	Sample Wet Weight (g)	Vol/Weight Ratio	Dried Cup + Dried Sed (g)	Bulk Density g/mL	EPA Prep Dilution	Corrected Bulk Density g/mL
M4-501-SFF	09-09-99 C-2	*	10.52172	80	90.6780	80.1563	0.9981	14.8775	0.0544	0.40	0.14
M4-508-SFF	"	*	10.52172	60	85.1097	74.5880	0.8044	14.2825	0.0627	0.25	0.25
M4-533-SFF	"	*	10.52172	80	92.6565	82.1348	0.9740	18.6297	0.1013	0.50	0.20
M4-538-SFF	"	*	10.52172	80	93.0177	82.4960	0.9697	26.2454	0.1965	0.40	0.49
M4-548-SFF	"	*	10.52172	70	77.1153	66.5936	1.0512	15.4151	0.0699	0.67	0.10
M4-556-SFF	"	*	10.52172	70	91.0047	80.4830	0.8697	24.4034	0.1983	1.00	0.20
M4-566-SFF	"	*	10.52172	60	81.8973	71.3756	0.8406	19.7691	0.1541	0.50	0.31
M4-568-SFF	"	*	10.52172	60	77.0048	66.4831	0.9025	16.7999	0.1046	0.33	0.32
M4-576-SFF	"	*	10.52172	90	110.6919	100.1702	0.8985	22.6543	0.1348	1.00	0.13
M4-586-SFF	"	*	10.52172	80	100.647	90.1253	0.8877	47.0813	0.4570	1.00	0.46
M4-594-SFF	"	*	10.52172	60	98.4345	87.9128	0.6825	22.1843	0.1944	0.67	0.29
M4-599-SFF	"	*	10.52172	40	88.1801	77.6584	0.5151	17.9186	0.1849	0.40	0.46
M4-809-SFF	"	*	10.52172	60	78.9859	68.4642	0.8764	14.7799	0.0710	0.33	0.22
M4-872-SFF	"	*	10.52172	70	92.1029	81.5812	0.8580	21.6815	0.1594	1.00	0.16

* There are no duplicates/replicates reported with this parameter set.

10 % Recalculated Results for Methylmercury in Soil Samples Analyzed by Florida International University Laboratory for the May 1999 Dry Season (M4)

Entered by mwb 02/02/00 Checked By: njs 04-12-00

Sampling Station ID	Data Qualifier Note	QC Batch	Wet Sample Weight (g)	Dry/Wet Weight Ratio	First Extraction Volume(ml)	Back Extraction Volume(ml)	Final Extraction Volume(ul)	Spiked Concentration (ng/g)	MeHg Peak Area	Y intercept	Slope	MeHg Concentration (ng/g)	RPD	Average MeHg Conc. (ng/g)	Standard Deviation	%R	Final Result ng/g
M4-501-SDF-A	"H"	32000a	4.650	0.028	4	0.6	200		4.63	0.12	1.730	8.56					
M4-501-SDF-B	"H"	32000a	4.046	0.028	4	0.6	200		1.54	0.12	1.730	3.27	89.38	5.92			
M4-501-SDF-C	"H"	32000a	5.910	0.028	3.8	0.6	200	4.530	6.99	0.12	1.730	10.71					
M4-501-SDF-D	"H"	32000a	6.463	0.028	4	0.6	200	4.140	8.76	0.12	1.730	11.66	-8.49	11.18		121.44	4.87
Correlation Coefficient	"NR"	32000a															
Blank-1		32000a			4.8	0.6	200		0.00								0
Blank-2		32000a			3.2	0.6	200		0.00								0
ccv		32000a				0.6	200	3.75	4.670	0.228	1.296	3.60				96.09	
M4-533-SDF-A	"H"	032400b	4.602	0.129	4	0.6	200		0.00	-0.049	2.360	0.00					
M4-533-SDF-B	"H"	032400b	4.753	0.129	4	0.6	200		0.00	-0.049	2.360	0.00	0.00	0.00			
M4-533-SDF-C	"H"	032400b	4.697	0.129	3.8	0.6	200	1.800	2.86	-0.049	2.360	0.88					
M4-533-SDF-D	"H", "M"	032400b	4.246	0.129	4	0.6	200	1.990	2.86	-0.049	2.360	0.92	-4.96	0.90		47.47	0.00
Correlation Coefficient	"NR"	032400b															
Blank-1		032400b			4	0.6	200		0.00								0
Blank-2		032400b			4	0.6	200		0.00								0
ccv		32000a				0.6	200	2.5	5.270	-0.049	2.360	2.23				89.32	
ccv		32000a				0.6	200	2.5	4.960	-0.049	2.360	2.10				84.07	
M4-548-SDF-A	"H"	80699	4.475	0.0745	3.6	0.8	200		1.52	-0.072	1.821	0.87					
M4-548-SDF-B	"H"	80699	4.095	0.0745	3.6	0.8	200		0.86	-0.072	1.821	0.54	47.17	0.70			
M4-548-SDF-C	"H"	80699	4.073	0.0745	2.2	0.8	200	3.30	4.65	-0.072	1.821	4.78					
M4-548-SDF-D	"H"	80699	4.088	0.0745	3.0	0.8	200	3.28	4.56	-0.072	1.821	3.43	33.03	4.10		103.35	0.68
M4-556-SDF-A	"H"	80699	4.190	0.171	3.4	0.8	200		1.27	-0.072	1.821	0.36					
M4-556-SDF-B	"H"	80699	4.153	0.171	3.0	0.8	200		0.60	-0.072	1.821	0.19	59.71	0.28			
M4-556-SDF-C	"H"	80699	4.467	0.171	2.9	0.8	200	1.31	3.06	-0.072	1.821	0.95					
M4-556-SDF-D	"H", "M"	80699	4.718	0.171	5.0	0.8	200	1.24	4.23	-0.072	1.821	0.72	27.39	0.83		43.80	0.63
Correlation Coefficient	"NR"	80699															
Blank-1		80699			5.0	0.8	200		0.00	-0.072	1.821						
Blank-2		80699			3.3	0.8	200		0.00	-0.072	1.821						
ccv		80699						3.75	7.10	-0.072	1.821	3.90				103.97	
ccv		80699						3.75	2.42	-0.072	1.821	1.33				35.44	
ccv		80699						3.75	9.65	-0.072	1.821	5.30				141.31	

Data Qualifiers

"H" Analysis digestion performed after holding times have expired.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 70 to 130% range.

"NR" Not Reviewed.

10 % Recalculated Results for Methylmercury in Soil Samples Analyzed by Florida International University Laboratory for the May 1999 Dry Season (M4)

Entered by mwb 02/02/00

Sampling Station ID	Data Qualifier Note	QC Batch	Wet Sample Weight (g)	Dry/Wet Weight Ratio	First Extraction Volume(ml)	Back Extraction Volume(ml)	Final Extraction Volume(ul)	Spiked Concentration (ng/g)	MeHg Peak Area	Y intercept	Slope	MeHg Concentration (ng/g)	RPD	Average MeHg Conc. (ng/g)	Standard Deviation	%R	Final Result ng/g
M4-566-SDF-A	"H"	81299	5.557	0.13	3.2	0.8	200		4.58	1.011	1.94	1.28					
M4-566-SDF-B	"H"	81299	5.435	0.13	3.4	0.8	200		2.00	1.011	1.94	0.54	81.65	0.91			
M4-566-SDF-C	"H"	81299	5.400	0.13	3.2	0.8	200	1.07	7.74	1.011	1.94	2.22					
M4-566-SDF-D	"H"	81299	5.156	0.13	3.2	0.8	200	1.12	6.53	1.011	1.94	1.96	12.36	2.09		108.16	0.84
Correlation Coefficient	"NR"	81299															
Blank-1		81299			4	0.8	200		0.00								0
Blank-2		81299			3.8	0.8	200		0.00								0
ccv	"DQO"	81299					200	2.5	7.750	1.011	1.94	3.99				159.79	
ccv	"DQO"	81299					200	2.5	7.640	1.011	1.94	3.94				157.53	
M4-568-SDF-A	"H"	121499	4.181	0.0727	3.6	0.8	200		0.00	0.42	2.13	0.00					
M4-568-SDF-B	"H"	121499	3.423	0.0727	3.0	0.8	200		0.00	0.42	2.13	0.00	0.00	0.00			
M4-568-SDF-C	"H"	121499	5.350	0.0727	3.0	0.8	200	1.93	5.11	0.42	2.13	2.57					
M4-568-SDF-D	"H"	121499	4.482	0.0727	3.0	0.8	200	2.30	4.48	0.42	2.13	2.69	-4.54	2.63		124.34	0.00
Correlation Coefficient	"NR"	121499															
Blank-1		121499			4.0	0.8	200		0.00	0.42	2.13						0
Blank-2		121499			4.0	0.8	200		0.00	0.42	2.13						0
ccv	"NR"	121499															
M4-576-SDF-A	"H"	32600	4.624	0.111	4.0	0.6	200		4.92	0.18	1.85	2.16					
M4-576-SDF-B	"H"	32600	4.560	0.111	3.6	0.6	200		3.91	0.18	1.85	1.93	11.04	2.05			
M4-576-SDF-C	"H"	32600	4.240	0.111	4.0	0.6	200	1.59	8.30	0.18	1.85	3.97					
M4-576-SDF-D	"H"	32600	6.438	0.111	3.2	0.6	200	1.05	7.71	0.18	1.85	3.04	26.67	3.50		110.50	1.85
Correlation Coefficient	"NR"	32600															
Blank-1		32600			4.0	0.6	200		0.00	0.18	1.85						0
Blank-2		32600			4.0	0.6	200		0.00	0.18	1.85						0
ccv		32600						2.5	4.95	0.18	1.85	2.68				107.03	
ccv		32600						2.5	4.03	0.18	1.85	2.18				87.14	
M4-586-SFF-A	"H"	82099	4.302	0.327	1.6	0.8	200		0.00	-0.436	1.430	0.00					
M4-586-SFF-B	"H"	82099	4.217	0.327	2.4	0.8	200		0.00	-0.436	1.430	0.00	0.00	0.00			
M4-586-SFF-C	"H"	82099	4.263	0.327	2.2	0.8	200	0.54	0.50	-0.436	1.430	0.14					
M4-586-SFF-D	"H", "M"	82099	5.321	0.327	2.0	0.8	200	0.43	0.52	-0.436	1.430	0.13	8.71	0.14		28.16	0.00
Correlation Coefficient	"NR"	82099															
Blank-1		82099			4.0	0.8	200		0	-0.436	1.430						0
Blank-2		82099			4.0	0.8	200		0	-0.436	1.430						0
ccv		82099						2.5	2.91	-0.436	1.430	2.03				81.40	

Data Qualifiers

"H" Analysis digestion performed after holding times have expired.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 70 to 130% range.

"NR" Not Reviewed.

10 % Recalculated Results for Methylmercury in Soil Samples Analyzed by Florida International University Laboratory for the May 1999 Dry Season (M4)

Entered by mwb 02/02/00

Sampling Station ID	Data Qualifier Note	QC Batch	Wet Sample Weight (g)	Dry/Wet Weight Ratio	First Extraction Volume(ml)	Back Extraction Volume(ml)	Final Extraction Volume(ul)	Spiked Concentration (ng/g)	MeHg Peak Area	Y intercept	Slope	MeHg Concentration (ng/g)	RPD	Average MeHg Conc. (ng/g)	Standard Deviation	%R	Final Result ng/g
M4-594-SFF-A	"H"	121599	4.296	0.105	3.2	0.8	200		2.49	0.39	2.09	1.03					
M4-594-SFF-B	"H"	121599	4.078	0.105	3.6	0.8	200		3.94	0.39	2.09	1.53	-38.82	1.28			
M4-594-SFF-C	"H"	121599	4.985	0.105	3.4	0.8	200	1.430	7.09	0.39	2.09	2.38					
M4-594-SFF-D	"H"	121599	4.492	0.105	3.2	0.6	200	1.590	4.96	0.39	2.09	2.62	-9.51	2.50		80.89	1.58
Correlation Coefficient	"NR"	121599															
Blank-1		121599			4.0	0.8	200		0.00	0.39	2.09						0
Blank-2		121599			4.0	0.8	200		0.00	0.39	2.09						0
ccv		121599						3.75	5.940	0.39	2.09	2.84				75.79	
M4-599-SFF-A	"H"	32500	4.440	0.102	3.8	0.6	200			-0.09	2.47						
M4-599-SFF-B	"H"	32500	5.363	0.102	4	0.6	200		2.57	-0.09	2.47	0.79		0.79			
M4-599-SFF-C	"H"	32500	6.011	0.102	4	0.6	200	1.220	7.68	-0.09	2.47	2.11					
M4-599-SFF-D	"H"	32500	5.235	0.102	4	0.6	200	1.400	9.09	-0.09	2.47	2.87	-30.44	2.49		129.76	0.61
Correlation Coefficient	"NR"	32500															
Blank-1		32500			4.0	0.6	200		0.00	-0.09	2.47						0
Blank-2		32500			4.0	0.6	200		0.00	-0.09	2.47						0
ccv		32500						2.5	5.270	-0.09	2.47	2.13				85.34	
ccv		32500						2.5	7.660	-0.09	2.47	3.10				124.05	
M4-809-SFF-A	"H"	91099	5.473	0.0416	3.8	0.8	200		0.00	0.289	2.168	0.00					
M4-809-SFF-B	"H"	91099	5.544	0.0416	3.6	0.8	200		0.00	0.289	2.168	0.00	0.00	0.00			
M4-809-SFF-C	"H"	91099	5.194	0.0416	3.8	0.8	200	3.47	1.77	0.289	2.168	1.24					
M4-809-SFF-D	"H", "M"	91099	5.326	0.0416	3.6	0.8	200	3.39	1.75	0.289	2.168	1.27	-1.76	1.25		36.56	0.00
Correlation Coefficient	"NR"	91099															
Blank-1		91099			4.0	0.8	200			0.289	2.168						0
Blank-2		91099			4.0	0.8	200			0.289	2.168						0
ccv		91099						3.75	6.80	0.289	2.168	3.14				83.64	
ccv		91099						3.75	6.22	0.289	2.168	2.87				76.51	
ccv		91099						3.75	7.35	0.289	2.168	3.39				90.41	
ccv		91099						3.75	5.71	0.289	2.168	2.63				70.23	
M4-872-SDF-A	"H"	032800b	4.517	0.119	4.0	0.6	200		0.83	0.08	3.82	0.17					
M4-872-SDF-B	"H"	032800b	4.562	0.119	4.0	0.6	200		1.39	0.08	3.82	0.28	-49.52	0.22			
M4-872-SDF-C	"H"	032800b	4.351	0.119	4.0	0.6	200	1.45	7.44	0.08	3.82	1.57					
M4-872-SDF-D	"H"	032800b	4.722	0.119	4.0	0.6	200	1.33	7.97	0.08	3.82	1.55	1.30	1.56		95.92	0.23
Correlation Coefficient	"NR"	032800b															
Blank-1		032800b			4.0	0.8	200		0.00	0.42	2.13						0
Blank-2		032800b			4.0	0.8	200		0.00	0.42	2.13						0
ccv	"NR"	032800b															

Data Qualifiers

"H" Analysis digestion performed after holding times have expired.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 70 to 130% range.

"NR" Not Reviewed.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M4-501-SFF Soil/Sediment

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	5-11-99/No Time	5-11-99/No Time	5-11-99/No Time	5-11-99/No Time	5-11-99/No Time
Digestion Date	Not Analyzed	03/20/00	09/03/99	06/25/99	NA
Analysis Date	Battelle Lab	03/20/00	09/09/99	06/25/99	Not Provided
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume		4.65, 4.046 g	0.025 g	0.025 g	80
Total Volume		200ul	0.025 g	0.025 g	80
Sample Volume Analyzed		aliquot	Aliquot	0.025 g	80
Dilution		See Worksheets	1	NA	NA
Results		4.87	153.3	0.9608	0.0544
Measuring Unit	ppm	ng/g	ug/g	%	g/mL
EPA Method	CVAF	CVAF	EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst		JL	Angel	SG/JL	SG/JL
Method Detection limit		0.2 ng/g	0.06		0.001 g/mL
Data Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	Yes
All Calculation Checked		Yes	X	X	X
Holding Time Met	28 days	No (Goal Only)	No (Goal Only)	NA	NA
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID		32000a	9-9-99 C-1	06/25/99	Not Provided
Method Blanks		NA	NA in Soils	NA	NA
Instrument Blanks		yes	-0.3, -0.2	NA	NA
Duplicates (RPD)		89.38	<20 RPD	None Reported	None Reported
Matrix Spike Recoveries (75-125)		121.44	96%R	NA	NA
Calibration % R (high and low range)		96.09	88 - 92%R	NA	NA
Detection Range		0.2 ng/g and >	60 ppm and >	NA	NA
Correlation Coefficient (>0.995)		NR	0.996	NA	NA
QC Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	Yes
All Calculation Checked		Yes	X	X	X
QC Limits Met	Battelle Lab	"DQO"		"DQO (NR)"	"DQO (NR)"

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time. Holding time is a goal only.

Some analysis for AFDW were Re-Run based on negative numbers reported, but no QA/QC batches were re-run.

No duplicate/replicate measurements were taken with the Bulk Density analysis.

Station ID M4-501-SFF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	X
Matrix	X	X	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage		Hg lab	Not Noted	Not Noted	Not Noted
Log-in Procedures	Internal COC	Internal COC	Internal COC	Internal COC	Internal COC
Raw Data (Attached)					
Sample Work Sheets		Yes	X	X	X
Sample Run Logs		Yes	X	X	X
Instrument Raw Data		Yes	X	NA	NA
Bench Sheets		Yes	X	X	X
Sample Preparation Logs		Yes	X	X	X
Raw Data (Verified)					
Sample ID Transferred		Yes	X	X	X
All Calculation Checked		Yes	X	X	X
Measuring Unit		ng/g	ug/g	%	g/mL
PE Results (Attached)					
Organization	NA	NA	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA	NA	NA
Validation Criteria					
Applied Qualifiers		"DQO"		"DQO (NR)"	"DQO (NR)"

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

" B " Analyte concentration in the associated blank was >3 times the MDL.

" H " Analysis digestion performed after holding times have expired.

" NR " Data was unavailable for review.

" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M4-508-SFF Soil/Sediment

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	5-11-99/No Time	5-11-99/No Time	5-11-99/No Time	5-11-99/No Time	5-11-99/No Time
Digestion Date	Not Analyzed	No Analysis Performed	09/03/99	06/25/99	NA
Analysis Date	Battelle Lab		10/01/99	06/25/99	Not Provided
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume			0.025 g	0.025 g	80
Total Volume			0.025 g	0.025 g	80
Sample Volume Analyzed			Aliquot	0.025 g	80
Dilution			1	NA	NA
Results		No Analysis Performed	291.7	0.9713	0.0627
Measuring Unit	ppm		ug/g	%	g/mL
EPA Method	CVAF		EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst			Angel	SG/JL	SG/JL
Method Detection limit			0.06		0.001 g/mL
Data Report (Verified)					
Lab Data Entry Checked by Analyst			Yes	Yes	Yes
All Calculation Checked			X	X	X
Holding Time Met	28 days		No (Goal Only)	NA	NA
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID			10-1-99 C-1	06/25/99	Not Provided
Method Blanks			NA in Soils	NA	NA
Instrument Blanks			None Reported	NA	NA
Duplicates (RPD)			<20 RPD	None Reported	None Reported
Matrix Spike Recoveries (75-125)			98 - 102%R	NA	NA
Calibration % R (high and low range)			96 - 102 %R	NA	NA
Detection Range			60 ppm and >	NA	NA
Correlation Coefficient (>0.995)			0.998	NA	NA
QC Report (Verified)					
Lab Data Entry Checked by Analyst			Yes	Yes	Yes
All Calculation Checked			X	X	X
QC Limits Met	Battelle Lab		"B (NR)"	"DQO (NR)"	"DQO (NR)"

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time. Holding time is a goal only.

Some analysis for AFDW were Re-Run based on negative numbers reported, but no QA/QC batches were re-run.

No duplicate/replicate measurements were taken with the Bulk Density analysis.

There is no analysis for MeHg.

Station ID M4-508-SFF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X		X	X	X
Matrix	X		X	X	X
Preservative (Acid, Temp..)	Not Noted		Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time		Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage			Not Noted	Not Noted	Not Noted
Log-in Procedures	Internal COC		Internal COC	Internal COC	Internal COC
Raw Data (Attached)					
Sample Work Sheets			X	X	X
Sample Run Logs			X	X	X
Instrument Raw Data			X	NA	NA
Bench Sheets			X	X	X
Sample Preparation Logs			X	X	X
Raw Data (Verified)					
Sample ID Transferred			X	X	X
All Calculation Checked			X	X	X
Measuring Unit			ug/g	%	g/mL
PE Results (Attached)					
Organization	NA		NA	NA	NA
Performance (Pass/Fail)	NA		NA	NA	NA
Validation Criteria					
Applied Qualifiers			"B (NR)"	"DQO (NR)"	"DQO (NR)"

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

" B " Analyte concentration in the associated blank was >3 times the MDL.

" H " Analysis digestion performed after holding times have expired.

" NR " Data was unavailable for review.

" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID **M4-533-SFF** **Soil/Sediment**

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	5-10-99/No Time	5-10-99/No Time	5-10-99/No Time	5-10-99/No Time	5-10-99/No Time
Digestion Date	Not Analyzed	03/24/00	09/03/99	06/25/99	NA
Analysis Date	Battelle Lab	03/24/00	10/01/99	06/25/99	Not Provided
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume		4.602, 4.753	0.025 g	0.025 g	80
Total Volume		200ul	0.025 g	0.025 g	80
Sample Volume Analyzed		aliquot	Aliquot	0.025 g	80
Dilution		See Worksheets	1	NA	NA
Results		0	231.2	0.9261	0.1013
Measuring Unit	ppm	ng/g	ug/g	%	g/mL
EPA Method	CVAF	CVAF	EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst		JL	Angel	SG/JL	SG/JL
Method Detection limit		0.2 ng/g	0.06		0.001 g/cc
Data Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	Yes
All Calculation Checked		Yes	X	X	X
Holding Time Met	28 days	No (Goal Only)	No (Goal Only)	NA	NA
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID		032400b	10-1-99 C-1	06/25/99	Not Provided
Method Blanks		NA	NA in Soils	NA	NA
Instrument Blanks		yes	None Reported	NA	NA
Duplicates (RPD)		0	<20 RPD	<20 RPD	None Reported
Matrix Spike Recoveries (75-125)		47.47	98 - 102%R	NA	NA
Calibration % R (high and low range)		89.32, 84.07	96 - 102 %R	NA	NA
Detection Range		0.2 ng/g and >	60 ppm and >	NA	NA
Correlation Coefficient (>0.995)		0.9913	0.998	NA	NA
QC Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	Yes
All Calculation Checked		Yes	X	X	X
QC Limits Met	Battelle Lab	"M"	"B (NR)"		"DQO (NR)"

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time. Holding time is a goal only.

Some analysis for AFDW were Re-Run based on negative numbers reported, but no QA/QC batches were re-run.

No duplicate/replicate measurements were taken with the Bulk Density analysis.

Station ID M4-533-SFF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	X
Matrix	X	X	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage		Hg lab	Not Noted	Not Noted	Not Noted
Log-in Procedures	Internal COC	Internal COC	Internal COC	Internal COC	Internal COC
Raw Data (Attached)					
Sample Work Sheets		Yes	X	X	X
Sample Run Logs		Yes	X	X	X
Instrument Raw Data		Yes	X	NA	NA
Bench Sheets		Yes	X	X	X
Sample Preparation Logs		Yes	X	X	X
Raw Data (Verified)					
Sample ID Transferred		Yes	X	X	X
All Calculation Checked		Yes	X	X	X
Measuring Unit		ng/g	ug/g	%	g/mL
PE Results (Attached)					
Organization	NA	NA	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA	NA	NA
Validation Criteria					
Applied Qualifiers		"M"	"B (NR)"		"DQO (NR)"

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

" B " Analyte concentration in the associated blank was >3 times the MDL.

" H " Analysis digestion performed after holding times have expired.

" NR " Data was unavailable for review.

" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID **M4-538-SFF** **Soil/Sediment**

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	5-10-99/No Time	5-10-99/No Time	5-10-99/No Time	5-10-99/No Time	5-10-99/No Time
Digestion Date	Not Analyzed	No Analysis Performed	09/03/99	06/25/99	NA
Analysis Date	Battelle Lab		10/01/99	06/25/99	Not Provided
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume			0.025 g	0.025 g	80
Total Volume			0.025 g	0.025 g	80
Sample Volume Analyzed			Aliquot	0.025 g	80
Dilution			1	NA	NA
Results		No Analysis Performed	105.6	0.48	0.1965
Measuring Unit	ppm		ug/g	%	g/mL
EPA Method	CVAF		EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst			Angel	SG/JL	SG/JL
Method Detection limit			0.06		0.001 g/cc
Data Report (Verified)					
Lab Data Entry Checked by Analyst			Yes	Yes	Yes
All Calculation Checked			X	X	X
Holding Time Met	28 days		No (Goal Only)	NA	NA
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID			10-1-99 C-1	06/25/99	Not Provided
Method Blanks			NA in Soils	NA	NA
Instrument Blanks			None Reported	NA	NA
Duplicates (RPD)			<20 RPD	<20 RPD	None Reported
Matrix Spike Recoveries (75-125)			98 - 102%R	NA	NA
Calibration % R (high and low range)			96 - 102 %R	NA	NA
Detection Range			60 ppm and >	NA	NA
Correlation Coefficient (>0.995)			0.998	NA	NA
QC Report (Verified)					
Lab Data Entry Checked by Analyst			Yes	Yes	Yes
All Calculation Checked			X	X	X
QC Limits Met	Battelle Lab		"B (NR)"		"DQO (NR)"

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time. Holding time is a goal only.

Some analysis for AFDW were Re-Run based on negative numbers reported, but no QA/QC batches were re-run.

No duplicate/replicate measurements were taken with the Bulk Density analysis.

Station ID M4-538-SFF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X		X	X	X
Matrix	X		X	X	X
Preservative (Acid, Temp..)	Not Noted		Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time		Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage			Not Noted	Not Noted	Not Noted
Log-in Procedures	Internal COC		Internal COC	Internal COC	Internal COC
Raw Data (Attached)					
Sample Work Sheets			X	X	X
Sample Run Logs			X	X	X
Instrument Raw Data			X	NA	NA
Bench Sheets			X	X	X
Sample Preparation Logs			X	X	X
Raw Data (Verified)					
Sample ID Transferred			X	X	X
All Calculation Checked			X	X	X
Measuring Unit			ug/g	%	g/mL
PE Results (Attached)					
Organization	NA		NA	NA	NA
Performance (Pass/Fail)	NA		NA	NA	NA
Validation Criteria					
Applied Qualifiers			"B (NR)"		"DQO (NR)"

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

" B " Analyte concentration in the associated blank was >3 times the MDL.

" H " Analysis digestion performed after holding times have expired.

" NR " Data was unavailable for review.

" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M4-548-SFF

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	5-9-99/No Time	5-9-99/No Time	5-9-99/No Time	5-9-99/No Time	5-9-99/No Time
Digestion Date	Not Analyzed	08/06/99	09/03/99	06/25/99	NA
Analysis Date	Battelle Lab	08/06/99	09/09/99	06/25/99	Not Provided
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume		4.475g,4.095g	0.025 g	0.025 g	80
Total Volume		200ul	0.025 g	0.025 g	80
Sample Volume Analyzed		aliquot	Aliquot	0.025 g	80
Dilution		See Worksheet	1	NA	NA
Results		0.68	337.2	0.91	0.0699
Measuring Unit	ppm	ng/g	ug/g	%	g/mL
EPA Method	CVAF	CVAF	EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst		JL	Angel	SG/JL	SG/JL
Method Detection limit		0.2 ng/g	0.06		0.001 g/cc
Data Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	Yes
All Calculation Checked		Yes	X	X	X
Holding Time Met	28 days	No (Goal Only)	No (Goal Only)	NA	NA
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID		80699	9-9-99 C-2	06/25/99	Not Provided
Method Blanks		NA	NA in Soils	NA	NA
Instrument Blanks		yes	-0.1, -0.2	NA	NA
Duplicates (RPD)		47.17	<20 RPD	<20 RPD	None Reported
Matrix Spike Recoveries (75-125)		103.35	89 - 93%R	NA	NA
Calibration % R (high and low range)		103.97, 35.44, 141.31	89 - 97%R	NA	NA
Detection Range		0.2 ng/g and >	60 ppm and >	NA	NA
Correlation Coefficient (>0.995)		0.9963	0.9969	NA	NA
QC Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	Yes
All Calculation Checked		Yes	X	X	X
QC Limits Met	Battelle Lab	"DQO"			"DQO (NR)"

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time. Holding time is a goal only.

Some analysis for AFDW were Re-Run based on negative numbers reported, but no QA/QC batches were re-run.

No duplicate/replicate measurements were taken with the Bulk Density analysis.

Station ID **M4-548-SFF** **M4-548-SDF**

Soil/Sediment

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	X
Matrix	X	X	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage		Hg lab	Not Noted	Not Noted	Not Noted
Log-in Procedures	Internal COC	Internal COC	Internal COC	Internal COC	Internal COC
Raw Data (Attached)					
Sample Work Sheets		Yes	X	X	X
Sample Run Logs		Yes	X	X	X
Instrument Raw Data		Yes	X	NA	NA
Bench Sheets		Yes	X	X	X
Sample Preparation Logs		Yes	X	X	X
Raw Data (Verified)					
Sample ID Transferred		Yes	X	X	X
All Calculation Checked		Yes	X	X	X
Measuring Unit		ng/g	ug/g	%	g/mL
PE Results (Attached)					
Organization	NA	NA	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA	NA	NA
Validation Criteria					
Applied Qualifiers		"DQO"			"DQO (NR)"

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

" B " Analyte concentration in the associated blank was >3 times the MDL.

" H " Analysis digestion performed after holding times have expired.

" NR " Data was unavailable for review.

" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M4-556-SFF

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	5-11-99/No Time		5-8-99/No Time	5-11-99/No Time	5-11-99/No Time
Digestion Date	Not Analyzed	08/06/99	09/03/99	06/25/99	NA
Analysis Date	Battelle Lab	08/06/99	09/09/99	06/25/99	Not Provided
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume		4.190g,4.153g	0.025 g	0.025 g	80
Total Volume		200ul	0.025 g	0.025 g	80
Sample Volume Analyzed		aliquot	Aliquot	0.025 g	80
Dilution		Blending Dilution	1	NA	NA
Results		0.63	85.7	0.4567	0.1983
Measuring Unit	ppm	ng/g	ug/g	%	g/mL
EPA Method	CVAF	CVAF	EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst		JL	Angel	SG/JL	SG/JL
Method Detection limit		0.2 ng/g	0.06		0.001 g/cc
Data Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	Yes
All Calculation Checked		Yes	X	X	X
Holding Time Met	28 days	No (Goal Only)	No (Goal Only)	NA	NA
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID		80699	9-9-99 C-2	06/25/99	Not Provided
Method Blanks		yes	NA in Soils	NA	NA
Instrument Blanks		NA	-0.1, -0.2	NA	NA
Duplicates (RPD)		59.71	<20 RPD	<20 RPD	None Reported
Matrix Spike Recoveries (75-125)		43.8	89 - 93%R	NA	NA
Calibration % R (high and low range)		103.97, 35.44, 141.31	89 - 97%R	NA	NA
Detection Range		0.2 ng/g and >	60 ppm and >	NA	NA
Correlation Coefficient (>0.995)		0.9963	0.9969	NA	NA
QC Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	Yes
All Calculation Checked		Yes	X	X	X
QC Limits Met	Battelle Lab	"M", "DQO"			"DQO (NR)"

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time. Holding time is a goal only.

Some analysis for AFDW were Re-Run based on negative numbers reported, but no QA/QC batches were re-run.

No duplicate/replicate measurements were taken with the Bulk Density analysis.

For MeHg samples, a matrix spike is performed with every sample and is used to correct the results for loss of analyte.

Station ID M4-556-SFF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	X
Matrix	X	X	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage		Hg lab	Not Noted	Not Noted	Not Noted
Log-in Procedures	Internal COC	Internal COC	Internal COC	Internal COC	Internal COC
Raw Data (Attached)					
Sample Work Sheets		Yes	X	X	X
Sample Run Logs		Yes	X	X	X
Instrument Raw Data		Yes	X	NA	NA
Bench Sheets		Yes	X	X	X
Sample Preparation Logs		Yes	X	X	X
Raw Data (Verified)					
Sample ID Transferred		Yes	X	X	X
All Calculation Checked		Yes	X	X	X
Measuring Unit		ng/g	ug/g	%	g/mL
PE Results (Attached)					
Organization	NA	NA	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA	NA	NA
Validation Criteria					
Applied Qualifiers		"M", "DQO"			"DQO (NR)"

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

" B " Analyte concentration in the associated blank was >3 times the MDL.

" H " Analysis digestion performed after holding times have expired.

" NR " Data was unavailable for review.

" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID **M4-566-SFF** **Soil/Sediment**

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	5-7-99/No Time	5-7-99/No Time	5-7-99/No Time	5-7-99/No Time	5-7-99/No Time
Digestion Date	Not Analyzed	08/12/99	09/03/99	06/25/99	NA
Analysis Date	Battelle Lab	08/13/99	09/09/99	06/25/99	Not Provided
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume		5.557, 5.435	0.025 g	0.025 g	80
Total Volume		200 ul	0.025 g	0.025 g	80
Sample Volume Analyzed		aliquot	Aliquot	0.025 g	80
Dilution		See Worksheets	1	NA	NA
Results		0.84	214.2	0.8627	0.1541
Measuring Unit	ppm	ng/g	ug/g	%	g/mL
EPA Method	CVAF	CVAF	EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst		JL	Angel	SG/JL	SG/JL
Method Detection limit		0.2 ng/g	0.06		0.001 g/cc
Data Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	Yes
All Calculation Checked		Yes	X	X	X
Holding Time Met	28 days	No (Goal Only)	No (Goal Only)	NA	NA
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID		81299	9-9-99 C-2	06/25/99	Not Provided
Method Blanks		NA	NA in Soils	NA	NA
Instrument Blanks		yes	-0.1, -0.2	NA	NA
Duplicates (RPD)		81.65	<20 RPD	<20 RPD	None Reported
Matrix Spike Recoveries (75-125)		108.16	89 - 93%R	NA	NA
Calibration % R (high and low range)		159	89 - 97%R	NA	NA
Detection Range		0.2 ng/g and >	60 ppm and >	NA	NA
Correlation Coefficient (>0.995)		0.9566	0.9969	NA	NA
QC Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	Yes
All Calculation Checked		Yes	X	X	X
QC Limits Met	Battelle Lab	"M", "DQO"			"DQO (NR)"

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time. Holding time is a goal only.

Some analysis for AFDW were Re-Run based on negative numbers reported, but no QA/QC batches were re-run.

No duplicate/replicate measurements were taken with the Bulk Density analysis.

For MeHg samples, a matrix spike is performed with every sample and is used to correct the results for loss of analyte.

Station ID M4-566-SFF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	X
Matrix	X	X	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage		Hg lab	Not Noted	Not Noted	Not Noted
Log-in Procedures	Internal COC	Internal COC	Internal COC	Internal COC	Internal COC
Raw Data (Attached)					
Sample Work Sheets		Yes	X	X	X
Sample Run Logs		Yes	X	X	X
Instrument Raw Data		Yes	X	NA	NA
Bench Sheets		Yes	X	X	X
Sample Preparation Logs		Yes	X	X	X
Raw Data (Verified)					
Sample ID Transferred		Yes	X	X	X
All Calculation Checked		Yes	X	X	X
Measuring Unit		ng/g	ug/g	%	g/mL
PE Results (Attached)					
Organization	NA	NA	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA	NA	NA
Validation Criteria					
Applied Qualifiers		"M", "DQO"			"DQO (NR)"

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

" B " Analyte concentration in the associated blank was >3 times the MDL.

" H " Analysis digestion performed after holding times have expired.

" NR " Data was unavailable for review.

" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M4-568-SFF Soil/Sediment

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	5-7-99/No Time	5-7-99/No Time	5-7-99/No Time	5-7-99/No Time	5-7-99/No Time
Digestion Date	Not Analyzed	12/14/99	09/03/99	06/25/99	NA
Analysis Date	Battelle Lab	12/14/99	09/09/99	06/25/99	Not Provided
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume		4.181g,3.423g	0.025 g	0.025 g	80
Total Volume		200ul	0.025 g	0.025 g	80
Sample Volume Analyzed		aliquot	Aliquot	0.025 g	80
Dilution		Blending Dilution	1	NA	NA
Results		0	162.1	0.87	0.1046
Measuring Unit	ppm	ng/g	ug/g	%	g/mL
EPA Method	CVAF	CVAF	EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst		JL	Angel	SG/JL	SG/JL
Method Detection limit		0.2 ng/g	0.06		0.001 g/cc
Data Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	Yes
All Calculation Checked		Yes	X	X	X
Holding Time Met	28 days	No (Goal Only)	No (Goal Only)	NA	NA
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID		121499	9-9-99 C-2	06/25/99	Not Provided
Method Blanks		NA	NA in Soils	NA	NA
Instrument Blanks		yes	-0.1, -0.2	NA	NA
Duplicates (RPD)		0.00	<20 RPD	<20 RPD	None Reported
Matrix Spike Recoveries (75-125)		124.34	89 - 93%R	NA	NA
Calibration % R (high and low range)		NR	89 - 97%R	NA	NA
Detection Range		0.2 ng/g and >	60 ppm and >	NA	NA
Correlation Coefficient (>0.995)		NR	0.9969	NA	NA
QC Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	Yes
All Calculation Checked		Yes	X	X	X
QC Limits Met	Battelle Lab	"DQO"			"DQO (NR)"

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time. Holding time is a goal only.

Some analysis for AFDW were Re-Run based on negative numbers reported, but no QA/QC batches were re-run.

No duplicate/replicate measurements were taken with the Bulk Density analysis.

For MeHg samples, a matrix spike is performed with every sample and is used to correct the results for loss of analyte.

Station ID M4-568-SFF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix		Narrative summaries will be written following the completion on the data analysis			
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	X
Matrix	X	X	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory	Is the same as station ID			
Sample Handling/Storage		Hg lab	Not Noted	Not Noted	Not Noted
Log-in Procedures	Internal COC	Internal COC	Internal COC	Internal COC	Internal COC
Raw Data (Attached)					
Sample Work Sheets		Yes	X	X	X
Sample Run Logs		Yes	X	X	X
Instrument Raw Data		Yes	X	NA	NA
Bench Sheets		Yes	X	X	X
Sample Preparation Logs		Yes	X	X	X
Raw Data (Verified)					
Sample ID Transferred		Yes	X	X	X
All Calculation Checked		Yes	X	X	X
Measuring Unit		ng/g	ug/g	%	g/mL
PE Results (Attached)					
Organization	NA	NA	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA	NA	NA
Validation Criteria					
Applied Qualifiers		"DQO"			"DQO (NR)"

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

" B " Analyte concentration in the associated blank was >3 times the MDL.

" H " Analysis digestion performed after holding times have expired.

" NR " Data was unavailable for review.

" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID **M4-576-SFF** **Soil/Sediment**

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	5-4-99/No Time	5-4-99/No Time	5-4-99/No Time	5-4-99/No Time	5-4-99/No Time
Digestion Date	Not Analyzed	03/26/00	09/03/99	06/25/99	NA
Analysis Date	Battelle Lab	03/26/00	09/09/99	06/25/99	Not Provided
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume		4.624	0.025 g	0.025 g	80
Total Volume		4.56	0.025 g	0.025 g	80
Sample Volume Analyzed		aliquot	Aliquot	0.025 g	80
Dilution		See Worksheet	1	NA	NA
Results		1.85	250.61	0.7171	0.1348
Measuring Unit	ppm	ng/g	ug/g	%	g/mL
EPA Method	CVAF	CVAF	EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst		JL	Angel	SG/JL	SG/JL
Method Detection limit		0.2 ng/g	0.06		0.001 g/cc
Data Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	Yes
All Calculation Checked		Yes	X	X	X
Holding Time Met	28 days	No (Goal Only)	No (Goal Only)	NA	NA
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID		32600	9-9-99 C-2	06/25/99	Not Provided
Method Blanks		NA	NA in Soils	NA	NA
Instrument Blanks		yes	-0.1, -0.2	NA	NA
Duplicates (RPD)		11.04	<20 RPD	<20 RPD	None Reported
Matrix Spike Recoveries (75-125)		110.5	89 - 93%R	NA	NA
Calibration % R (high and low range)		107.03, 87.14	89 - 97%R	NA	NA
Detection Range		0.2 ng/g and >	60 ppm and >	NA	NA
Correlation Coefficient (>0.995)		NR	0.9969	NA	NA
QC Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	Yes
All Calculation Checked		Yes	X	X	X
QC Limits Met	Battelle Lab				"DQO (NR)"

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time. Holding time is a goal only.

Some analysis for AFDW were Re-Run based on negative numbers reported, but no QA/QC batches were re-run.

No duplicate/replicate measurements were taken with the Bulk Density analysis.

For MeHg samples, a matrix spike is performed with every sample and is used to correct the results for loss of analyte.

Station ID M4-576-SFF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix		Narrative summaries will be written following the completion on the data analysis			
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	X
Matrix	X	X	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory	Is the same as station ID			
Sample Handling/Storage		Hg lab	Not Noted	Not Noted	Not Noted
Log-in Procedures	Internal COC	Internal COC	Internal COC	Internal COC	Internal COC
Raw Data (Attached)					
Sample Work Sheets		Yes	X	X	X
Sample Run Logs		Yes	X	X	X
Instrument Raw Data		Yes	X	NA	NA
Bench Sheets		Yes	X	X	X
Sample Preparation Logs		Yes	X	X	X
Raw Data (Verified)					
Sample ID Transferred		Yes	X	X	X
All Calculation Checked		Yes	X	X	X
Measuring Unit		ng/g	ug/g	%	g/mL
PE Results (Attached)					
Organization	NA	NA	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA	NA	NA
Validation Criteria					
Applied Qualifiers					"DQO (NR)"

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

" B " Analyte concentration in the associated blank was >3 times the MDL.

" H " Analysis digestion performed after holding times have expired.

" NR " Data was unavailable for review.

" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M4-586-SFF Soil/Sediment

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	5-6-99/No Time	5-6-99/No Time	5-6-99/No Time	5-6-99/No Time	5-6-99/No Time
Digestion Date	Not Analyzed	08/20/99	09/03/99	06/25/99	NA
Analysis Date	Battelle Lab	08/20/99	09/09/99	06/25/99	Not Provided
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume		4.302g,4.217g	0.025 g	0.025 g	80
Total Volume		200ul	0.025 g	0.025 g	80
Sample Volume Analyzed		aliquot	Aliquot	0.025 g	80
Dilution		Blending Dilution	1	NA	NA
Results		0	63	0.2126	0.457
Measuring Unit	ppm	ng/g	ug/g	%	g/mL
EPA Method	CVAF	CVAF	EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst		JL	Angel	SG/JL	SG/JL
Method Detection limit		0.2 ng/g	0.06		0.001 g/cc
Data Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	Yes
All Calculation Checked		Yes	X	X	X
Holding Time Met	28 days	No (Goal Only)	No (Goal Only)	NA	NA
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID		82099	9-9-99 C-2	06/25/99	Not Provided
Method Blanks		NA	NA in Soils	NA	NA
Instrument Blanks		yes	-0.1, -0.2	NA	NA
Duplicates (RPD)		0	<20 RPD	>20 RPD	None Reported
Matrix Spike Recoveries (75-125)		28.16	89 - 93%R	NA	NA
Calibration % R (high and low range)		81.4	89 - 97%R	NA	NA
Detection Range		0.2 ng/g and >	60 ppm and >	NA	NA
Correlation Coefficient (>0.995)		0.9713	0.9969	NA	NA
QC Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	Yes
All Calculation Checked		Yes	X	X	X
QC Limits Met	Battelle Lab	"M"		"DQO"	"DQO (NR)"

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time. Holding time is a goal only.

Some analysis for AFDW were Re-Run based on negative numbers reported, but no QA/QC batches were re-run.

No duplicate/replicate measurements were taken with the Bulk Density analysis.

For MeHg samples, a matrix spike is performed with every sample and is used to correct the results for loss of analyte.

Station ID M4-586-SFF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	X
Matrix	X	X	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage		Hg lab	Not Noted	Not Noted	Not Noted
Log-in Procedures	Internal COC	Internal COC	Internal COC	Internal COC	Internal COC
Raw Data (Attached)					
Sample Work Sheets		Yes	X	X	X
Sample Run Logs		Yes	X	X	X
Instrument Raw Data		Yes	X	NA	NA
Bench Sheets		Yes	X	X	X
Sample Preparation Logs		Yes	X	X	X
Raw Data (Verified)					
Sample ID Transferred		Yes	X	X	X
All Calculation Checked		Yes	X	X	X
Measuring Unit		ng/g	ug/g	%	g/mL
PE Results (Attached)					
Organization	NA	NA	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA	NA	NA
Validation Criteria					
Applied Qualifiers		"M"		"DQO"	"DQO (NR)"

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

" B " Analyte concentration in the associated blank was >3 times the MDL.

" H " Analysis digestion performed after holding times have expired.

" NR " Data was unavailable for review.

" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M4-594-SFF Soil/Sediment

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	5-5-99/No Time	5-5-99/No Time	5-5-99/No Time	5-5-99/No Time	5-5-99/No Time
Digestion Date	Not Analyzed	12/15/99	09/03/99	06/25/99	NA
Analysis Date	Battelle Lab	12/15/99	09/09/99	06/25/99	Not Provided
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume		4.296g,4.078g	0.025 g	0.025 g	80
Total Volume		200ul	0.025 g	0.025 g	80
Sample Volume Analyzed		aliquot	Aliquot	0.025 g	80
Dilution		See Worksheet	1	NA	NA
Results		1.58	330.6	0.6824	0.1944
Measuring Unit	ppm	ng/g	ug/g	%	g/mL
EPA Method	CVAF	CVAF	EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst		JL	Angel	SG/JL	SG/JL
Method Detection limit		0.2 ng/g	0.06		0.001 g/cc
Data Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	Yes
All Calculation Checked		Yes	X	X	X
Holding Time Met	28 days	No (Goal Only)	No (Goal Only)	NA	NA
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID		121599	9-9-99 C-1a	06/25/99	Not Provided
Method Blanks		NA	NA in Soils	NA	NA
Instrument Blanks		yes	-0.3, -0.4	NA	NA
Duplicates (RPD)		-38.82	<20 RPD	<20 RPD	None Reported
Matrix Spike Recoveries (75-125)		80.89	95 - 96%R	NA	NA
Calibration % R (high and low range)		75.79	62 - 100%R	NA	NA
Detection Range		0.2 ng/g and >	60 ppm and >	NA	NA
Correlation Coefficient (>0.995)		0.9881	0.9952	NA	NA
QC Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	Yes
All Calculation Checked		Yes	X	X	X
QC Limits Met	Battelle Lab	"DQO"	"DQO"		"DQO (NR)"

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time. Holding time is a goal only.

Some analysis for AFDW were Re-Run based on negative numbers reported, but no QA/QC batches were re-run.

No duplicate/replicate measurements were taken with the Bulk Density analysis.

For MeHg samples, a matrix spike is performed with every sample and is used to correct the results for loss of analyte.

Station ID M4-594-SFF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	X
Matrix	X	X	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage		Hg lab	Not Noted	Not Noted	Not Noted
Log-in Procedures	Internal COC	Internal COC	Internal COC	Internal COC	Internal COC
Raw Data (Attached)					
Sample Work Sheets		Yes	X	X	X
Sample Run Logs		Yes	X	X	X
Instrument Raw Data		Yes	X	NA	NA
Bench Sheets		Yes	X	X	X
Sample Preparation Logs		Yes	X	X	X
Raw Data (Verified)					
Sample ID Transferred		Yes	X	X	X
All Calculation Checked		Yes	X	X	X
Measuring Unit		ng/g	ug/g	%	g/mL
PE Results (Attached)					
Organization	NA	NA	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA	NA	NA
Validation Criteria					
Applied Qualifiers		"DQO"	"DQO"		"DQO (NR)"

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

" B " Analyte concentration in the associated blank was >3 times the MDL.

" H " Analysis digestion performed after holding times have expired.

" NR " Data was unavailable for review.

" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID **M4-599-SFF** **Soil/Sediment**

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	5-6-99/No Time	5-6-99/No Time	5-6-99/No Time	5-6-99/No Time	5-6-99/No Time
Digestion Date	Not Analyzed	03/25/00	10/13/99	06/25/99	NA
Analysis Date	Battelle Lab	03/25/00	10/29/99	06/25/99	Not Provided
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume		4.44, 5.363	0.025 g	0.025 g	80
Total Volume		200ul	0.025 g	0.025 g	80
Sample Volume Analyzed		aliquot	Aliquot	0.025 g	80
Dilution		See Worksheet	1	NA	NA
Results		0.61	176.6	0.9124	0.1849
Measuring Unit	ppm	ng/g	ug/g	%	g/mL
EPA Method	CVAF	CVAF	EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst		JL	Angel	SG/JL	SG/JL
Method Detection limit		0.2 ng/g	0.06		0.001 g/cc
Data Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	Yes
All Calculation Checked		Yes	X	X	X
Holding Time Met	28 days	No (Goal Only)	No (Goal Only)	NA	NA
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID		32500	10-29-99 C-1	06/25/99	Not Provided
Method Blanks		NA	NA in Soils	NA	NA
Instrument Blanks		yes	0.0, -0.1	NA	NA
Duplicates (RPD)		-30.44	<20 RPD	<20 RPD	None Reported
Matrix Spike Recoveries (75-125)		129.76	86 - 91%R	NA	NA
Calibration % R (high and low range)		85.34, 124.05	93 - 101%R	NA	NA
Detection Range		0.2 ng/g and >	60 ppm and >	NA	NA
Correlation Coefficient (>0.995)		0.9985	0.9962	NA	NA
QC Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	Yes
All Calculation Checked		Yes	X	X	X
QC Limits Met	Battelle Lab	"DQO"			"DQO (NR)"

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time. Holding time is a goal only.

Some analysis for AFDW were Re-Run based on negative numbers reported, but no QA/QC batches were re-run.

No duplicate/replicate measurements were taken with the Bulk Density analysis.

For MeHg samples, a matrix spike is performed with every sample and is used to correct the results for loss of analyte.

Station ID M4-599-SFF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	X
Matrix	X	X	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage		Hg lab	Not Noted	Not Noted	Not Noted
Log-in Procedures	Internal COC	Internal COC	Internal COC	Internal COC	Internal COC
Raw Data (Attached)					
Sample Work Sheets		Yes	X	X	X
Sample Run Logs		Yes	X	X	X
Instrument Raw Data		Yes	X	NA	NA
Bench Sheets		Yes	X	X	X
Sample Preparation Logs		Yes	X	X	X
Raw Data (Verified)					
Sample ID Transferred		Yes	X	X	X
All Calculation Checked		Yes	X	X	X
Measuring Unit		ng/g	ug/g	%	g/mL
PE Results (Attached)					
Organization	NA	NA	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA	NA	NA
Validation Criteria					
Applied Qualifiers		"DQO"			"DQO (NR)"

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

" B " Analyte concentration in the associated blank was >3 times the MDL.

" H " Analysis digestion performed after holding times have expired.

" NR " Data was unavailable for review.

" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M4-809-SFF Soil/Sediment

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	5-11-99/No Time	5-11-99/No Time	5-11-99/No Time	5-11-99/No Time	5-11-99/No Time
Digestion Date	Not Analyzed	09/10/99	10/13/99	06/25/99	NA
Analysis Date	Battelle Lab	09/10/99	10/29/99	06/25/99	Not Provided
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume		5.473g,5.544g	0.025 g	0.025 g	80
Total Volume		200ul	0.025 g	0.025 g	80
Sample Volume Analyzed		aliquot	Aliquot	0.025 g	80
Dilution		Blending Dilution	1	NA	NA
Results		ND	179.5	0.9644	0.071
Measuring Unit	ppm	ng/g	ug/g	%	g/mL
EPA Method	CVAF	CVAF	EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst		JL	Angel	SG/JL	SG/JL
Method Detection limit		0.2 ng/g	0.06		0.001 g/cc
Data Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	Yes
All Calculation Checked		Yes	X	X	X
Holding Time Met	28 days	No (Goal Only	No (Goal Only)	NA	NA
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID		91099	10-29-99 C-1	06/25/99	Not Provided
Method Blanks		NA	NA in Soils	NA	NA
Instrument Blanks		yes	0.0, -0.1	NA	NA
Duplicates (RPD)		0	<20 RPD	<20 RPD	None Reported
Matrix Spike Recoveries (75-125)		36.57	86 - 91%R	NA	NA
Calibration % R (high and low range)		good	93 - 101%R	NA	NA
Detection Range		0.2 ng/g and >	60 ppm and >	NA	NA
Correlation Coefficient (>0.995)		0.9957	0.9962	NA	NA
QC Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	Yes
All Calculation Checked		Yes	X	X	X
QC Limits Met	Battelle Lab	"M"			"DQO (NR)"

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time. Holding time is a goal only.

Some analysis for AFDW were Re-Run based on negative numbers reported, but no QA/QC batches were re-run.

No duplicate/replicate measurements were taken with the Bulk Density analysis.

For MeHg samples, a matrix spike is performed with every sample and is used to correct the results for loss of analyte.

Station ID M4-809-SFF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	X
Matrix	X	X	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage		Hg lab	Not Noted	Not Noted	Not Noted
Log-in Procedures	Internal COC	Internal COC	Internal COC	Internal COC	Internal COC
Raw Data (Attached)					
Sample Work Sheets		Yes	X	X	X
Sample Run Logs		Yes	X	X	X
Instrument Raw Data		Yes	X	NA	NA
Bench Sheets		Yes	X	X	X
Sample Preparation Logs		Yes	X	X	X
Raw Data (Verified)					
Sample ID Transferred		Yes	X	X	X
All Calculation Checked		Yes	X	X	X
Measuring Unit		ng/g	ug/g	%	g/mL
PE Results (Attached)					
Organization	NA	NA	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA	NA	NA
Validation Criteria					
Applied Qualifiers		"M"			"DQO (NR)"

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

" M " Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

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" NR " Data was unavailable for review.

" DQO " Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

" X " = Attached or Verified

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M4-872-SFF Soil/Sediment

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	5-8-99/No Time	5-8-99/No Time	5-8-99/No Time	5-8-99/No Time	5-8-99/No Time
Digestion Date	Not Analyzed	03/28/00	10/13/99	06/25/99	NA
Analysis Date	Battelle Lab	03/28/00	10/29/99	06/25/99	Not Provided
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume		4.517, 4.562	0.025 g	0.025 g	80
Total Volume		200ul	0.025 g	0.025 g	80
Sample Volume Analyzed		aliquot	Aliquot	0.025 g	80
Dilution		See Worksheet	1	NA	NA
Results		0.23	154.9	0.628	0.1594
Measuring Unit	ppm	ng/g	ug/g	%	g/mL
EPA Method	CVAF	CVAF	EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst		JL	Angel	SG/JL	SG/JL
Method Detection limit		0.2 ng/g	0.06		0.001 g/cc
Data Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	Yes
All Calculation Checked		Yes	X	X	X
Holding Time Met	28 days	No (Goal Only)	No (Goal Only)	NA	NA
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID		32800b	10-29-99 C-1	06/25/99	Not Provided
Method Blanks		NA	NA in Soils	NA	NA
Instrument Blanks		yes	0.0, -0.1	NA	NA
Duplicates (RPD)		-49.52	<20 RPD	<20 RPD	None Reported
Matrix Spike Recoveries (75-125)		95.92	86 - 91%R	NA	NA
Calibration % R (high and low range)		NR	93 - 101%R	NA	NA
Detection Range		0.2 ng/g and >	60 ppm and >	NA	NA
Correlation Coefficient (>0.995)		NR	0.9962	NA	NA
QC Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	Yes
All Calculation Checked		Yes	X	X	X
QC Limits Met	Battelle Lab	"DQO (NR)"			"NR"

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time. Holding time is a goal only.

Some analysis for AFDW were Re-Run based on negative numbers reported, but no QA/QC batches were re-run.

No duplicate/replicate measurements were taken with the Bulk Density analysis.

For MeHg samples, a matrix spike is performed with every sample and is used to correct the results for loss of analyte.

Station ID M4-872-SFF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	X
Matrix	X	X	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage		Hg lab	Not Noted	Not Noted	Not Noted
Log-in Procedures	Internal COC	Internal COC	Internal COC	Internal COC	Internal COC
Raw Data (Attached)					
Sample Work Sheets		Yes	X	X	X
Sample Run Logs		Yes	X	X	X
Instrument Raw Data		Yes	X	NA	NA
Bench Sheets		Yes	X	X	X
Sample Preparation Logs		Yes	X	X	X
Raw Data (Verified)					
Sample ID Transferred		Yes	X	X	X
All Calculation Checked		Yes	X	X	X
Measuring Unit		ng/g	ug/g	%	g/mL
PE Results (Attached)					
Organization	NA	NA	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA	NA	NA
Validation Criteria					
Applied Qualifiers		"DQO"			"NR"

Data Qualifiers/Footnotes:

" J " Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

" Reject " Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

" R2 " Correlation Coefficient is out of QAPP limits.

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" X " = Attached or Verified

10 % Recalculated Results for Total Mercury in Fish Tissue

Analyzed by Florida International University (SERC) for the May 1999 Dry Season (M4)

Entered by gtc 12/01/99 Checked by njs

Sample	QC Batch	Qualifier Note	Instrument Reading Peak Height	Y Intercept Slope	Hg Concentration (ppt)	Vol. added (mL)	Average Reagent Blank (ng)	Corrected Hg Concentration (ng)	Weight of fish (g)	Hg Concentration ppb	Averaged Result ppb	Averaged Reported Result (ppb)	Detection Limit/*3 ppb	SPK CONC (ppb)	R%	Standard Deviation	Relative Standard Deviation
M4-508-FIF-1	HG01GF1		107.9	0.4200	256.9	0.5	0.386	31.21	0.2244	139.10			3.2/9.6				
	"		109.4	0.4200	260.5	0.5	0.386	31.65	0.2244	141.05	140.08		3.2/9.6				
M4-508-FIF-2	"		36.5	0.4200	86.9	0.5	0.386	10.30	0.1461	70.52			3.2/9.6				
	"		36	0.4200	85.7	0.5	0.386	10.16	0.1461	69.52	70.02		3.2/9.6				
M4-508-FIF-3	"		39.3	0.4200	93.6	1	0.386	5.51	0.0989	55.70			3.2/9.6				
	"		39.1	0.4200	93.1	1	0.386	5.48	0.0989	55.40	55.55		3.2/9.6				
M4-508-FIF-4	"		83.3	0.4200	198.3	1	0.386	12.11	0.0918	131.91			3.2/9.6				
	"		83.6	0.4200	199.0	1	0.386	12.15	0.0918	132.40	132.15		3.2/9.6				
M4-508-FIF-5	"		47.3	0.4200	112.6	1	0.386	6.71	0.1362	49.26			3.2/9.6				
	"		46.9	0.4200	111.7	1	0.386	6.65	0.1362	48.82	49.04		3.2/9.6				
M4-508-FIF-6	"		45.4	0.4200	108.1	1	0.386	6.42	0.0972	66.09			3.2/9.6				
	"		45.7	0.4200	108.8	1	0.386	6.47	0.0972	66.55	66.32		3.2/9.6				
M4-508-FIF-7	"		32.6	0.4200	77.6	1	0.386	4.50	0.1196	37.66			3.2/9.6				
	"		32.3	0.4200	76.9	1	0.386	4.46	0.1196	37.28	37.47	78.66	3.2/9.6				
M4-533-FIF-1	"		93.8	0.4200	223.3	0.2	0.386	67.28	0.3507	191.86			3.2/9.6				
	"		93.8	0.4200	223.3	0.2	0.386	67.28	0.3507	191.86	191.86		3.2/9.6				
M4-533-FIF-2	"		85.7	0.4200	204.0	0.2	0.386	61.44	0.428	143.55			3.2/9.6				
	"		84	0.4200	200.0	0.2	0.386	60.21	0.428	140.69	142.12		3.2/9.6				
M4-533-FIF-3	"		97.6	0.4200	232.4	0.2	0.386	70.03	0.3388	206.69			3.2/9.6				
	"		97.1	0.4200	231.2	0.2	0.386	69.66	0.3388	205.62	206.15		3.2/9.6				
M4-533-FIF-4	"		186	0.4200	442.9	0.5	0.386	54.09	0.2082	259.78			3.2/9.6				
	"		185.5	0.4200	441.7	0.5	0.386	53.94	0.2082	259.07	259.42		3.2/9.6				
M4-533-FIF-5	"		116.7	0.4200	277.9	0.2	0.386	83.80	0.3355	249.79			3.2/9.6				
	"		116.7	0.4200	277.9	0.2	0.386	83.80	0.3355	249.79	249.79		3.2/9.6				
M4-533-FIF-6	"		127.1	0.4200	302.6	1	0.386	18.68	0.1482	126.04			3.2/9.6				
	"		127.4	0.4200	303.3	1	0.386	18.72	0.1482	126.34	126.19		3.2/9.6				
M4-533-FIF-7	"		173.1	0.4200	412.1	1	0.386	25.58	0.0942	271.54			3.2/9.6				
	"		174.5	0.4200	415.5	1	0.386	25.79	0.0942	273.77	272.65	206.88	3.2/9.6				
METHOD BLK-1	"		2.457	0.4200	5.9	1		0.37					3.2/9.6				
	"		1.503	0.4200	3.6	1		0.23			0.30		3.2/9.6				
METHOD BLK-0.5	"		1.98	0.4200	4.7	0.5		0.58					3.2/9.6				
	"		1.264	0.4200	3.0	0.5		0.37			0.48	0.386	3.2/9.6				
Instrument Blank	"		0.3	0.4200	0.7					0.0007	0.0007	0.0007	3.2/9.6				
DORM2#12	"		36.1	0.4200	86.0	0.1	0.386	51.44	0.0098	5249.31			3.2/9.6	4600	#####		
DORM2#12	"		36.3	0.4200	86.4	0.1	0.386	51.73	0.0098	5278.62	5263.97		3.2/9.6	4600	#####		
DORM2#14	"		33.4	0.4200	79.5	0.1	0.386	47.57	0.01	4756.69			3.2/9.6	4600	#####		
DORM2#14	"		33.6	0.4200	80.0	0.1	0.386	47.85	0.01	4785.40	4771.04	5017.50	3.2/9.6	4600	#####	285.08	5.7
CCV-1	"		81.4	0.4200	193.8					0.1938			3.2/9.6	0.2	96.90		
CCV-2	"		82.2	0.4200	195.7					0.1957			3.2/9.6	0.2	97.86		
CCV-3	"		80.1	0.4200	190.7					0.1907			3.2/9.6	0.2	95.36		
CCV-4	"		80.4	0.4200	191.4					0.1914			3.2/9.6	0.2	95.71		
CCV-5	"		80.6	0.4200	191.9					0.1919			3.2/9.6	0.2	95.95		
CCV-6	"		80.4	0.4200	191.4					0.1914			3.2/9.6	0.2	95.71		
CCV-7	"		78.9	0.4200	187.9					0.1879			3.2/9.6	0.2	93.93		
CCV-8	"		80.1	0.4200	190.7					0.1907			3.2/9.6	0.2	95.36		
CCV-9	"		81.5	0.4200	194.0					0.1940			3.2/9.6	0.2	97.02		
CCV10-	"		81.4	0.4200	193.8					0.1938			3.2/9.6	0.2	96.90		
CCV-11	"		79.9	0.4200	190.2					0.1902			3.2/9.6	0.2	95.12		
CCV-12	"		79	0.4200	188.1					0.1881		0.192	3.2/9.6	0.2	94.05	0.0024	1.24

10 % Recalculated Results for Total Mercury in Fish Tissue

Analyzed by Florida International University (SERC) for the May 1999 Dry Season (M4)

Entered by gtc 12/01/99 Checked by njs

Sample	QC Batch	Qualifier Note	Instrument Reading Peak Height	Y Intercept Slope	Hg Concentration (ppt)	Vol. added (mL)	Average Reagent Blank (ng)	Corrected Hg Concentration (ng)	Weight of fish (g)	Hg Concentration ppb	Averaged Result ppb	Averaged Reported Result (ppb)	Detection Limit/*3 ppb	SPK CONC (ppb)	R%	Standard Deviation	Relative Standard Deviation
M4-538-FIF-1	HG02GF1		22.4	0.4213	53.2	1	0.074	3.28	0.1210	27.1			3.2/9.6				
	"		22.2	0.4213	52.7	1	0.074	3.25	0.1210	26.8	26.95		3.2/9.6				
M4-538-FIF-2	"		13.5	0.4213	32.0	0.5	0.074	3.87	0.1413	27.4			3.2/9.6				
	"		13.7	0.4213	32.5	0.5	0.074	3.93	0.1413	27.8	27.58		3.2/9.6				
M4-538-FIF-3	"		29.7	0.4213	70.5	0.5	0.074	8.60	0.2727	31.5			3.2/9.6				
	"		29.5	0.4213	70.0	0.5	0.074	8.54	0.2727	31.3	31.42		3.2/9.6				
M4-538-FIF-4	"		16.5	0.4213	39.2	1	0.074	2.39	0.098	24.4			3.2/9.6				
	"		16.8	0.4213	39.9	1	0.074	2.44	0.098	24.9	24.65		3.2/9.6				
M4-538-FIF-5	"		24.4	0.4213	57.9	1	0.074	3.57	0.1133	31.6			3.2/9.6				
	"		24.3	0.4213	57.7	1	0.074	3.56	0.1133	31.4	31.48		3.2/9.6				
M4-538-FIF-6	"		15	0.4213	35.6	1	0.074	2.17	0.0966	22.5			3.2/9.6				
	"		14.6	0.4213	34.7	1	0.074	2.11	0.0966	21.8	22.14		3.2/9.6				
M4-538-FIF-7	"		15	0.4213	35.6	1	0.074	2.17	0.1019	21.3			3.2/9.6				
	"		14.7	0.4213	34.9	1	0.074	2.12	0.1019	20.8	21.07	26.47	3.2/9.6				
M4-548-FIF-1	"		50.5	0.4213	119.9	1	0.074	7.48	0.0557	134.2			3.2/9.6				
	"		50.9	0.4213	120.8	1	0.074	7.54	0.0557	135.3	134.79		3.2/9.6				
M4-548-FIF-2	"		79.6	0.4213	188.9	1	0.074	11.83	0.0688	171.9			3.2/9.6				
	"		80	0.4213	189.9	1	0.074	11.89	0.0688	172.8	172.37		3.2/9.6				
M4-548-FIF-3	"		30.9	0.4213	73.3	1	0.074	4.55	0.0389	116.9			3.2/9.6				
	"		30.2	0.4213	71.7	1	0.074	4.44	0.0389	114.2	115.54		3.2/9.6				
M4-548-FIF-4	"		19.2	0.4213	45.6	1	0.074	2.80	0.0696	40.2			3.2/9.6				
	"		19.1	0.4213	45.3	1	0.074	2.78	0.0696	40.0	40.08		3.2/9.6				
M4-548-FIF-5	"		41.1	0.4213	97.6	1	0.074	6.07	0.0609	99.7			3.2/9.6				
	"		40.1	0.4213	95.2	1	0.074	5.92	0.0609	97.2	98.48		3.2/9.6				
M4-548-FIF-6	"																
	"																
M4-548-FIF-7	"		25.8	0.4213	61.2	1	0.074	3.78	0.0306	123.7			3.2/9.6				
	"		24.4	0.4213	57.9	1	0.074	3.57	0.0306	116.8	120.24	113.58	3.2/9.6				
Method Blank-1	"		1	0.4213	2.4	1		0.15					3.2/9.6				
	"		0.4	0.4213	0.9	1		0.06			0.10		3.2/9.6				
Method Blank-0.5	"		0.2	0.4213	0.5	0.5		0.06					3.2/9.6				
	"		0.1	0.4213	0.2	0.5		0.03			0.04	0.074	3.2/9.6				
Instrument Blank	"		1	0.4213	2.4					0.0024	0.0024	0.0024	3.2/9.6				
DORM2#3	"		31.7	0.4213	75.2	0.1	0.074	45.30	0.0101	4484.9			3.2/9.6	4600	97.50		
DORM2#3	"		31.3	0.4213	74.3	0.1	0.074	44.73	0.0101	4428.2	4456.58		3.2/9.6	4600	96.27		
DORM2#4	"		36.6	0.4213	86.9	0.1	0.074	52.31	0.0145	3607.7			3.2/9.6	4600	78.43		
DORM2#4	"		36.9	0.4213	87.6	0.1	0.074	52.74	0.0145	3637.3	3622.46	4039.520	3.2/9.6	4600	79.07	589.81	14.6
CCV-1	"		88	0.4213	208.9					0.2089			3.2/9.6	0.2	#####		
CCV-2	"		87.4	0.4213	207.5					0.2075			3.2/9.6	0.2	#####		
CCV-3	"		84.2	0.4213	199.9					0.1999			3.2/9.6	0.2	99.93		
CCV-4	"		85.5	0.4213	202.9					0.2029			3.2/9.6	0.2	#####		
CCV-5	"		86.6	0.4213	205.6					0.2056			3.2/9.6	0.2	#####		
CCV-6	"		86.8	0.4213	206.0					0.2060			3.2/9.6	0.2	#####		
CCV-7	"		86.2	0.4213	204.6					0.2046			3.2/9.6	0.2	#####		
CCV-8	"		87	0.4213	206.5					0.2065			3.2/9.6	0.2	#####		
CCV-9	"		87.8	0.4213	208.4					0.2084			3.2/9.6	0.2	#####		
CCV-10	"		88.1	0.4213	209.1					0.2091			3.2/9.6	0.2	#####		
CCV-11	"		84.6	0.4213	200.8					0.2008			3.2/9.6	0.2	#####		
CCV-12	"		86.1	0.4213	204.4					0.2044			3.2/9.6	0.2	#####		
CCV-13	"		88.4	0.4213	209.8					0.2098			3.2/9.6	0.2	#####		
CCV-14	"		88.9	0.4213	211.0					0.2110		0.206	3.2/9.6	0.2	#####	0.0033	1.62

10 % Recalculated Results for Total Mercury in Fish Tissue

Analyzed by Florida International University (SERC) for the May 1999 Dry Season (M4)

Entered by gtc 12/01/99 Checked by njs

Sample	QC Batch	Qualifier Note	Instrument Reading Peak Height	Y Intercept Slope	Hg Concentration (ppt)	Vol. added (mL)	Average Reagent Blank (ng)	Corrected Hg Concentration (ng)	Weight of fish (g)	Hg Concentration ppb	Averaged Result ppb	Averaged Reported Result (ppb)	Detection Limit/*3 ppb	SPK CONC (ppb)	R%	Standard Deviation	Relative Standard Deviation
M4-556-FIF-1	HG07GF1		39.2	0.3958	99.0	1	0.244	6.00	0.102	58.78			3.2/9.6				
	"		38.4	0.3958	97.0	1	0.244	5.87	0.102	57.53	58.16		3.2/9.6				
M4-556-FIF-2	"		31.9	0.3958	80.6	1	0.244	4.83	0.1084	44.59			3.2/9.6				
	"		31.5	0.3958	79.6	1	0.244	4.77	0.1084	44.00	44.30		3.2/9.6				
M4-556-FIF-3	"		7.1	0.3958	17.9	1	0.244	0.89	0.0293	30.24			3.2/9.6				
	"		6.6	0.3958	16.7	1	0.244	0.81	0.0293	27.53	28.88	43.78	3.2/9.6				
M4-566-FIF-1	"		86.3	0.3958	218.0	1	0.244	13.49	0.0952	141.73			3.2/9.6				
	"		87.3	0.3958	220.6	1	0.244	13.65	0.0952	143.40	142.56		3.2/9.6				
M4-566-FIF-2	"		60.8	0.3958	153.6	0.5	0.244	18.65	0.2015	92.56			3.2/9.6				
	"		61.2	0.3958	154.6	0.5	0.244	18.77	0.2015	93.17	92.87		3.2/9.6				
M4-566-FIF-3	"		35	0.3958	88.4	0.5	0.244	10.63	0.1619	65.67			3.2/9.6				
	"		34.7	0.3958	87.7	0.5	0.244	10.54	0.1619	65.10	65.39		3.2/9.6				
M4-566-FIF-4	"		64.3	0.3958	162.5	0.2	0.244	48.98	0.3513	139.43			3.2/9.6				
	"		64.6	0.3958	163.2	0.2	0.244	49.21	0.3513	140.08	139.75		3.2/9.6				
M4-566-FIF-5	"		91.6	0.3958	231.4	1	0.244	14.34	0.1386	103.43			3.2/9.6				
	"		90.9	0.3958	229.7	1	0.244	14.22	0.1386	102.63	103.03		3.2/9.6				
M4-566-FIF-6	"		47.9	0.3958	121.0	1	0.244	7.38	0.0744	99.20			3.2/9.6				
	"		47.7	0.3958	120.5	1	0.244	7.35	0.0744	98.77	98.98		3.2/9.6				
M4-566-FIF-7	"		58.6	0.3958	148.1	1	0.244	9.08	0.1407	64.56			3.2/9.6				
	"		59	0.3958	149.1	1	0.244	9.15	0.1407	65.01	64.79	101.05	3.2/9.6				
METHOD BLK-1	"		1.9	0.3958	4.8	1		0.30					3.2/9.6				
	"		1.3	0.3958	3.3	1		0.21			0.25		3.2/9.6				
METHOD BLK-0.5	"		0.8	0.3958	2.0	0.5		0.25					3.2/9.6				
	"		0.7	0.3958	1.8	0.5		0.22			0.23	0.244	3.2/9.6				
Instrument Blank	"		1.2	0.3958	3.0					0.00303	0.0030	0.0030	3.2/9.6				
SRM	"		29.2	0.3958	73.8	0.1	0.244	44.24	0.0098	4514.50			3.2/9.6	4600	98.14		
DORM2#5	"		28.8	0.3958	72.8	0.1	0.244	43.63	0.0098	4452.32	4483.41		3.2/9.6	4600	96.79		
SRM	"		28	0.3958	70.7	0.1	0.244	42.41	0.01	4241.39			3.2/9.6	4600	92.20		
DORM2#6+A308	"		27.9	0.3958	70.5	0.1	0.244	42.26	0.01	4226.16	4233.77	4358.59	3.2/9.6	4600	91.87	146.48	3.4
CCV-1	"		77.1	0.3958	194.8					0.1948			3.2/9.6	0.2	97.40		
CCV-2	"		77.1	0.3958	194.8					0.1948			3.2/9.6	0.2	97.40		
CCV-3	"		74.2	0.3958	187.5					0.1875			3.2/9.6	0.2	93.73		
CCV-4	"		74.7	0.3958	188.7					0.1887			3.2/9.6	0.2	94.37		
CCV-5	"		78	0.3958	197.1					0.1971			3.2/9.6	0.2	98.53		
CCV-6	"		70.2	0.3958	177.4					0.1774			3.2/9.6	0.2	88.68		
CCV-7	"		69.7	0.3958	176.1					0.1761			3.2/9.6	0.2	88.05		
CCV-8	"		70.4	0.3958	177.9					0.1779			3.2/9.6	0.2	88.93		
CCV-9	"		78.2	0.3958	197.6					0.1976			3.2/9.6	0.2	98.79		
CCV10-	"		78.5	0.3958	198.3					0.1983			3.2/9.6	0.2	99.17		
CCV-11	"		78.4	0.3958	198.1					0.1981			3.2/9.6	0.2	99.04		
CCV-12	"		78.1	0.3958	197.3					0.1973			3.2/9.6	0.2	98.66		
CCV-13	"		75	0.3958	189.5					0.1895			3.2/9.6	0.2	94.74		
CCV-14	"		74.3	0.3958	187.7					0.1877			3.2/9.6	0.2	93.86		
CCV-15	"		79.4	0.3958	200.6					0.2006			3.2/9.6	0.2	#####		
CCV-16	"		78.7	0.3958	198.8					0.1988			3.2/9.6	0.2	99.42		
CCV-17	"		77.6	0.3958	196.1					0.1961			3.2/9.6	0.2	98.03		
CCV-18	"		78.2	0.3958	197.6					0.1976		0.192	3.2/9.6	0.2	98.79	0.0079	4.13

RR = Rerun

10 % Recalculated Results for Total Mercury in Fish Tissue

Analyzed by Florida International University (SERC) for the May 1999 Dry Season (M4)

Entered by gtc 12/01/99 Checked by njs

Sample	QC Batch	Qualifier Note	Instrument Reading Peak Height	Y Intercept Slope	Hg Concentration (ppt)	Vol. added (mL)	Average Reagent Blank (ng)	Corrected Hg Concentration (ng)	Weight of fish (g)	Hg Concentration ppb	Averaged Result ppb	Averaged Reported Result (ppb)	Detection Limit/*3 ppb	SPK CONC (ppb)	R%	Standard Deviation	Relative Standard Deviation
M4-568-FIF-1	HG08GF1		104.83	0.4207	249.2	1	0.997	14.70	0.141	104.26			3.2/9.6				
	"		100.55	0.4207	239.0	1	0.997	14.06	0.141	99.72	101.99		3.2/9.6				
M4-568-FIF-2	"		76.78	0.4207	182.5	1	0.997	10.50	0.0919	114.26			3.2/9.6				
	"		76.78	0.4207	182.5	1	0.997	10.50	0.0919	114.26	114.26		3.2/9.6				
M4-568-FIF-3	"		90.09	0.4207	214.1	1	0.997	12.49	0.069	181.07			3.2/9.6				
	"		89.86	0.4207	213.6	1	0.997	12.46	0.069	180.57	180.82		3.2/9.6				
M4-568-FIF-4	"		70.13	0.4207	166.7	1	0.997	9.50	0.0942	100.90			3.2/9.6				
	"		70.6	0.4207	167.8	1	0.997	9.58	0.0942	101.65	101.28		3.2/9.6				
M4-568-FIF-5	"		65.85	0.4207	156.5	1	0.997	8.86	0.0917	96.66			3.2/9.6				
	"		65.37	0.4207	155.4	1	0.997	8.79	0.0917	95.88	96.27		3.2/9.6				
M4-568-FIF-6	"		50.87	0.4207	120.9	1	0.997	6.62	0.0701	94.45			3.2/9.6				
	"		50.87	0.4207	120.9	1	0.997	6.62	0.0701	94.45	94.45		3.2/9.6				
M4-568-FIF-7	"		66.56	0.4207	158.2	1	0.997	8.97	0.0538	166.74			3.2/9.6				
	"		66.32	0.4207	157.6	1	0.997	8.93	0.0538	166.07	166.40	122.21	3.2/9.6				
METHOD BLK-1	"		8.2	0.4207	19.5	1		1.23					3.2/9.6				
	"		7.9	0.4207	18.8	1		1.18			1.21		3.2/9.6				
METHOD BLK-0.5	"		2.5	0.4207	5.9	0.5		0.73					3.2/9.6				
	"		2.9	0.4207	6.9	0.5		0.85			0.79	0.997	3.2/9.6				
Instrument Blank	"		1.2	0.4207	2.9					0.0029	0.0029	0.0029	3.2/9.6				
DORM2#12	"		42.3	0.4207	100.5	0.1	0.997	59.63	0.0118	5053.62			3.2/9.6	4600	#####		
DORM2#12	"		42.9	0.4207	102.0	0.1	0.997	60.49	0.0118	5126.50	5090.06		3.2/9.6	4600	#####		
DORM2#14	"		38.3	0.4207	91.0	0.1	0.997	53.90	0.0116	4646.50			3.2/9.6	4600	#####		
DORM2#14	"		39	0.4207	92.7	0.1	0.997	54.90	0.0116	4732.99	4689.74	4889.90	3.2/9.6	4600	#####	235.69	4.8
CCV-1	"		87.6	0.4207	208.2					0.2082			3.2/9.6	0.2	#####		
CCV-2	"		88.2	0.4207	209.7					0.2097			3.2/9.6	0.2	#####		
CCV-3	"		84.1	0.4207	199.9					0.1999			3.2/9.6	0.2	99.95		
CCV-4	"		83.8	0.4207	199.2					0.1992			3.2/9.6	0.2	99.60		
CCV-5	"		84.5	0.4207	200.9					0.2009			3.2/9.6	0.2	#####		
CCV-6	"		85.7	0.4207	203.7					0.2037			3.2/9.6	0.2	#####		
CCV-7	"		85.8	0.4207	203.9					0.2039			3.2/9.6	0.2	#####		
CCV-8	"		85.2	0.4207	202.5					0.2025			3.2/9.6	0.2	#####		
CCV-9	"		77	0.4207	183.0					0.1830			3.2/9.6	0.2	91.51		
CCV10-	"		77.2	0.4207	183.5					0.1835			3.2/9.6	0.2	91.75		
CCV-11	"		90	0.4207	213.9					0.2139			3.2/9.6	0.2	#####		
CCV-12	"		90.7	0.4207	215.6					0.2156			3.2/9.6	0.2	#####		
CCV-13	"		83.4	0.4207	198.2					0.1982			3.2/9.6	0.2	99.12		
CCV-14	"		82.5	0.4207	196.1					0.1961		0.201	3.2/9.6	0.2	98.05	0.0096	4.75

RR = Rerun

10 % Recalculated Results for Total Mercury in Fish Tissue

Analyzed by Florida International University (SERC) for the May 1999 Dry Season (M4)

Entered by gtc 12/01/99 Checked by njs

Sample	QC Batch	Qualifier Note	Instrument Reading Peak Height	Y Intercept Slope	Hg Concentration (ppt)	Vol. added (mL)	Average Reagent Blank (ng)	Corrected Hg Concentration (ng)	Weight of fish (g)	Hg Concentration ppb	Averaged Result ppb	Averaged Reported Result (ppb)	Detection Limit/*3 ppb	SPK CONC (ppb)	R%	Standard Deviation	Relative Standard Deviation
M4-576-FIF-1	HG12GF1		63.9	0.4396	145.4	0.2	0.071	43.97	0.3209	137.03			3.2/9.6				
	"		65.5	0.4396	149.0	0.2	0.071	45.08	0.3209	140.47	138.75		3.2/9.6				
M4-576-FIF-2	"		147.2	0.4396	334.8	0.2	0.071	101.39	0.3221	314.77			3.2/9.6				
	"		147.8	0.4396	336.2	0.2	0.071	101.80	0.3221	316.06	315.42		3.2/9.6				
M4-576-FIF-3	"		94.8	0.4396	215.7	0.5	0.071	26.45	0.1871	141.39			3.2/9.6				
	"		89.6	0.4396	203.8	0.5	0.071	25.00	0.1871	133.61	137.50		3.2/9.6				
M4-576-FIF-4	"		145.7	0.4396	331.4	0.2	0.071	100.35	0.3248	308.97			3.2/9.6				
	"		148.6	0.4396	338.0	0.2	0.071	102.35	0.3248	315.13	312.05		3.2/9.6				
M4-576-FIF-5	"		71.7	0.4396	163.1	0.2	0.071	49.35	0.2094	235.67			3.2/9.6				
	"		70.1	0.4396	159.5	0.2	0.071	48.25	0.2094	230.40	233.04		3.2/9.6				
M4-576-FIF-6	"		133.7	0.4396	304.1	0.2	0.071	92.08	0.2676	344.11			3.2/9.6				
	"		136.4	0.4396	310.3	0.2	0.071	93.94	0.2676	351.06	347.59		3.2/9.6				
M4-576-FIF-7	"		115.5	0.4396	262.7	0.2	0.071	79.54	0.3355	237.08			3.2/9.6				
	"		111.1	0.4396	252.7	0.2	0.071	76.51	0.3355	228.04	232.56	245.27	3.2/9.6				
M4-594-FIF-2	"		151.1	0.4396	343.7	0.5	0.071	42.21	0.218	193.61			3.2/9.6				
	"		150.7	0.4396	342.8	0.5	0.071	42.09	0.218	193.10	193.35		3.2/9.6				
M4-594-FIF-3	"		114.7	0.4396	260.9	0.5	0.071	32.02	0.1744	183.61			3.2/9.6				
	"		108.5	0.4396	246.8	0.5	0.071	30.29	0.1744	173.67	178.64		3.2/9.6				
M4-594-FIF-4	"		127.2	0.4396	289.4	1	0.071	18.16	0.0729	249.09			3.2/9.6				
	"		128.3	0.4396	291.9	1	0.071	18.32	0.0729	251.25	250.17		3.2/9.6				
M4-594-FIF-5	"		89.2	0.4396	202.9	1	0.071	12.71	0.0945	134.52			3.2/9.6				
	"		87.7	0.4396	199.5	1	0.071	12.50	0.0945	132.25	133.39		3.2/9.6				
M4-594-FIF-6	"		81.3	0.4396	184.9	1	0.071	11.58	0.0787	147.14			3.2/9.6				
	"		81.6	0.4396	185.6	1	0.071	11.62	0.0787	147.69	147.42		3.2/9.6				
M4-594-FIF-7	"		144.2	0.4396	328.0	1	0.071	20.59	0.1148	179.40			3.2/9.6				
	"		144.5	0.4396	328.7	1	0.071	20.64	0.1148	179.77	179.58	180.42	3.2/9.6				
METHOD BLK-1	"		0.8	0.4396	1.8	1		0.11					3.2/9.6				
	"		0.2	0.4396	0.5	1		0.03			0.07		3.2/9.6				
METHOD BLK-0.5	"		0.2	0.4396	0.5	0.5		0.06					3.2/9.6				
	"		0.3	0.4396	0.7	0.5		0.08			0.07	0.071	3.2/9.6				
Instrument Blank	"		1.5	0.4396	3.4					0.0034	0.0034	0.0034	3.2/9.6				
DORM2#12	"		38.7	0.4396	88.0	0.1	0.071	53.01	0.0116	4570.2			3.2/9.6	4600	99.35		
DORM2#12	"		38.8	0.4396	88.3	0.1	0.071	53.15	0.0116	4582.0	4576.07		3.2/9.6	4600	99.61		
DORM2#14	"		40	0.4396	91.0	0.1	0.071	54.80	0.0119	4604.8			3.2/9.6	4600	#####		
DORM2#14	"		39.6	0.4396	90.1	0.1	0.071	54.25	0.0119	4558.7	4581.74	4578.91	3.2/9.6	4600	99.10	4.01	0.1
CCV-1	"		77.6	0.4396	176.5					0.1765			3.2/9.6	0.2	88.26		
CCV-2	"		77.6	0.4396	176.5					0.1765			3.2/9.6	0.2	88.26		
CCV-3	"		77.2	0.4396	175.6					0.1756			3.2/9.6	0.2	87.81		
CCV-4	"		78.8	0.4396	179.3					0.1793			3.2/9.6	0.2	89.63		
CCV-5	"		78.3	0.4396	178.1					0.1781			3.2/9.6	0.2	89.06		
CCV-6	"		79.7	0.4396	181.3					0.1813			3.2/9.6	0.2	90.65		
CCV-7	"		77.2	0.4396	175.6					0.1756			3.2/9.6	0.2	87.81		
CCV-8	"		76.4	0.4396	173.8					0.1738			3.2/9.6	0.2	86.90		
CCV-9	"		74.7	0.4396	169.9					0.1699			3.2/9.6	0.2	84.96		
CCV10-	"		74.2	0.4396	168.8					0.1688			3.2/9.6	0.2	84.39		
CCV-11	"		85.9	0.4396	195.4					0.1954			3.2/9.6	0.2	97.70		
CCV-12	"		85.6	0.4396	194.7					0.1947			3.2/9.6	0.2	97.36		
CCV-13	"		82.5	0.4396	187.7					0.1877			3.2/9.6	0.2	93.84		
CCV-14	"		81.6	0.4396	185.6					0.1856			3.2/9.6	0.2	92.81		
CCV-15	"		82	0.4396	186.5					0.1865			3.2/9.6	0.2	93.27		
CCV-16	"		81.2	0.4396	184.7					0.1847			3.2/9.6	0.2	92.36		
CCV-17	"		83.7	0.4396	190.4					0.1904			3.2/9.6	0.2	95.20		
CCV-18	"		83.5	0.4396	189.9					0.1899			3.2/9.6	0.2	94.97		
CCV-19	"		79.5	0.4396	180.8					0.1808			3.2/9.6	0.2	90.42		
CCV-20	"		79.5	0.4396	180.8					0.1808		0.182	3.2/9.6	0.2	90.42	0.0076	4.19

10 % Recalculated Results for Total Mercury in Fish Tissue

Analyzed by Florida International University (SERC) for the May 1999 Dry Season (M4)

Entered by gtc 12/01/99 Checked by njs

Sample	QC Batch	Qualifier Note	Instrument Reading Peak Height	Y Intercept Slope	Hg Concentration (ppt)	Vol. added (mL)	Average Reagent Blank (ng)	Corrected Hg Concentration (ng)	Weight of fish (g)	Hg Concentration ppb	Averaged Result ppb	Averaged Reported Result (ppb)	Detection Limit/*3 ppb	SPK CONC (ppb)	R%	Standard Deviation	Relative Standard Deviation
M4-599-FIF-1	HG13GF1		97.7	0.4347	224.8	0.2	1.858	66.24	0.2005	330.38			3.2/9.6				
	"		97.6	0.4347	224.5	0.2	1.858	66.17	0.2005	330.04	330.21		3.2/9.6				
M4-599-FIF-2	"		143.3	0.4347	329.7	1	1.858	18.91	0.1279	147.85			3.2/9.6				
	"		142.6	0.4347	328.0	1	1.858	18.81	0.1279	147.06	147.45		3.2/9.6				
M4-599-FIF-3	"		143.4	0.4347	329.9	0.5	1.858	38.72	0.116	333.77			3.2/9.6				
	"		150.9	0.4347	347.1	0.5	1.858	40.84	0.116	352.07	342.92		3.2/9.6				
M4-599-FIF-4	"		122.2	0.4347	281.1	0.2	1.858	83.32	0.2133	390.62			3.2/9.6				
	"		124.3	0.4347	285.9	0.2	1.858	84.78	0.2133	397.48	394.05		3.2/9.6				
M4-599-FIF-5	"		140.6	0.4347	323.4	0.2	1.858	96.14	0.3574	269.01			3.2/9.6				
	"		141.7	0.4347	326.0	0.2	1.858	96.91	0.3574	271.16	270.08		3.2/9.6				
M4-599-FIF-6	"		104.2	0.4347	239.7	0.2	1.858	70.77	0.2894	244.55			3.2/9.6				
	"		104.9	0.4347	241.3	0.2	1.858	71.26	0.2894	246.24	245.39		3.2/9.6				
M4-599-FIF-7	"		123.7	0.4347	284.6	0.5	1.858	33.14	0.119	278.52			3.2/9.6				
	"		123.5	0.4347	284.1	0.5	1.858	33.09	0.119	278.04	278.28	286.91	3.2/9.6				
M4-809-FIF-1	"		47.6	0.4347	109.5	0.2	1.858	31.32	0.2891	108.34			3.2/9.6				
	"		46.7	0.4347	107.4	0.2	1.858	30.69	0.2891	106.17	107.25		3.2/9.6				
M4-809-FIF-2	"		38.9	0.4347	89.5	0.2	1.858	25.26	0.2561	98.62			3.2/9.6				
	"		39.8	0.4347	91.6	0.2	1.858	25.88	0.2561	101.07	99.84		3.2/9.6				
M4-809-FIF-3	"		79.4	0.4347	182.7	0.5	1.858	20.61	0.1951	105.63			3.2/9.6				
	"		80.3	0.4347	184.7	0.5	1.858	20.86	0.1951	106.94	106.28		3.2/9.6				
M4-809-FIF-4	"		46.5	0.4347	107.0	0.5	1.858	11.30	0.1553	72.76			3.2/9.6				
	"		45.8	0.4347	105.4	0.5	1.858	11.10	0.1553	71.48	72.12		3.2/9.6				
M4-809-FIF-5	"		44.4	0.4347	102.1	0.2	1.858	29.09	0.2348	123.89			3.2/9.6				
	"		44.9	0.4347	103.3	0.2	1.858	29.44	0.2348	125.38	124.64		3.2/9.6				
M4-809-FIF-6	"		43.2	0.4347	99.4	0.2	1.858	28.25	0.321	88.02			3.2/9.6				
	"		43.9	0.4347	101.0	0.2	1.858	28.74	0.321	89.54	88.78		3.2/9.6				
M4-809-FIF-7	"		116.6	0.4347	268.2	0.5	1.858	31.13	0.1989	156.53			3.2/9.6				
	"		116.6	0.4347	268.2	0.5	1.858	31.13	0.1989	156.53	156.53	107.92	3.2/9.6				
METHOD BLK-1	"		13.2	0.4347	30.4	1		1.91					3.2/9.6				
	"		12.7	0.4347	29.2	1		1.84			1.88		3.2/9.6				
METHOD BLK-0.5	"		6.4	0.4347	14.7	0.5		1.81					3.2/9.6				
	"		6.6	0.4347	15.2	0.5		1.87			1.84	1.858	3.2/9.6				
Instrument Blank	"		0.9	0.4347	2.1					0.0021	0.0021	0.0021	3.2/9.6				
DORM2#16	"		37	0.4347	85.1	0.1	1.858	49.47	0.0119	4156.9			3.2/9.6	4600	90.37		
DORM2#16	"		37	0.4347	85.1	0.1	1.858	49.47	0.0119	4156.9	4156.90		3.2/9.6	4600	90.37		
DORM2#17	"		40	0.4347	92.0	0.1	1.858	53.63	0.0121	4432.1			3.2/9.6	4600	96.35		
DORM2#17	"		40.3	0.4347	92.7	0.1	1.858	54.04	0.0121	4466.5	4449.31	4303.101	3.2/9.6	4600	97.10	206.77	4.8
CCV-1	"		88.1	0.4347	202.7					0.2027			3.2/9.6	0.2	#####		
CCV-2	"		88.6	0.4347	203.8					0.2038			3.2/9.6	0.2	#####		
CCV-3	"		87.7	0.4347	201.7					0.2017			3.2/9.6	0.2	#####		
CCV-4	"		87.2	0.4347	200.6					0.2006			3.2/9.6	0.2	#####		
CCV-5	"		82.2	0.4347	189.1					0.1891			3.2/9.6	0.2	94.55		
CCV-6	"		81.7	0.4347	187.9					0.1879			3.2/9.6	0.2	93.97		
CCV-7	"		85.9	0.4347	197.6					0.1976			3.2/9.6	0.2	98.80		
CCV-8	"		85.5	0.4347	196.7					0.1967			3.2/9.6	0.2	98.34		
CCV-9	"		79.3	0.4347	182.4					0.1824			3.2/9.6	0.2	91.21		
CCV-10	"		79.9	0.4347	183.8					0.1838			3.2/9.6	0.2	91.90		
CCV-11	"		81	0.4347	186.3					0.1863			3.2/9.6	0.2	93.17		
CCV-12	"		81.8	0.4347	188.2					0.1882			3.2/9.6	0.2	94.09		
CCV-13	"		81.5	0.4347	187.5					0.1875			3.2/9.6	0.2	93.74		
CCV-14	"		81.1	0.4347	186.6					0.1866		0.192	3.2/9.6	0.2	93.28	0.0076	3.95

RR = Rerun

10 % Recalculated Results for Total Mercury in Fish Tissue

Analyzed by Florida International University (SERC) for the May 1999 Dry Season (M4)

Entered by gtc 12/01/99 Checked by njs

Sample	QC Batch	Qualifier Note	Instrument Reading Peak Height	Y Intercept Slope	Hg Concentration (ppt)	Vol. added (mL)	Average Reagent Blank (ng)	Corrected Hg Concentration (ng)	Weight of fish (g)	Hg Concentration ppb	Averaged Result ppb	Averaged Reported Result (ppb)	Detection Limit/*3 ppb	SPK CONC (ppb)	R%	Standard Deviation	Relative Standard Deviation
M4-872-FIF-1	HG22GF1		98.8	0.4589	215.3	0.5	0.658	25.82	0.131	197.13			3.2/9.6				
	"		99.6	0.4589	217.0	0.5	0.658	26.04	0.131	198.76	197.95		3.2/9.6				
M4-872-FIF-2	"		141.2	0.4589	307.7	0.5	0.658	37.19	0.2128	174.76			3.2/9.6				
	"		139.8	0.4589	304.6	0.5	0.658	36.81	0.2128	172.99	173.87		3.2/9.6				
M4-872-FIF-3	"		87.9	0.4589	191.5	0.5	0.658	22.90	0.1423	160.94			3.2/9.6				
	"		87.9	0.4589	191.5	0.5	0.658	22.90	0.1423	160.94	160.94		3.2/9.6				
M4-872-FIF-4	"		120.2	0.4589	261.9	0.5	0.658	31.56	0.1774	177.90			3.2/9.6				
	"		120	0.4589	261.5	0.5	0.658	31.51	0.1774	177.60	177.75		3.2/9.6				
M4-872-FIF-5	"		46.9	0.4589	102.2	1	0.658	5.78	0.0742	77.91			3.2/9.6				
	"		46.5	0.4589	101.3	1	0.658	5.73	0.0742	77.17	77.54		3.2/9.6				
M4-872-FIF-6	"		73.3	0.4589	159.7	1	0.658	9.40	0.0517	181.91			3.2/9.6				
	"		72.6	0.4589	158.2	1	0.658	9.31	0.0517	180.06	180.99		3.2/9.6				
M4-872-FIF-7	"		98	0.4589	213.6	1	0.658	12.80	0.0704	181.76			3.2/9.6				
	"		98.8	0.4589	215.3	1	0.658	12.91	0.0704	183.32	182.54	164.51	3.2/9.6				
METHOD BLK-1	"		5	0.4589	10.9	1		0.69					3.2/9.6				
	"		4.8	0.4589	10.5	1		0.66			0.67		3.2/9.6				
METHOD BLK-0.5	"		2.3	0.4589	5.0	0.5		0.62					3.2/9.6				
	"		2.5	0.4589	5.4	0.5		0.67			0.64	0.658	3.2/9.6				
Instrument Blank	"		1.3	0.4589	2.8					0.0028	0.0028	0.0028	3.2/9.6				
DORM2#18	"		32.9	0.4589	71.7	0.1	0.658	42.57	0.0101	4215.1			3.2/9.6	4600	91.63		
DORM2#18	"		32.6	0.4589	71.0	0.1	0.658	42.18	0.0101	4176.1	4195.63		3.2/9.6	4600	90.79		
DORM2#19	"		39.8	0.4589	86.7	0.1	0.658	51.64	0.0145	3561.4			3.2/9.6	4600	77.42		
DORM2#19	"		39.8	0.4589	86.7	0.1	0.658	51.64	0.0145	3561.4	3561.36	3878.494	3.2/9.6	4600	77.42	448.50	11.6
CCV-1	"		91.2	0.4589	198.7					0.1987			3.2/9.6	0.2	99.37		
CCV-2	"		91.8	0.4589	200.0					0.2000			3.2/9.6	0.2	#####		
CCV-3	"		90.6	0.4589	197.4					0.1974			3.2/9.6	0.2	98.71		
CCV-4	"		90.6	0.4589	197.4					0.1974			3.2/9.6	0.2	98.71		
CCV-5	"		89.1	0.4589	194.2					0.1942			3.2/9.6	0.2	97.08		
CCV-6	"		89.5	0.4589	195.0					0.1950			3.2/9.6	0.2	97.52		
CCV-7	"		89.2	0.4589	194.4					0.1944			3.2/9.6	0.2	97.19		
CCV-8	"		90.9	0.4589	198.1					0.1981			3.2/9.6	0.2	99.04		
CCV-9	"		88.9	0.4589	193.7					0.1937			3.2/9.6	0.2	96.86		
CCV-10	"		89.7	0.4589	195.5					0.1955			3.2/9.6	0.2	97.73		
CCV-11	"		89.1	0.4589	194.2					0.1942			3.2/9.6	0.2	97.08		
CCV-12	"		89	0.4589	193.9					0.1939			3.2/9.6	0.2	96.97		
CCV-13	"		88.7	0.4589	193.3					0.1933			3.2/9.6	0.2	96.64		
CCV-14	"		89	0.4589	193.9					0.1939		0.196	3.2/9.6	0.2	96.97	0.0022	1.12

RR = Rerun

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID **M4-501-FIF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	No Samples were Collected	No Samples were Collected	No Samples were Collected
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	No Samples were Collected	No Samples were Collected	No Samples were Collected
Digestion Date			
Analysis Date			
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume			
Total Volume			
Sample Volume Analyzed			
Dilution			
Results			
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	No Sample	No Sample	No Sample
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst			
All Calculation Checked			
Holding Time Met			
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID		By: Date/Time	By: Date/Time
Method Blanks			
Instrument Blanks			
Duplicates (RPD)			
Matrix Spike Recoveries (75-125)			
Blank Spike/CCV Recoveries			
Detection Range			
Correlation Coefficient (>0.995)			
QC Report (Verified)			
Lab Data Entry Checked by Analyst			
All Calculation Checked			
QC Limits Met			

Notes

Samples were not collected and/or delivered to the SERC Laboratory.

Station ID **M4-501-FIF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records	No Chain of Custody---------- Sample log summary only		
Sampling Location ID			
Matrix			
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets			
Sample Run Logs			
Instrument Raw Data			
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs			
Raw Data (Verified)			
Sample ID Transferred			
All Calculation Checked			
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers	No Samples were Collected	No Samples were Collected	No Samples were Collected

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID **M4-508-FIF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	X	X	X
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	5-11-99/No Time	5-11-99/No Time	5-11-99/No Time
Digestion Date	06/03/99	06/03/99	06/03/99
Analysis Date	07/01/99	06/03/99	06/03/99
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume	7 Fish	NA	NA
Total Volume	7 Fish	7 Fish	7 Fish
Sample Volume Analyzed	Aliquot	7 Fish	7 Fish
Dilution	500-1000	NA	NA
Results	89.7 (Average of 7)	22-30	0.0989 - 0.2244
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	FDL/JL	MB	MB
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst	No	No	No
All Calculation Checked	X	X	X
Holding Time Met	Yes (28 day Holding Time)	No (Goal Only)	No (Goal Only)
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID	HG01GF1	By: Date/Time	By: Date/Time
Method Blanks	0.386	NA	NA
Instrument Blanks	0.0007	NA	NA
Duplicates (RPD)	<20 RPD	NA	NA
Matrix Spike Recoveries (75-125)	DORM 103 - 115%R	NA	NA
Blank Spike/CCV Recoveries	93 - 98%R	NA	NA
Detection Range	3.2 ppb and >	NA	NA
Correlation Coefficient (>0.995)	0.9999	NA	NA
QC Report (Verified)			
Lab Data Entry Checked by Analyst	No	No	No
All Calculation Checked	X	X	X
QC Limits Met			

Notes

No descriptive narratives were provided by FIU.

Holding time goals set for the length and weight measurements are guidelines only, not a hard deadline.

Measurements of fish length and weight were measured after holding time goals.

Documentation on how the fish samples were stored (Frozen, 4 C, preserved) was not provided.

Station ID **M4-508-FIF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID **M4-533-FIF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	X	X	X
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	5-11-99/No Time	5-11-99/No Time	5-11-99/No Time
Digestion Date	06/03/99	06/03/99	06/03/99
Analysis Date	07/01/99	06/03/99	06/03/99
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume	7 Fish	NA	NA
Total Volume	7 Fish	7 Fish	7 Fish
Sample Volume Analyzed	Aliquot	7 Fish	7 Fish
Dilution	200-1000	NA	NA
Results	206.9 (Average of 7)	21-32	0.0942 - 0.428
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	FDL/JL	MB	MB
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst	No	No	No
All Calculation Checked	X	X	X
Holding Time Met	Yes (28 day Holding Time)	No (Goal Only)	No (Goal Only)
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID	HG01GF1	By: Date/Time	By: Date/Time
Method Blanks	0.386	NA	NA
Instrument Blanks	0.0007	NA	NA
Duplicates (RPD)	<20 RPD	NA	NA
Matrix Spike Recoveries (75-125)	DORM 103 - 115%R	NA	NA
Blank Spike/CCV Recoveries	93 - 98%R	NA	NA
Detection Range	3.2 ppb and >	NA	NA
Correlation Coefficient (>0.995)	0.9999	NA	NA
QC Report (Verified)			
Lab Data Entry Checked by Analyst	No	No	No
All Calculation Checked	X	X	X
QC Limits Met			

Notes

No descriptive narratives were provided by FIU.

Holding time goals set for the length and weight measurements are guidelines only, not a hard deadline.

Measurements of fish length and weight were measured after holding time goals.

Documentation on how the fish samples were stored (Frozen, 4 C, preserved) was not provided.

Station ID **M4-533-FIF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID **M4-538-FIF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	X	X	X
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	5-10-99/No Time	5-10-99/No Time	5-10-99/No Time
Digestion Date	06/03/99	06/03/99	06/03/99
Analysis Date	07/02/99	06/03/99	06/03/99
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume	7 Fish	NA	NA
Total Volume	7 Fish	7 Fish	7 Fish
Sample Volume Analyzed	Aliquot	7 Fish	7 Fish
Dilution	500-1000	NA	NA
Results	26.47 (Average of 7)	20 - 25	0.0966 - 0.2727
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	FDL/JL	MB	MB
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst	No	No	No
All Calculation Checked	X	X	X
Holding Time Met	Yes (28 day Holding Time)	No (Goal Only)	No (Goal Only)
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID	HG02GF1	By: Date/Time	By: Date/Time
Method Blanks	0.074	NA	NA
Instrument Blanks	0.0024	NA	NA
Duplicates (RPD)	<20 RSD	NA	NA
Matrix Spike Recoveries (75-125)	78 - 98%R	NA	NA
Blank Spike/CCV Recoveries	100 - 106%R	NA	NA
Detection Range	3.2 ppb and >	NA	NA
Correlation Coefficient (>0.995)	0.9991	NA	NA
QC Report (Verified)			
Lab Data Entry Checked by Analyst	No	No	No
All Calculation Checked	X	X	X
QC Limits Met			

Notes

No descriptive narratives were provided by FIU.

Holding time goals set for the length and weight measurements are guidelines only, not a hard deadline.

Measurements of fish length and weight were measured after holding time goals.

Documentation on how the fish samples were stored (Frozen, 4 C, preserved) was not provided.

Station ID **M4-538-FIF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID **M4-548-FIF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	X	X	X
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	5-9-99/No Time	5-9-99/No Time	5-9-99/No Time
Digestion Date	06/04/99	06/04/99	06/04/99
Analysis Date	07/02/99	06/04/99	06/04/99
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume	6 Fish	NA	NA
Total Volume	6 Fish	7 Fish	7 Fish
Sample Volume Analyzed	Aliquot	7 Fish	7 Fish
Dilution	1000	NA	NA
Results	113.6 (Average of 6)	15 - 23	0.0306 - 0.0696
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	FDL/JL	MB	MB
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst	No	No	No
All Calculation Checked	X	X	X
Holding Time Met	Yes (28 day Holding Time)	No (Goal Only)	No (Goal Only)
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID	HG02GF1	By: Date/Time	By: Date/Time
Method Blanks	0.074	NA	NA
Instrument Blanks	0.0024	NA	NA
Duplicates (RPD)	<20 RSD	NA	NA
Matrix Spike Recoveries (75-125)	78 - 98%R	NA	NA
Blank Spike/CCV Recoveries	100 - 106%R	NA	NA
Detection Range	3.2 ppb and >	NA	NA
Correlation Coefficient (>0.995)	0.9991	NA	NA
QC Report (Verified)			
Lab Data Entry Checked by Analyst	No	No	No
All Calculation Checked	X	X	X
QC Limits Met			

Notes

No descriptive narratives were provided by FIU.

Holding time goals set for the length and weight measurements are guidelines only, not a hard deadline.

Measurements of fish length and weight were measured after holding time goals.

Documentation on how the fish samples were stored (Frozen, 4 C, preserved) was not provided.

Station ID **M4-548-FIF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID **M4-556-FIF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	X	X	X
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	5-8-99/No Time	5-8-99/No Time	5-8-99/No Time
Digestion Date	06/04/99	06/04/99	06/04/99
Analysis Date	07/07/99	06/04/99	06/04/99
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume	3 Fish	NA	NA
Total Volume	3 Fish	3 Fish	3 Fish
Sample Volume Analyzed	Aliquot	3 Fish	3 Fish
Dilution	1000	NA	NA
Results	43.78 (Average of 3)	15 - 25	0.0293 - 0.1084
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	FDL/JL	MB	MB
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst	No	No	No
All Calculation Checked	X	X	X
Holding Time Met	Yes (28 day Holding Time)	No (Goal Only)	No (Goal Only)
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID	HG07GF1	By: Date/Time	By: Date/Time
Method Blanks	0.244	NA	NA
Instrument Blanks	0.003	NA	NA
Duplicates (RPD)	<20 RSD	NA	NA
Matrix Spike Recoveries (75-125)	92 - 98%R	NA	NA
Blank Spike/CCV Recoveries	88 - 100%R	NA	NA
Detection Range	3.2 ppb and >	NA	NA
Correlation Coefficient (>0.995)	0.9999	NA	NA
QC Report (Verified)			
Lab Data Entry Checked by Analyst	No	No	No
All Calculation Checked	X	X	X
QC Limits Met			

Notes

No descriptive narratives were provided by FIU.

Holding time goals set for the length and weight measurements are guidelines only, not a hard deadline.

Measurements of fish length and weight were measured after holding time goals.

Documentation on how the fish samples were stored (Frozen, 4 C, preserved) was not provided.

Station ID **M4-556-FIF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID **M4-566-FIF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	X	X	X
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	5-7-99/No Time	5-7-99/No Time	5-7-99/No Time
Digestion Date	06/09/99	06/09/99	06/09/99
Analysis Date	07/07/99	06/09/99	06/09/99
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume	7 Fish	NA	NA
Total Volume	7 Fish	7 Fish	7 Fish
Sample Volume Analyzed	Aliquot	7 Fish	7 Fish
Dilution	200-1000	NA	NA
Results	101.1 (Average of 7)	22 - 35	0.0744 - 0.3513
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	FDL/JL	MB	MB
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst	No	No	No
All Calculation Checked	X	X	X
Holding Time Met	No (Goal Only)	No (Goal Only)	No (Goal Only)
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID	HG07GF1	By: Date/Time	By: Date/Time
Method Blanks	0.244	NA	NA
Instrument Blanks	0.003	NA	NA
Duplicates (RPD)	<20 RSD	NA	NA
Matrix Spike Recoveries (75-125)	92 - 98%R	NA	NA
Blank Spike/CCV Recoveries	88 - 100%R	NA	NA
Detection Range	3.2 ppb and >	NA	NA
Correlation Coefficient (>0.995)	0.9999	NA	NA
QC Report (Verified)			
Lab Data Entry Checked by Analyst	No	No	No
All Calculation Checked	X	X	X
QC Limits Met	"H"		

Notes

No descriptive narratives were provided by FIU.

Sample for total mercury was digested past holding time.

Holding time goals set for the length and weight measurements are guidelines only, not a hard deadline.

Measurements of fish length and weight were measured after holding time goals.

Documentation on how the fish samples were stored (Frozen, 4 C, preserved) was not provided.

Station ID **M4-566-FIF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID **M4-568-FIF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	X	X	X
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	5-7-99/No Time	5-7-99/No Time	5-7-99/No Time
Digestion Date	06/16/99	06/16/99	06/16/99
Analysis Date	07/08/99	06/16/99	06/16/99
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume	7 Fish	NA	NA
Total Volume	7 Fish	7 Fish	7 Fish
Sample Volume Analyzed	Aliquot	7 Fish	7 Fish
Dilution	1000	NA	NA
Results	122.2 (Average of 7)	20 - 25	0.0538 - 0.141
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	FDL/JL	MB	MB
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst	No	No	No
All Calculation Checked	X	X	X
Holding Time Met	No (Goal Only)	No (Goal Only)	No (Goal Only)
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID	HG08GF1	By: Date/Time	By: Date/Time
Method Blanks	0.997	NA	NA
Instrument Blanks	0.0027	NA	NA
Duplicates (RPD)	<20 RSD	NA	NA
Matrix Spike Recoveries (75-125)	102 - 111 %R	NA	NA
Blank Spike/CCV Recoveries	91 - 108 %R	NA	NA
Detection Range	3.2 ppb and >	NA	NA
Correlation Coefficient (>0.995)	0.999	NA	NA
QC Report (Verified)			
Lab Data Entry Checked by Analyst	No	No	No
All Calculation Checked	X	X	X
QC Limits Met	"H"		

Notes

No descriptive narratives were provided by FIU.

Sample for total mercury was digested past holding time.

Holding time goals set for the length and weight measurements are guidelines only, not a hard deadline.

Measurements of fish length and weight were measured after holding time goals.

Documentation on how the fish samples were stored (Frozen, 4 C, preserved) was not provided.

Station ID **M4-568-FIF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID **M4-576-FIF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	X	X	X
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	5-4-99/No Time	5-4-99/No Time	5-4-99/No Time
Digestion Date	06/18/99	06/18/99	06/18/99
Analysis Date	07/12/99	06/18/99	06/18/99
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume	7 Fish	NA	NA
Total Volume	7 Fish	7 Fish	7 Fish
Sample Volume Analyzed	Aliquot	7 Fish	7 Fish
Dilution	200-500	NA	NA
Results	245.3 (Average of 7)	24 - 31	0.1871 - 0.3355
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	FDL/JL	MB	MB
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (MB)	No	No
All Calculation Checked	X	X	X
Holding Time Met	No (Goal Only)	No (Goal Only)	No (Goal Only)
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID	HG12GF1	By: Date/Time	By: Date/Time
Method Blanks	0.071	NA	NA
Instrument Blanks	0.0034	NA	NA
Duplicates (RPD)	<20 RSD	NA	NA
Matrix Spike Recoveries (75-125)	99 - 100 %R	NA	NA
Blank Spike/CCV Recoveries	84 - 97%R	NA	NA
Detection Range	3.2 ppb and >	NA	NA
Correlation Coefficient (>0.995)	0.9998	NA	NA
QC Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (MB)	No	No
All Calculation Checked	X	X	X
QC Limits Met	"H"		

Notes

No descriptive narratives were provided by FIU.

Sample for total mercury was digested past holding time.

Holding time goals set for the length and weight measurements are guidelines only, not a hard deadline.

Measurements of fish length and weight were measured after holding time goals.

Documentation on how the fish samples were stored (Frozen, 4 C, preserved) was not provided.

Station ID **M4-576-FIF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID **M4-586-FIF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	No Samples were Collected	No Samples were Collected	No Samples were Collected
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	No Samples were Collected	No Samples were Collected	No Samples were Collected
Digestion Date			
Analysis Date			
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume			
Total Volume			
Sample Volume Analyzed			
Dilution			
Results			
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	No Sample	No Sample	No Sample
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst			
All Calculation Checked			
Holding Time Met			
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID		By: Date/Time	By: Date/Time
Blanks			
Duplicates (RPD)			
Matrix Spike Recoveries (75-125)			
Blank Spike/CCV Recoveries			
Detection Range			
Correlation Coefficient (>0.995)			
QC Report (Verified)			
Lab Data Entry Checked by Analyst			
All Calculation Checked			
QC Limits Met			

Notes	
	Samples were not collected and/or delivered to the SERC Laboratory.

Station ID **M4-586-FIF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID			
Matrix			
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets			
Sample Run Logs			
Instrument Raw Data			
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs			
Raw Data (Verified)			
Sample ID Transferred			
All Calculation Checked			
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID **M4-594-FIF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	X	X	X
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	5-5-99/No Time	5-5-99/No Time	5-5-99/No Time
Digestion Date	06/23/99	06/23/99	06/23/99
Analysis Date	07/12/99	06/23/99	06/23/99
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume	6 Fish	NA	NA
Total Volume	6 Fish	7 Fish	7 Fish
Sample Volume Analyzed	Aliquot	7 Fish	7 Fish
Dilution	200 - 1000	NA	NA
Results	180.4 (Average of 6)	22 - 30	0.0729 - 0.218
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	FDL/JL	MB	MB
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (MB)	No	No
All Calculation Checked	X	X	X
Holding Time Met	No (Goal Only)	No (Goal Only)	No (Goal Only)
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID	HG12GF1	By: Date/Time	By: Date/Time
Method Blanks	0.071	NA	NA
Instrument Blanks	0.0034	NA	NA
Duplicates (RPD)	<20 RSD	NA	NA
Matrix Spike Recoveries (75-125)	99 - 100 %R	NA	NA
Blank Spike/CCV Recoveries	84 - 97%R	NA	NA
Detection Range	3.2 ppb and >	NA	NA
Correlation Coefficient (>0.995)	0.9998	NA	NA
QC Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (MB)	No	No
All Calculation Checked	X	X	X
QC Limits Met	"H"		

Notes

No descriptive narratives were provided by FIU.

Sample for total mercury was digested past holding time.

Holding time goals set for the length and weight measurements are guidelines only, not a hard deadline.

Measurements of fish length and weight were measured after holding time goals.

Documentation on how the fish samples were stored (Frozen, 4 C, preserved) was not provided.

Station ID **M4-594-FIF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID **M4-599-FIF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	X	X	X
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	5-6-99/No Time	5-6-99/No Time	5-6-99/No Time
Digestion Date	06/23/99	06/23/99	06/23/99
Analysis Date	07/13/99	06/23/99	06/23/99
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume	7 Fish	NA	NA
Total Volume	7 Fish	7 Fish	7 Fish
Sample Volume Analyzed	Aliquot	7 Fish	7 Fish
Dilution	200 - 1000	NA	NA
Results	286.9 (Average of 7)	23 - 35	0.116 - 0.3574
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	FDL/JL	MB	MB
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (MB)	No	No
All Calculation Checked	X	X	X
Holding Time Met	No (Goal Only)	No (Goal Only)	No (Goal Only)
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID	HG13GF1	By: Date/Time	By: Date/Time
Method Blanks	1.858	NA	NA
Instrument Blanks	0.0024	NA	NA
Duplicates (RPD)	<20 RSD	NA	NA
Matrix Spike Recoveries (75-125)	90 - 97	NA	NA
Blank Spike/CCV Recoveries	93 - 102	NA	NA
Detection Range	3.2 ppb and >	NA	NA
Correlation Coefficient (>0.995)	0.9988	NA	NA
QC Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (MB)	No	No
All Calculation Checked	X	X	X
QC Limits Met	"H"		

Notes

No descriptive narratives were provided by FIU.

Sample for total mercury was digested past holding time.

Holding time goals set for the length and weight measurements are guidelines only, not a hard deadline.

Measurements of fish length and weight were measured after holding time goals.

Documentation on how the fish samples were stored (Frozen, 4 C, preserved) was not provided.

Station ID **M4-599-FIF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID **M4-809-FIF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	X	X	X
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	5-11-99/No Time	5-11-99/No Time	5-11-99/No Time
Digestion Date	07/06/99	07/06/99	07/06/99
Analysis Date	07/13/99	07/06/99	07/06/99
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume	7 Fish	NA	NA
Total Volume	7 Fish	7 Fish	7 Fish
Sample Volume Analyzed	Aliquot	7 Fish	7 Fish
Dilution	200 - 500	NA	NA
Results	107.9 (Average of 7)	Unable to read raw data	0.1553 - 0.321
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	FDL/JL	MB	MB
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (MB)	No	No
All Calculation Checked	X	X	X
Holding Time Met	No (Goal Only)	No (Goal Only)	No (Goal Only)
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID	HG13GF1	By: Date/Time	By: Date/Time
Method Blanks	1.858	NA	NA
Instrument Blanks	0.0024	NA	NA
Duplicates (RPD)	<20 RSD	NA	NA
Matrix Spike Recoveries (75-125)	90 - 97	NA	NA
Blank Spike/CCV Recoveries	93 - 102	NA	NA
Detection Range	3.2 ppb and >	NA	NA
Correlation Coefficient (>0.995)	0.9988	NA	NA
QC Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (MB)	No	No
All Calculation Checked	X	X	X
QC Limits Met	"H"		

Notes

No descriptive narratives were provided by FIU.

Sample for total mercury was digested past holding time.

Holding time goals set for the length and weight measurements are guidelines only, not a hard deadline.

Measurements of fish length and weight were measured after holding time goals.

Documentation on how the fish samples were stored (Frozen, 4 C, preserved) was not provided.

Station ID **M4-809-FIF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID **M4-872-FIF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	X	X	X
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	5-8-99/No Time	5-8-99/No Time	5-8-99/No Time
Digestion Date	07/06/99	07/06/99	07/06/99
Analysis Date	07/22/99	07/06/99	07/06/99
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume	7 Fish	NA	NA
Total Volume	7 Fish	7 Fish	7 Fish
Sample Volume Analyzed	Aliquot	7 Fish	7 Fish
Dilution	500-1000	NA	NA
Results	164.5 (Average of 7)	Unable to read raw data	0.0517 - 0.2128
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	FDL/JL	MB	MB
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst	No	No	No
All Calculation Checked	X	X	X
Holding Time Met	No (Goal Only)	No (Goal Only)	No (Goal Only)
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID	HG22GF1	By: Date/Time	By: Date/Time
Method Blanks	0.658	NA	NA
Instrument Blanks	0.0028	NA	NA
Duplicates (RPD)	<20 RSD	NA	NA
Matrix Spike Recoveries (75-125)	77 - 92 %R	NA	NA
Blank Spike/CCV Recoveries	96 - 100 %R	NA	NA
Detection Range	3.2 ppb and >	NA	NA
Correlation Coefficient (>0.995)	0.9998	NA	NA
QC Report (Verified)			
Lab Data Entry Checked by Analyst	No	No	No
All Calculation Checked	X	X	X
QC Limits Met	"H"		

Notes

No descriptive narratives were provided by FIU.

Sample for total mercury was digested past holding time.

Holding time goals set for the length and weight measurements are guidelines only, not a hard deadline.

Measurements of fish length and weight were measured after holding time goals.

Documentation on how the fish samples were stored (Frozen, 4 C, preserved) was not provided.

Station ID **M4-872-FIF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	Date/No Time	Date/No Time	Date/No Time
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

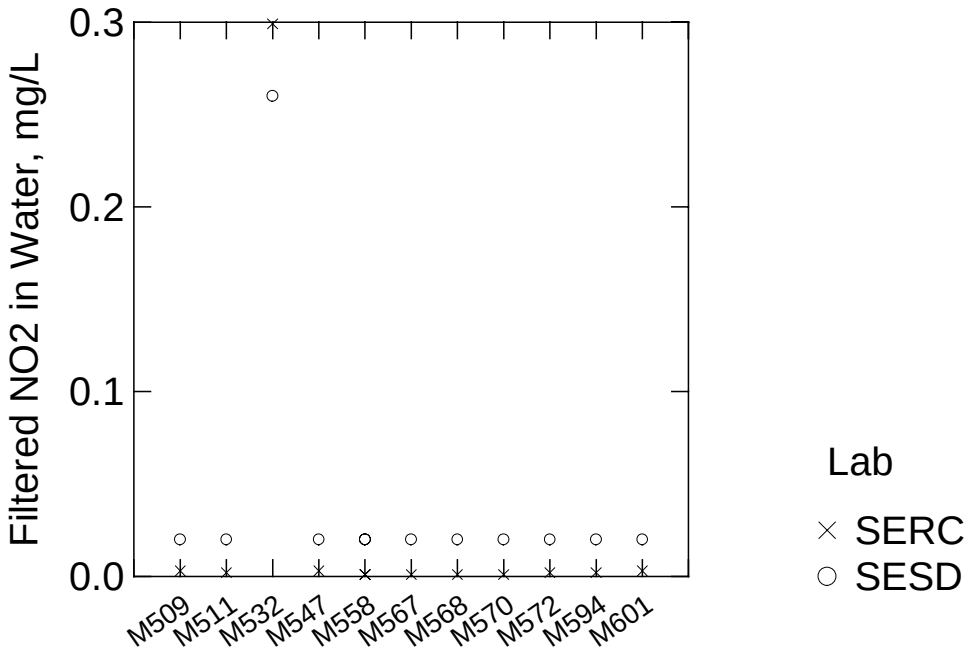
"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

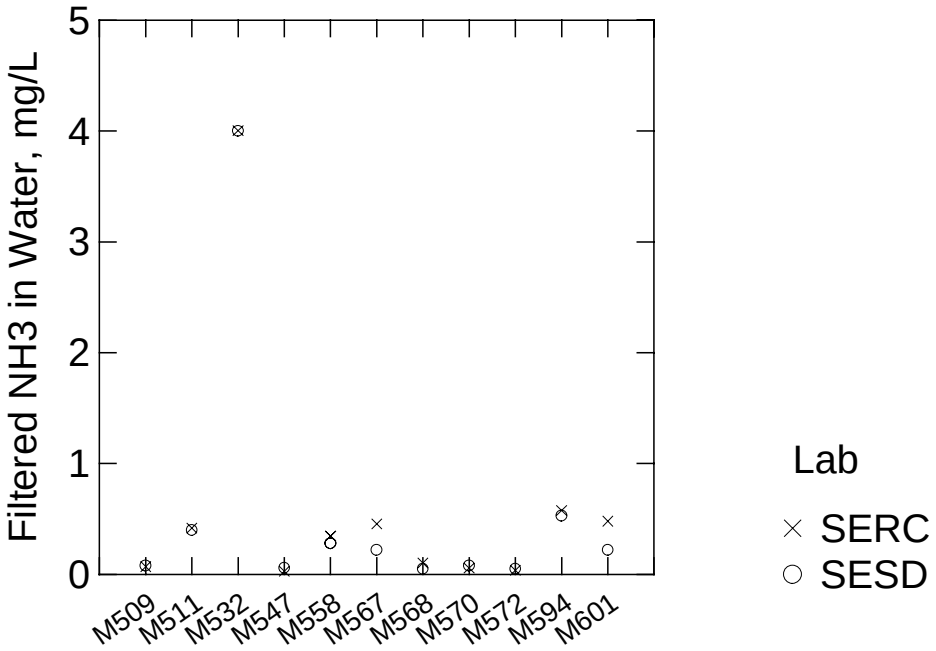
"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

INTERLABORATORY COMPARISONS

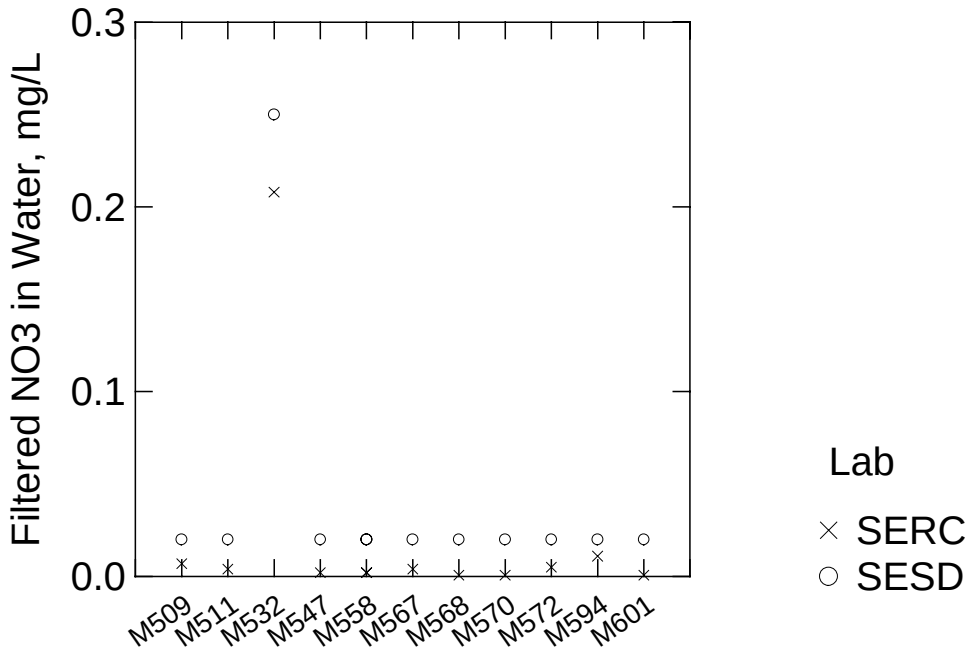
May 1999 Samples



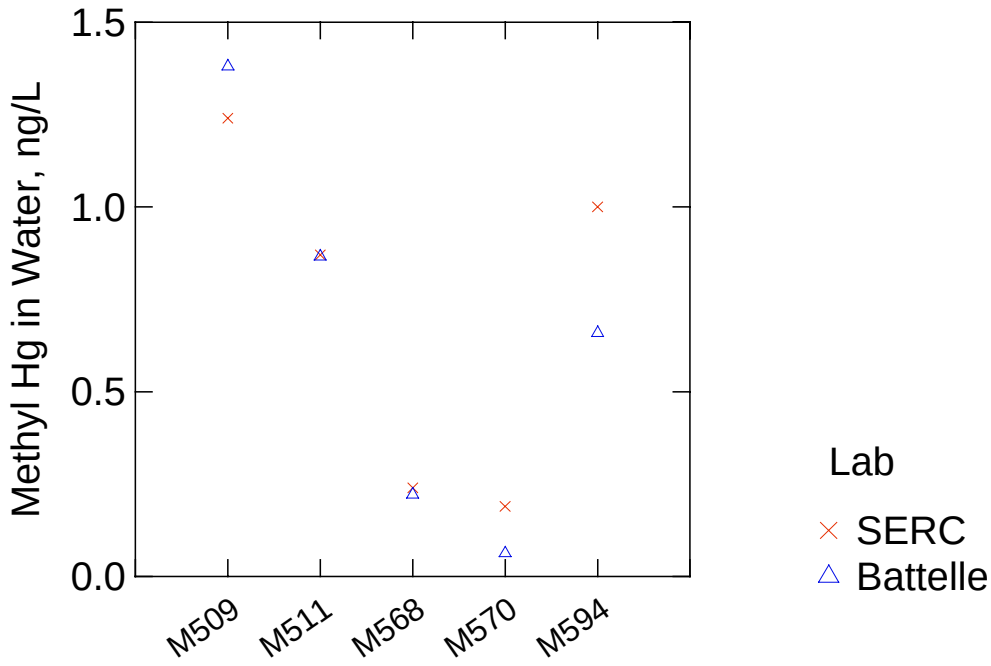
May 1999 Samples



May 1999 Samples



May 1999 Samples



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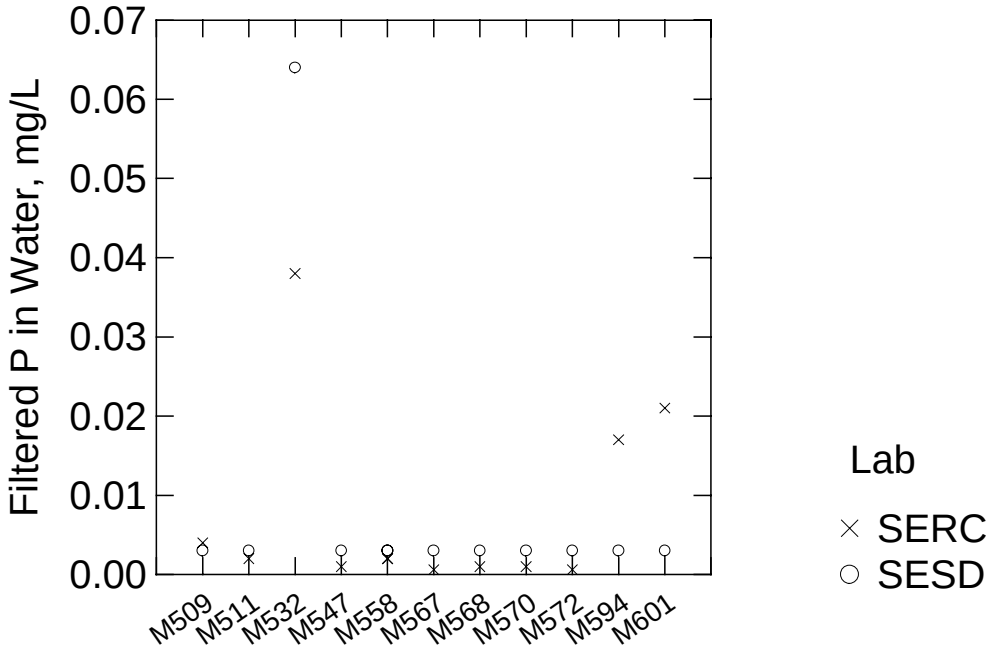

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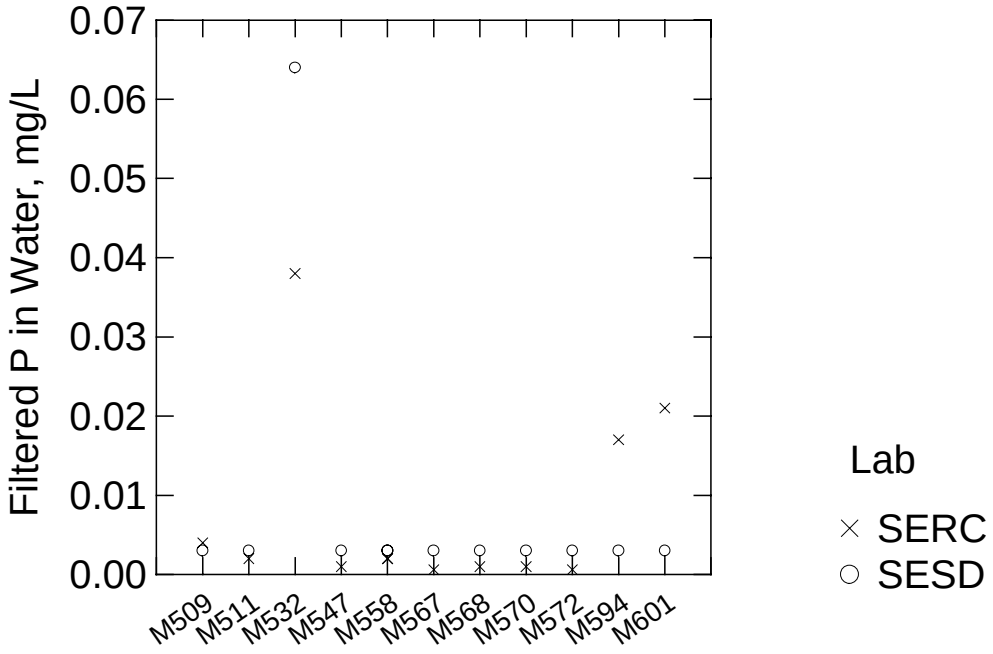
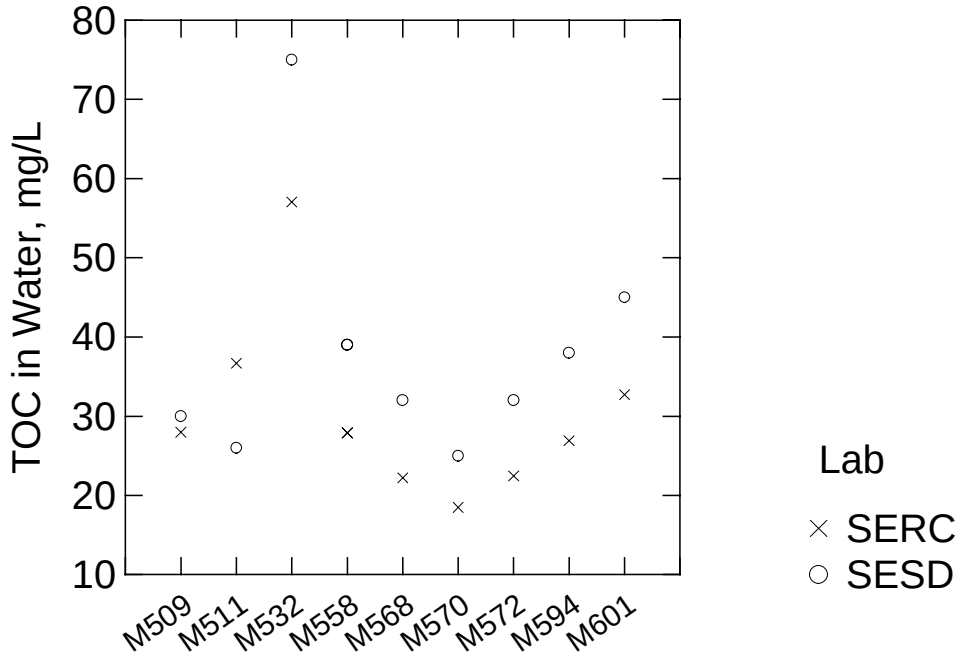
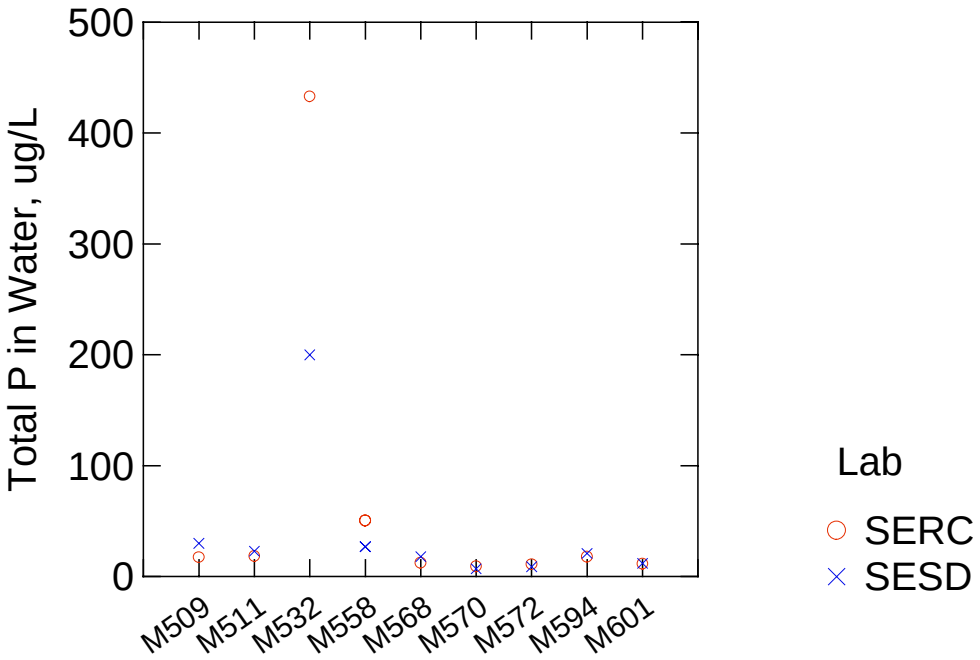


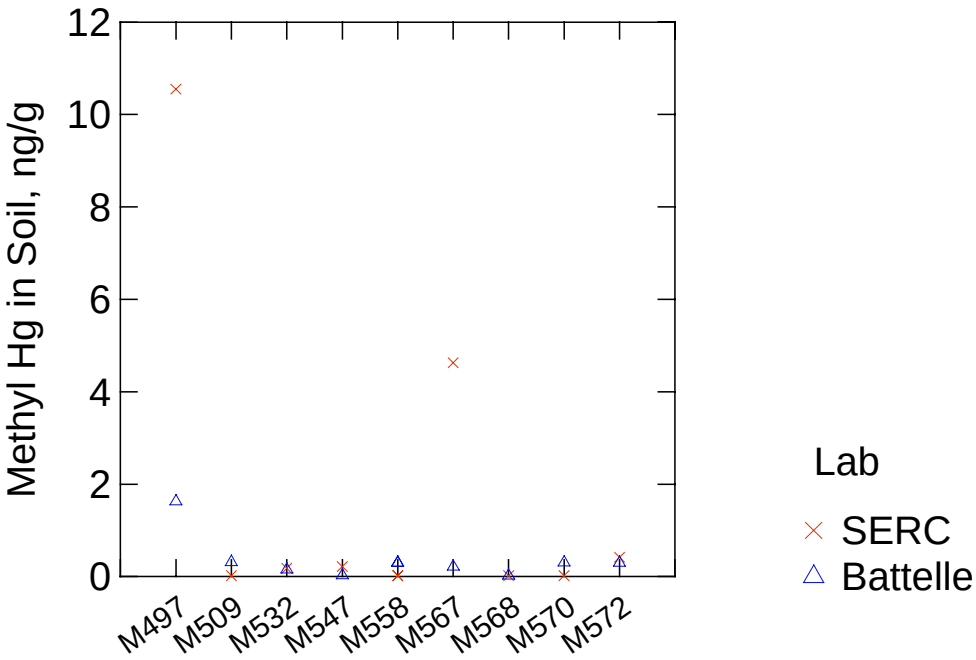
May 1999 Samples



May 1999 Samples



May 1999 Samples



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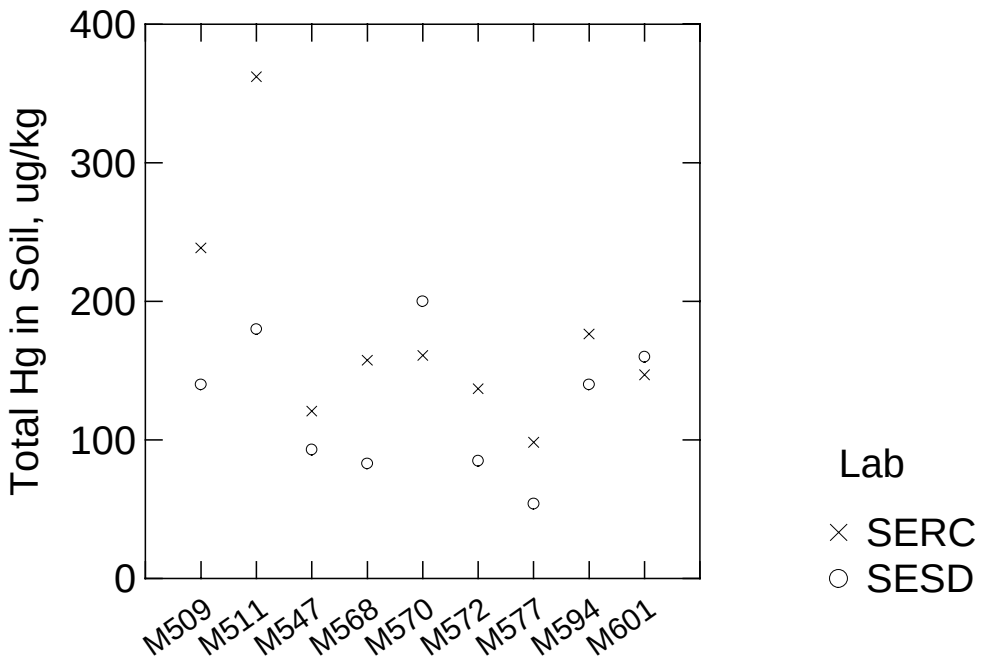
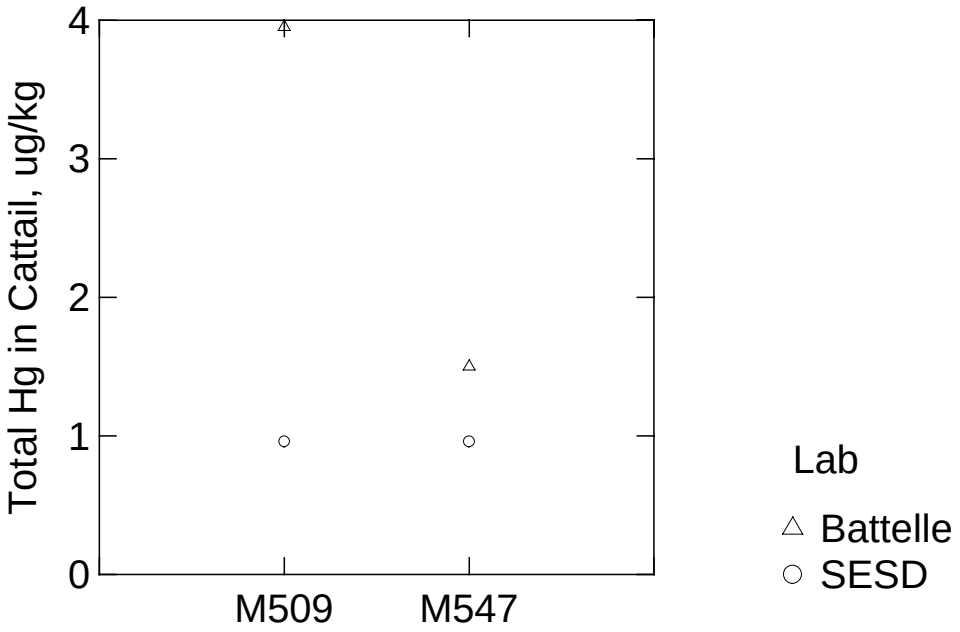
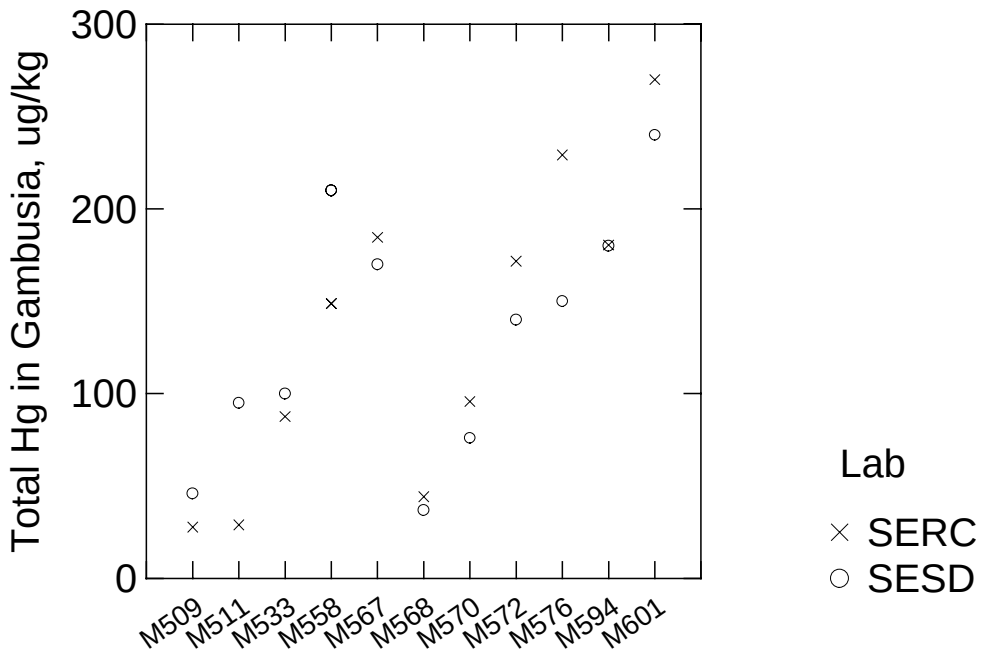


May 1999 Samples



May 1999 Samples



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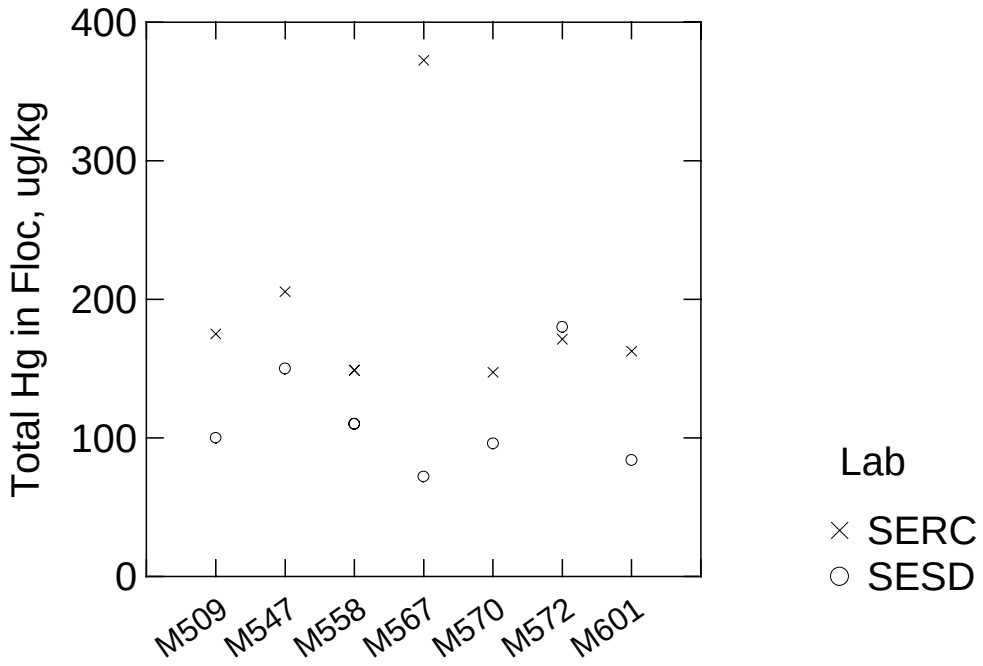
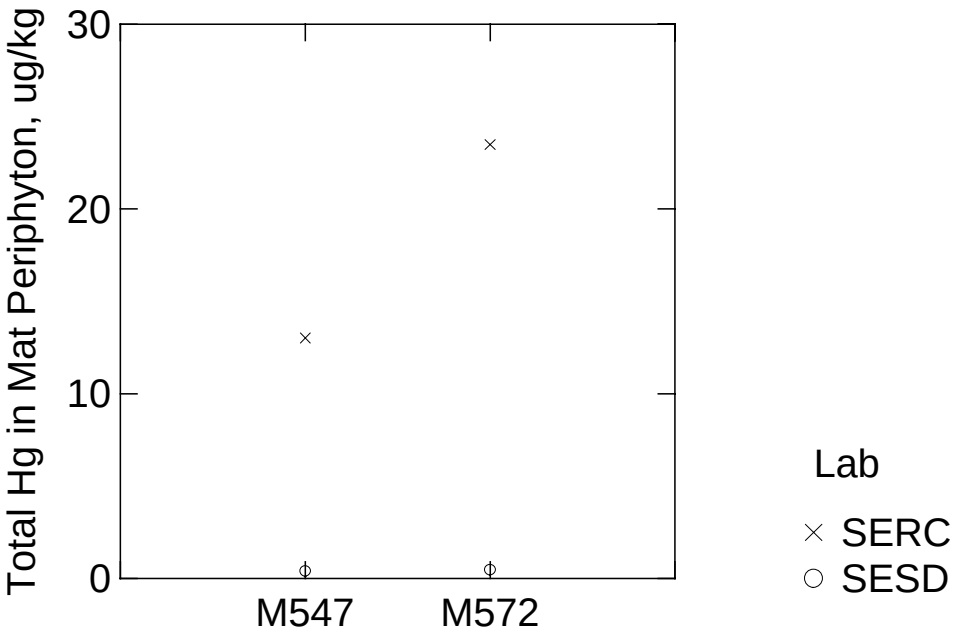
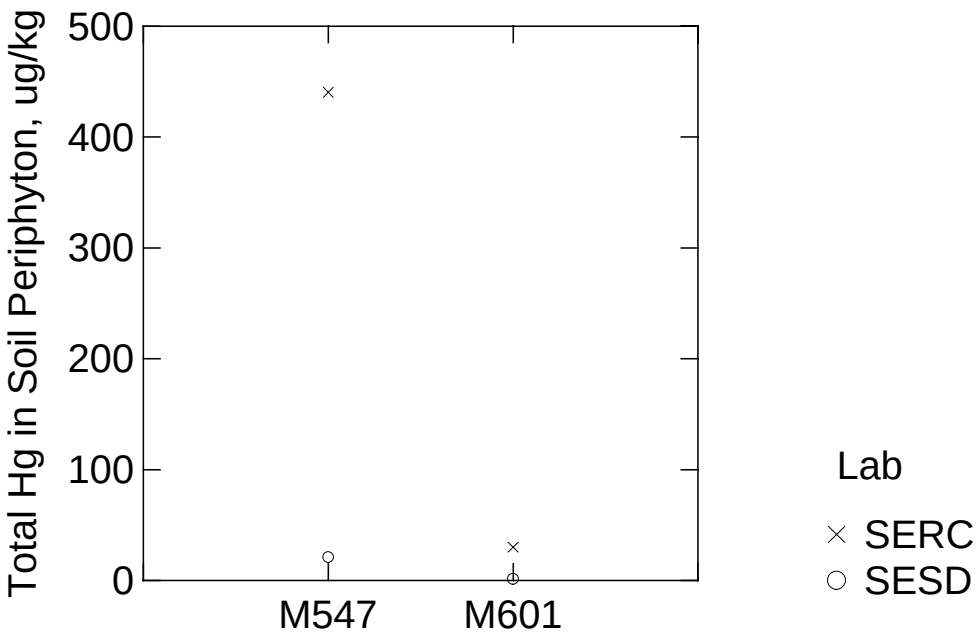
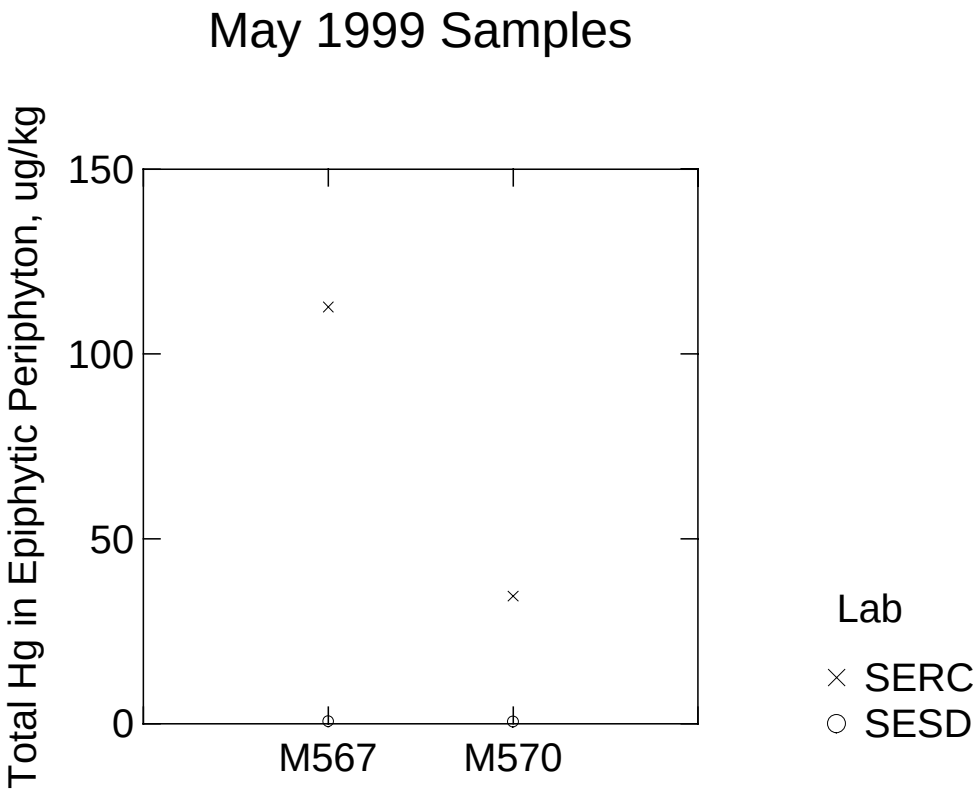


May 1999 Samples

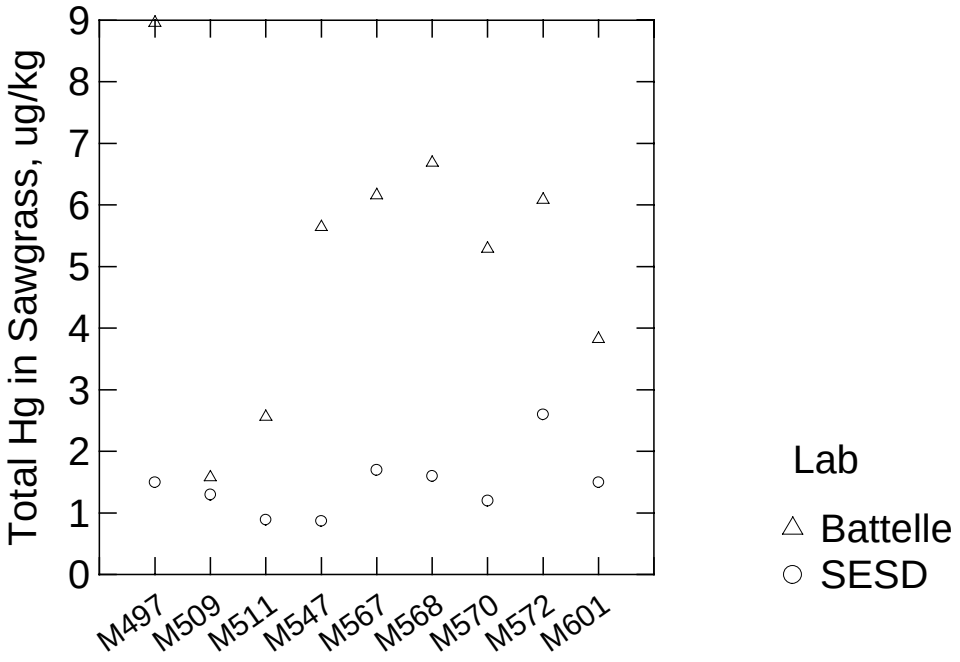


May 1999 Samples




May 1999 Samples
Total Hg in Epiphytic Periphyton, ug/kg
150
100
50
0
M567
M570
Lab
SERC
SESD

May 1999 Samples



EVERGLADES ECOSYSTEM ASSESSMENT
(PHASE II REMAP)

Data Review
September 1999 Sampling

Data Review, September 1999 Sampling

Foreward

The data review documents developed by the US Environmental Protection Agency (EPA) as part of the Investigation of Mercury Contamination in the Florida Everglades Ecosystem and Everglades Ecosystem Assessment (Phase II REMAP) Project are presented in the Data Review May 1999 (M4) and September 1999 Sampling (M5) documents.

The Phase II data review determines whether the Data Quality Objectives (DQO) have been satisfied as outlined in the Quality Assurance Project Plan (QAPP). The M4 and M5 Sampling results were analyzed to determine whether they met the criteria developed during the planning phase and whether the total error within the tolerable decision error ranges as specified in the QAPP to support decisions.

The Data Review, September 1999 Sampling document summarizes the assessments of the critical and non-critical parameters. Ten percent of the samples were randomly selected during the validation process to characterize the quality of the data set. Three of the eleven critical parameters are qualified with a “J”. Parameters associated with this qualifier should be considered an estimate for a number of quality control variances. The results for methylmercury in surface water, methylmercury and in soil and bulk density in soil should be considered an estimate based on findings. A table summarizing the critical and non-critical parameters is enclosed in the Data Review document along with the detailed calculations and criteria for each selected sample and parameter.

TABLE OF CONTENTS

CRITICAL QA/QC SUMMARIES

EPA SESD South Florida Phase II Wet Season Sampling: September 1999
Summarized Qualifiers for 10% of the Critical Parameters Reviewed

	Surface Water					Soil/ Sediment				Fish	
	SERC Laboratory				Battelle Lab	SERC Laboratory				SERC Laboratory	
Station ID	Total Phosphorus	Total Nitrogen	TOC	Total Mercury	Methyl Mercury	Methyl Mercury	Total Phosphorus	AFDW	Bulk Density	Total Mercury	Length/Weight
M5-622	"**"	"**"					"B (NR)"	"DQO (NR)"	"DQO (NR)"		
M5-633	"**"	"**"			"M"	"M"	"B (NR)"	"DQO (NR)"	"DQO (NR)"	"M"	
M5-643	"**"	"**"			"M"	"M"	"B (NR)"	"DQO (NR)"	"DQO (NR)"		
M5-653	"**"	"**"		"M"		"DQO (NR)"	"B (NR)"	"DQO (NR)"	"DQO (NR)"		
M5-663	"**"	"**"				"M"	"B (NR)"	"DQO (NR)"	"DQO (NR)"		
M5-673	"B(NR)"	"**"					"B (NR)"	"DQO (NR)"	"DQO (NR)"		
M5-683	"B(NR)"	"**"				"M", "DQO"	"B (NR)"	"DQO (NR)"	"DQO (NR)"		
M5-693	"B(NR)"	"**"				"M", "DQO"	"B (NR)"	"DQO (NR)"	"DQO (NR)"		
M5-703	"B(NR)"	"**"				"M", "DQO"	"B (NR)"	"DQO (NR)"	"DQO (NR)"		
M5-714	"B(NR)"	"**"	"H"			"M", "DQO"	"B (NR)"	"DQO (NR)"	"DQO (NR)"		
M5-726	"**"	"**"	"H"		"M", "DQO"	"M"	"B (NR)"	"DQO (NR)"	"DQO (NR)"		
M5-738	"**"	"**"	"H"		"M", "DQO"	"DQO"	"B (NR)"	"DQO (NR)"	"DQO (NR)"		
M5-828	"**"	"**"				"DQO (NR)", "M"	"B (NR)"	"DQO (NR)"	"DQO (NR)"		
M5-944	"**"	"**","M"	"H"			"M", "DQO"	"B (NR)"	"DQO (NR)"	"DQO (NR)"		
M5-656					"M"						
M5-661					"M"						
M5-672					"M"						
M5-684					"M"						
M5-712											
M5-823											
M5-920					"M", "DQO"						
Qualifier Notation					**"J"**	**"J"**			**"J", "***"**		

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.
"B" Analyte concentration in the associated blank was >3 times the MDL.
"H" Analysis digestion performed after holding times have expired.
"NR" Data was unavailable for review.
"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

"*" The matrix recoveries were below QAPP QA limits. MeHg matrix spikes are run with every sample and the sample is adjusted based on matrix affects.
"**" Blanks were reported above the MDL. Lab water was used as the blank water but not in the sample digestion process.
"***" Phase II soil/sediment samples were prepped with a high speed blender which may alter comparability with the phase I bulk density values.

Comments:
The coefficient of variance (R2) values for methyl mercury were not reviewed.

EPA SESD South Florida Phase II Wet Season Sampling: September 1999
Summarized Findings of the Full QA/QC Review
Total Mercury in Surface Water Analyzed By SERC

Sample ID by QC Batch	Matrix Spike Not Reviewed for Batch	Data Entry Check Not Noted	Holding Time Exceeded	CCV Not Reviewed	Blank Result >3 MDL	Corr. Coef. Not Reviewed	Cannot Reproduce Results Calcs.	Precision Criteria Not Met	No Dups/Reps Reviewed	No Blanks Reviewed in Batch	Potential Matrix Effect (Data User Note)
M5-622-SWF											
M5-633-SWF											
M5-643-SWF											
M5-653-SWF											X
M5-663-SWF											
M5-673-SWF M5-683-SWF											
M5-693-SWF M5-703-SWF											
M5-714-SWF											
M5-726-SWF											
M5-738-SWF M5-828-SWF											
M5-944-SWF											

Footnotes:

" X " Indicates this situation did occur.

EPA SESD South Florida Phase II Wet Season Sampling: September 1999
Summarized Findings of the Full QA/QC Review
Total Mercury in Fish Analyzed By SERC

Sample ID by QC Batch	Matrix Spike Not Reviewed for Batch	Data Entry Check Not Noted	Holding Time Exceeded	CCV Not Reviewed	Blank Result >3 MDL	Corr. Coef. Not Reviewed	Cannot Reproduce Results Calcs.	Precision Criteria Not Met	No Dups/Reps Reviewed	No Blanks Reviewed in Batch	Potential Matrix Effect (Data User Note)
M5-622-FIF											
M5-633-FIF											X
M5-643-FIF											
M5-653-FIF											
M5-663-FIF			***								
M5-673-FIF			***								
M5-683-FIF			***								
M5-693-FIF			***								
M5-703-FIF			***								
M5-714-FIF			***								
M5-726-FIF			***								
M5-738-FIF			***								
M5-828-FIF			***								
M5-944-FIF			***								

Footnotes:
" *** " Holding time goal only.
" X " Indicates this situation did occur.

EPA SESD South Florida Phase II Wet Season Sampling: September 1999
Summarized Findings of the Full QA/QC Review
Total Phosphorus in Surface Water Analyzed By SERC

Sample ID by QC Batch	Matrix Spike Not Reviewed for Batch	Data Entry Check Not Noted	Holding Time Exceeded	CCV Not Reviewed	Blank Result >3 MDL	Corr. Coef. Not Reviewed	Cannot Reproduce Results Calcs.	Precision Criteria Not Met	No Dups/Reps Reviewed	No Blanks Reviewed in Batch	Potential Matrix Effect (Data User Note)
M5-622-SWF					***						
M5-633-SWF					***						
M5-643-SWF					***						
M5-653-SWF					***						
M5-663-SWF					***						
M5-673-SWF					NR						
M5-683-SWF					NR						
M5-693-SWF					NR						
M5-703-SWF					NR						
M5-714-SWF					NR						
M5-726-SWF					***						
M5-738-SWF					***						
M5-828-SWF					***						
M5-944-SWF					***						

Footnotes:

" *** " Blanks were reported above the MDL. Lab water was used as the blank water but not in the sample digestion process. Procedures have been modified to correct this.

"NR" Data was not available for review.

" X " Indicates this situation did occur.

EPA SESD South Florida Phase II Wet Season Sampling: September 1999
Summarized Findings of the Full QA/QC Review
Total Nitrogen in Surface Water Analyzed By SERC

Sample ID by QC Batch	Matrix Spike Not Reviewed for Batch	Data Entry Check Not Noted	Holding Time Exceeded	CCV Not Reviewed	Blank Result >3 MDL	Corr. Coef. Not Reviewed	Cannot Reproduce Results Calcs.	Precision Criteria Not Met	No Dups/Reps Reviewed	No Blanks Reviewed in Batch	Potential Matrix Effect (Data User Note)
M5-622-SWF					***						
M5-633-SWF					***						
M5-643-SWF					***						
M5-653-SWF					***						
M5-663-SWF					***						
M5-673-SWF					***						
M5-683-SWF					***						
M5-693-SWF					***						
M5-703-SWF					***						
M5-714-SWF					***						
M5-726-SWF					***						
M5-738-SWF					***						
M5-828-SWF					***						
M5-944-SWF			X		***						X

Comments:

1. Blanks were not reviewed in this data set.

Footnotes:

" *** " Blanks were reported above the MDL. Lab water was used as the blank water but not in the sample digestion process Procedures have been modified to correct this.

" X " Indicates this situation did occur.

EPA SESD South Florida Phase II Wet Season Sampling: September 1999
Summarized Findings of the Full QA/QC Review
Total Organic Carbon in Surface Water Analyzed By SERC

Sample ID by QC Batch	Matrix Spike Not Reviewed for Batch	Data Entry Check Not Noted	Holding Time Exceeded	CCV Not Reviewed	Blank Result >3 MDL	Corr. Coef. Not Reviewed	Cannot Reproduce Results Calcs.	Precision Criteria Not Met	No Dups/Reps Reviewed	No Blanks Reviewed in Batch	Potential Matrix Effect (Data User Note)
M5-622-SWF											
M5-633-SWF											
M5-643-SWF											
M5-653-SWF											
M5-663-SWF											
M5-673-SWF											
M5-683-SWF											
M5-693-SWF											
M5-703-SWF											
M5-714-SWF			X								
M5-726-SWF			X								
M5-738-SWF			X								
M5-828-SWF											
M5-944-SWF			X								

Comments:
1. The laboratory comparisons that were performed did show a bias between the two laboratories. The two laboratories are using two separate but approved EPA methods and instruments for the TOC analysis. This comparative difference may be cause by the methodology difference.

Footnotes:
" X " Indicates this situation did occur.

EPA SESD South Florida Phase II Wet Season Sampling: September 1999
Summarized Findings of the Full QA/QC Review
Total Phosphorus in Soil Analyzed By SERC

Sample ID by QC Batch	Preservation Not Documented	Matrix Spike Not Reviewed for Batch	Data Entry Check Not Noted	Holding Time Exceeded	CCV Not Reviewed	Blank Result >3 MDL	Corr. Coef. Not Reviewed	Cannot Reproduce Results Calcs.	Precision Criteria Not Met	No Dups/Reps Reviewed	No Blanks Reviewed in Batch	Potential Matrix Effect (Data User Note)
M5-622-SDF				*							X	
M5-643-SDF				*							X	
M5-663-SDF				*							X	
M5-633-SDF				*							X	
M5-653-SDF				*							X	
M5-738-SDF				*							X	
M5-673-SDF				*							X	
M5-683-SDF				*							X	
M5-693-SDF				*							X	
M5-703-SDF				*							X	
M5-714-SDF				*							X	
M5-726-SDF				*							X	
M5-828-SDF				*							X	
M5-944-SDF				*							X	

Comments:
1. No blank results were reviewed.

Footnotes:
" * " Holding time goal only.
" X " Indicates this situation did occur.

EPA SESD South Florida Phase II Wet Season Sampling: September 1999
Summarized Findings of the Full QA/QC Review
Soil Ash-Free Dry Weight Analyzed By SERC

Sample ID by QC Batch	Preservation Not Documented	Matrix Spike Not Reviewed for Batch	Data Entry Check Not Noted	Holding Time Exceeded	CCV Not Reviewed	Blank Result >3 MDL	Corr. Coef. Not Reviewed	Cannot Reproduce Results Calcs.	Precision Criteria Not Met	No Dups/Reps Reviewed	No Blanks Reviewed in Batch	Potential Matrix Effect (Data User Note)
M5-622-SDF										X		
M5-633-SDF										X		
M5-643-SDF										X		
M5-653-SDF										X		
M5-663-SDF										X		
M5-673-SDF										X		
M5-683-SDF										X		
M5-693-SDF										X		
M5-703-SDF										X		
M5-714-SDF										X		
M5-726-SDF										X		
M5-738-SDF										X		
M5-828-SDF										X		
M5-944-SDF										X		

Footnotes:
" X " Indicates this situation did occur.

EPA SESD South Florida Phase II Wet Season Sampling: September 1999
Summarized Findings of the Full QA/QC Review
Soil Bulk Density Analyzed By SERC

Sample ID by QC Batch	Preservation Not Documented	Matrix Spike Not Reviewed for Batch	Data Entry Check Not Noted	Holding Time Exceeded	CCV Not Reviewed	Blank Result >3 MDL	Corr. Coef. Not Reviewed	Cannot Reproduce Results Calcs.	Precision Criteria Not Met	No Dups/Reps Reviewed	No Blanks Reviewed in Batch	Potential Matrix Effect (Data User Note)
M5-622-SDF										NR		
M5-633-SDF										NR		
M5-643-SDF										NR		
M5-653-SDF										NR		
M5-663-SDF										NR		
M5-673-SDF										NR		
M5-683-SDF										NR		
M5-693-SDF										NR		
M5-703-SDF										NR		
M5-714-SDF										NR		
M5-726-SDF										NR		
M5-738-SDF										NR		
M5-828-SDF										NR		
M5-944-SDF												

Footnotes:
"NR" Not Reviewed
" X " Indicates this situation did occur.

EPA SESD South Florida Phase II Wet Season Sampling: September 1999
Summarized Findings of the Full QA/QC Review
Methylmercury in Surface Water Analyzed By Battelle Laboratory

Sample ID by QC Batch	Matrix Spike Not Reviewed for Batch	Data Entry Check Not Noted	Holding Time Exceeded	CCV Not Reviewed	Blank Result >3 MDL	Corr. Coef. Not Reviewed	Cannot Reproduce Results Calcs.	Precision Criteria Not Met	No Dups/Reps Reviewed	No Blanks Reviewed in Batch	Potential Matrix Effect (Data User Note)
M5-622-SWB M5-633-SWB											
M5-643-SWB M5-656-SWB											X X
M5-661-SWB											X
M5-672-SWB											X
M5-684-SWB											X
M5-693-SWB M5-703-SWB											
M5-712-SWB											
M5-726-SWB M5-738-SWB								X X			X X
M5-823-SWB											X
M5-920-SWB								X			X

Footnotes:
" X " Indicates this situation did occur.

NON-CRITICAL QA/QC SUMMARIES

Non-Critical Parameters Analyzed by SERC
Review of the September, 1999 (M5) Data Set

	Supporting Documentation												
			Laboratory Records										
Analysis	Approved Project QA Plan	Comprehensive QA Plan (Lab	Calibration Verification	Analysis/Digestion Date/Time	Parameters Analyzed	Spikes Utilized	Duplicates Utilized	Instrument Maintenance Logs	Digestion Logs	Bench Sheets	Run Logs	Supplies/Consumables	Performance Evaluations
NH4	X	X	X	X	X	X	X	X	X	X	X	X	X
NO2	X	X	X	X	X	X	X	X	X	X	X	X	X
NO3	X	X	X	X	X	X	X	X	X	X	X	X	X
PO4	X	X	X	X	X	X	X	X	X	X	X	X	NR
CH4	X	X	X	X	X	NR	X	X	X	X	X	NR	NR
CO2	X	X	X	X	X	NR	X	X	X	X	X	NR	NR
APA	X	X	NR	X	X	NR	X	X	X	X	X	NR	NR
Mineral Content	X	X	NA	X	X	NA	X	X	X	X	X	X	NR
Diatoms	X	X	X	X	X	NR	NR	NR	NR	NR	NR	NR	NR
Pigments	**	**	**	**	**	**	**	**	**	**	**	**	**
Chlorophyll a	**	**	**	**	**	**	**	**	**	**	**	**	**
Ethyl Mercury	**	**	**	**	**	**	**	**	**	**	**	**	**

Footnotes:

" * " Analyses are in the process of being analyzed.

" ** " No analyses required.

" NR " Not Reviewed

" NA " Not Applicable

" X " Indicates this situation did occur.

Non-Critical Parameters Analyzed by Battelle Laboratories
Review of the September, 1999 (M5) Data Set

	Supporting Documentation												
			Laboratory Records										
Analysis	Approved Project QA Plan	Comprehensive QA Plan (Lab	Calibration Verification	Analysis/Digestion Date/Time	Parameters Analyzed	Spikes Utilized	Duplicates Utilized	Instrument Maintenance Logs	Digestion Logs	Bench Sheets	Run Logs	Supplies/Consumables	Performance Evaluations
Total Mercury (water)	X	X	X	X	X	X	X	X	X	X	X	NR	NR
Methylmercury (soil)	X	X	X	X	X	X	X	X	X	X	X	NR	NR
Methylmercury (floc)	X	X	X	X	X	X	X	X	X	X	X	NR	NR
Methylmercury (periphyton)	X	X	X	X	X	X	X	X	X	X	X	NR	NR

Footnotes:
"NR" Not Reviewed
" X " Indicates this situation did occur.

CRITICAL QA/QC REVIEW

Water - SERC

10 % Recalculated Results for Total Phosphorus in Surface Water

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-4-00 Checked by njs 4-13-00

Sample	QC Batch	Qualifier Note	Dilution	Rep um	Base Line Factor um	Peak - Base Line um	MW g	Corrected Peak * MW	Sample Results ppm	Detection Limit ppm
M5-622-SWF	EPA1027A		1	0.220441	-0.07	0.290441	31	9.003671	0.0090	0.0006
M5-633-SWF	EPA1027A		1	0.922642	-0.07	0.992642	31	30.771902	0.031	0.0006
M5-643-SWF	EPA1027A		1	1.001121	-0.07	1.071121	31	33.204751	0.033	0.0006
M5-653-SWF	EPA1027A		1	2.309137	-0.07	2.379137	31	73.753247	0.074	0.0006
M5-663-SWF	EPA1027A		1	0.182714	-0.07	0.252714	31	7.834134	0.0078	0.0006

Sample	QC Batch	Qualifier Note	Dilution	Rep um	Base Line Factor um	Peak - Base Line um	MW g	Corrected Peak * MW	Sample Results ppm	Detection Limit*3 ppm	SPK CONC (ppm)	R%	RPD
MS	EPA1027A		1	1.732597	-0.07	1.803	31	55.88	0.05588	0.0018	0.04092	90.7	
MS	EPA1027A		1	1.78027	-0.07	1.850	31	57.36	0.05736	0.0018	0.04092	93.2	-2.610
UMS	EPA1027A		1	0.535236	-0.07	0.605	31	18.76	0.01876	0.0018			
UMS	EPA1027A		1	0.550322	-0.07	0.620	31	19.23	0.01923	0.0018			-2.462
CCV	EPA1027A		1	1.444091	-0.07	1.552	31	48.13	0.04813	0.0018	0.05	96.25	
CCV	EPA1027A		1	1.482448	-0.07	1.572	31	48.75	0.04875	0.0018	0.05	97.49	
CCV	EPA1027A		1	1.502425	-0.07	1.571	31	48.69	0.04869	0.0018	0.05	97.38	
CCV	EPA1027A		1	1.500608	-0.07	1.599	31	49.56	0.04956	0.0018	0.05	99.11	
CCV	EPA1027A		1	1.528572	-0.07	1.599	31	49.56	0.04956	0.0018	0.05	99.12	
CCV	EPA1027A		1	1.52873	-0.07	1.539	31	47.72	0.04772	0.0018	0.05	95.44	
CCV	EPA1027A		1	1.469296	-0.07	1.074	31	33.30	0.03330	0.0018	0.05	66.60	
M632	EPA1027A		1	1.004262	-0.07	1.086	31	33.68	0.03368	0.0018			
M632D	EPA1027A		1	1.016345	-0.07	1.086	31	33.68	0.03368	0.0018			0.000
M642	EPA1027A		1	3.067382	-0.07	3.137	31	97.26	0.09726	0.0018			
M642D	EPA1027A		1	2.946699	-0.07	3.017	31	93.52	0.09352	0.0018			3.922
M652	EPA1027A		1	0.16731	-0.07	0.237	31	7.36	0.00736	0.0018			
M652D	EPA1027A		1	0.191554	-0.07	0.262	31	8.11	0.00811	0.0018			-9.720
M662	EPA1027A		1	0.193647	-0.07	0.264	31	8.17	0.00817	0.0018			
M662D	EPA1027A		1	0.178326	-0.07	0.248	31	7.70	0.00770	0.0018			5.985
Digestion Blk	EPA1027A	"B"	1	0.08028	-0.07	0.150	31	4.66	0.00466	0.0018			
Digestion Blk	EPA1027A	"B"	1	0.073422	-0.07	0.143	31	4.45	0.00445	0.0018			

10 % Recalculated Results for Total Phosphorus in Surface Water

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-4-00 Checked by njs 4-13-00

Sample	QC Batch	Qualifier Note	Dilution	Rep um	Base Line Factor um	Peak - Base Line um	MW g	Corrected Peak * MW	Sample Results ppm	Detection Limit ppm
M5-673-SWF	EPA1027B		1	0.199175	-0.06	0.259175	31	8.034425	0.0080	0.0006
M5-683-SWF	EPA1027B		1	0.115462	-0.06	0.175462	31	5.439322	0.0054	0.0006
M5-693-SWF	EPA1027B		1	0.119933	-0.06	0.179933	31	5.577923	0.0056	0.0006
M5-703-SWF	EPA1027B		1	0.111397	-0.06	0.171397	31	5.313307	0.0053	0.0006
M5-714-SWF	EPA1027B		1	0.089338	-0.06	0.149338	31	4.629478	0.0046	0.0006

Sample	QC Batch	Qualifier Note	Dilution	Rep um	Base Line Factor um	Peak - Base Line um	MW g	Corrected Peak * MW	Sample Results ppm	Detection Limit*3 ppm	SPK CONC (ppm)	R%	RPD
MS	EPA1027B		1	1.787068	-0.06	1.847	31	57.26	0.05726	0.0018	0.04092	90.6	
MS	EPA1027B		1	1.88099	-0.06	1.941	31	60.17	0.06017	0.0018	0.04092	112.0	-4.959
UMS	EPA1027B		1	0.591248	-0.06	0.651	31	20.19	0.02019	0.0018			
UMS	EPA1027B		1	0.402878	-0.06	0.463	31	14.35	0.01435	0.0018			33.815
CCV	EPA1027B		1	1.432253	-0.06	1.492	31	46.26	0.04626	0.0018	0.05	92.52	
CCV	EPA1027B		1	1.482066	-0.06	1.542	31	47.80	0.04780	0.0018	0.05	95.61	
CCV	EPA1027B		1	1.43088	-0.06	1.491	31	46.22	0.04622	0.0018	0.05	92.43	
CCV	EPA1027B		1	1.482898	-0.06	1.543	31	47.83	0.04783	0.0018	0.05	95.66	
CCV	EPA1027B		1	1.430812	-0.06	1.491	31	46.22	0.04622	0.0018	0.05	92.43	
CCV	EPA1027B		1	1.474779	-0.06	1.535	31	47.58	0.04758	0.0018	0.05	95.16	
CCV	EPA1027B		1	1.441135	-0.06	1.501	31	46.54	0.04654	0.0018	0.05	93.07	
Digestion Blk	EPA1027B	Not Reported											
Digestion Blk	EPA1027B	Not Reported											

Sample	QC Batch	Qualifier Note	Dilution	Rep um	Base Line Factor um	Peak - Base Line um	MW g	Corrected Peak * MW	Sample Results ppm	Detection Limit ppm
M5-726-SWF	EPA1027C		1	0.093085	-0.07	0.163	31	5.055635	0.005	0.0006
M5-738-SWF	EPA1027C		1	0.092087	-0.07	0.162	31	5.024697	0.005	0.0006
M5-828-SWF	EPA1027C		1	0.199503	-0.07	0.270	31	8.354593	0.008	0.0006
M5-944-SWF	EPA1027C		1	0.108243	-0.07	0.178	31	5.525533	0.006	0.0006

Sample	QC Batch	Qualifier Note	Dilution	Rep um	Base Line Factor um	Peak - Base Line um	MW g	Corrected Peak * MW	Sample Results ppm	Detection Limit*3 ppm	SPK CONC (ppm)	R%	RPD
MS	EPA1027C		1	1.696953	-0.07	1.767	31	54.78	0.05478	0.0018	0.04092	84.5	
MS	EPA1027C		1	1.848965	-0.07	1.919	31	59.49	0.05949	0.0018	0.04092	101.8	-8.248
UMS	EPA1027C		1	0.582121	-0.07	0.652	31	20.22	0.02022	0.0018			
UMS	EPA1027C		1	0.504826	-0.07	0.575	31	17.82	0.01782	0.0018			12.600
CCV	EPA1027C		1	1.461504	-0.07	1.532	31	47.48	0.04748	0.0018	0.05	94.95	
CCV	EPA1027C		1	1.387512	-0.07	1.458	31	45.18	0.04518	0.0018	0.05	90.37	
CCV	EPA1027C		1	1.437132	-0.07	1.507	31	46.72	0.04672	0.0018	0.05	93.44	
CCV	EPA1027C		1	1.462591	-0.07	1.533	31	47.51	0.04751	0.0018	0.05	95.02	
CCV	EPA1027C		1	1.432883	-0.07	1.503	31	46.59	0.04659	0.0018	0.05	93.18	
Digestion Blk	EPA1027C	"B"	1	0.06032	-0.07	0.130	31	4.04	0.00404	0.0018			
Digestion Blk	EPA1027C	"B"	1	0.065028	-0.07	0.135	31	4.19	0.00419	0.0018			

: Analysis not performed

: Analysis not required

: Average of Two

SERC Lab / EPA REMAP results

Total Phosphorus (TP) in Water by EPA Method 365.1 (modified)

Data Entered by: njs 03-22-00

Data Entry Checked by: mwb 03-28-00

Sampling Station ID	Matrix	Collection Date	Collection Time	Digestion Date	Run Date	Time Elapsed From Dig	Holding Time (Days)	uM Instrument Reading	Blank Correction	Corrected uM Reading	Phosphorus Units (ppm, ug/g)	Detection Limit (ppm)	QA/QC Batch ID	QA Data %R	QA Data %RPD	QA Data Matrix %R	QA Data Sample RPD	Notes
M5-622-SWF	SW	09/30/99	1125	10/08/99	10/27/99	8	28	0.220441	-0.07	0.290441	0.0090	0.0006	EPA1027A	83	2.7	97.1		
M5-623-SWF	SW	09/30/99	915	10/08/99	10/27/99	8	28	0.324684	-0.07	0.394684	0.012	0.0006	EPA1027A	83	2.7	97.1		
M5-624-SWF	SW	09/30/99	1018	10/08/99	10/27/99	8	28	0.173010	-0.07	0.243010	0.0075	0.0006	EPA1027A	83	2.7	97.1		
M5-625-SWF	SW	09/30/99	1257	10/08/99	10/27/99	8	28	0.196394	-0.07	0.266394	0.0083	0.0006	EPA1027A	83	2.7	97.1		
M5-626-SWF	SW	09/30/99	908	10/08/99	10/27/99	8	28	0.191195	-0.07	0.261195	0.0081	0.0006	EPA1027A	83	2.7	97.1		
M5-627-SWF	SW	09/30/99	1050	10/08/99	10/27/99	8	28	0.417754	-0.07	0.487754	0.015	0.0006	EPA1027A	83	2.7	97.1		
M5-628-SWF	SW	09/29/99	1716	10/08/99	10/27/99	9	28	0.221019	-0.07	0.291019	0.0090	0.0006	EPA1027A	83	2.7	97.1		
M5-630-SWF	SW	09/28/99	1520	10/08/99	10/27/99	10	28	0.228900	-0.07	0.298900	0.0093	0.0006	EPA1027A	83	2.7	97.1		
M5-631-SWF	SW	09/29/99	1414	10/08/99	10/27/99	9	28	0.243414	-0.07	0.313414	0.0097	0.0006	EPA1027A	83	2.7	97.1		
M5-632-SWF	SW	09/29/99	1010	10/08/99	10/27/99	9	28	1.010300	-0.07	1.080300	0.033	0.0006	EPA1027A	83	2.7	97.1	-1.2	Averaged Result
M5-633-SWF	SW	09/29/99	1115	10/08/99	10/27/99	9	28	0.922642	-0.07	0.992642	0.031	0.0006	EPA1027A	83	2.7	97.1		
M5-634-SWF	SW	09/29/99	1510	10/08/99	10/27/99	9	28	0.241825	-0.07	0.311825	0.0097	0.0006	EPA1027A	83	2.7	97.1		
M5-635-SWF	SW	09/29/99	1216	10/08/99	10/27/99	9	28	0.240206	-0.07	0.310206	0.0096	0.0006	EPA1027A	83	2.7	97.1		
M5-636-SWF	SW	09/29/99	1405	10/08/99	10/27/99	9	28	0.207492	-0.07	0.277492	0.009	0.0006	EPA1027A	83	2.7	97.1		
M5-637-SWF	SW	09/29/99	1210	10/08/99	10/27/99	9	28	0.284648	-0.07	0.354648	0.011	0.0006	EPA1027A	83	2.7	97.1		
M5-638-SWF	SW	09/29/99	1630	10/08/99	10/27/99	9	28	0.227518	-0.07	0.297518	0.0092	0.0006	EPA1027A	83	2.7	97.1		
M5-639-SWF	SW	09/28/99	1715	10/08/99	10/27/99	10	28	1.362922	-0.07	1.432922	0.044	0.0006	EPA1027A	83	2.7	97.1		
M5-640-SWF	SW	09/29/99	1116	10/08/99	10/27/99	9	28	0.410096	-0.07	0.480096	0.015	0.0006	EPA1027A	83	2.7	97.1		
M5-641-SWF	SW	09/28/99	1615	10/08/99	10/27/99	10	28	0.398678	-0.07	0.468678	0.015	0.0006	EPA1027A	83	2.7	97.1		
M5-642-SWF	SW	09/29/99	1011	10/08/99	10/27/99	9	28	3.007040	-0.07	3.077040	0.095	0.0006	EPA1027A	83	2.7	97.1	4.01	Averaged Result
M5-643-SWF	SW	09/29/99	910	10/08/99	10/27/99	9	28	1.001121	-0.07	1.071121	0.0332	0.0006	EPA1027A	83	2.7	97.1		
M5-644-SWF	SW	09/30/99	1145	10/08/99	10/27/99	8	28	0.236817	-0.07	0.306817	0.0095	0.0006	EPA1027A	83	2.7	97.1		
M5-645-SWF	SW	09/28/99	1447	10/08/99	10/27/99	10	28	0.924768	-0.07	0.994768	0.031	0.0006	EPA1027A	83	2.7	97.1		
M5-646-SWF	SW	09/28/99	1515	10/08/99	10/27/99	10	28	0.440584	-0.07	0.510584	0.016	0.0006	EPA1027A	83	2.7	97.1		
M5-647-SWF	SW	09/28/99	1102	10/08/99	10/27/99	10	28	0.348737	-0.07	0.418737	0.013	0.0006	EPA1027A	83	2.7	97.1		
M5-648-SWF	SW	09/28/99	1300	10/08/99	10/27/99	10	28	0.265804	-0.07	0.335804	0.010	0.0006	EPA1027A	83	2.7	97.1		
M5-649-SWF	SW	09/28/99	1620	10/08/99	10/27/99	10	28	0.222554	-0.07	0.292554	0.0091	0.0006	EPA1027A	83	2.7	97.1		
M5-650-SWF	SW	09/28/99	1158	10/08/99	10/27/99	10	28	0.126759	-0.07	0.196759	0.0061	0.0006	EPA1027A	83	2.7	97.1		
M5-651-SWF	SW	09/28/99	1410	10/08/99	10/27/99	10	28	0.381606	-0.07	0.451606	0.014	0.0006	EPA1027A	83	2.7	97.1		
M5-652-SWF	SW	09/28/99	959	10/08/99	10/27/99	10	28	0.179430	-0.07	0.249430	0.0077	0.0006	EPA1027A	83	2.7	97.1	-13.5	Averaged Result
M5-653-SWF	SW	09/28/99	1300	10/08/99	10/27/99	10	28	2.309137	-0.07	2.379137	0.074	0.0006	EPA1027A	83	2.7	97.1		
M5-654-SWF	SW	09/28/99	1145	10/08/99	10/27/99	10	28	0.296783	-0.07	0.366783	0.011	0.0006	EPA1027A	83	2.7	97.1		
M5-655-SWF	SW	09/28/99	1028	10/08/99	10/27/99	10	28	2.465914	-0.07	2.535914	0.079	0.0006	EPA1027A	83	2.7	97.1		
M5-656-SWF	SW	09/28/99	900	10/08/99	10/27/99	10	28	0.254429	-0.07	0.324429	0.0101	0.0006	EPA1027A	83	2.7	97.1		
M5-657-SWF	SW	09/27/99	1751	10/08/99	10/27/99	11	28	0.432250	-0.07	0.502250	0.0156	0.0006	EPA1027A	83	2.7	97.1		
M5-658-SWF	SW	09/28/99	1722	10/08/99	10/27/99	10	28	0.242427	-0.07	0.312427	0.0097	0.0006	EPA1027A	83	2.7	97.1		
M5-659-SWF	SW	09/27/99	1205	10/08/99	10/27/99	11	28	0.215606	-0.07	0.285606	0.0089	0.0006	EPA1027A	83	2.7	97.1		
M5-660-SWF	SW	09/27/99	1450	10/08/99	10/27/99	11	28	0.842130	-0.07	0.912130	0.028	0.0006	EPA1027A	83	2.7	97.1		
M5-661-SWF	SW	09/29/99	857	10/08/99	10/27/99	9	28	0.186499	-0.07	0.256499	0.0080	0.0006	EPA1027A	83	2.7	97.1		
M5-662-SWF	SW	09/27/99	1610	10/08/99	10/27/99	11	28	0.185990	-0.07	0.255990	0.0079	0.0006	EPA1027A	83	2.7	97.1	-3.8	Averaged Result
M5-663-SWF	SW	09/27/99	1330	10/08/99	10/27/99	11	28	0.182714	-0.07	0.252714	0.0078	0.0006	EPA1027A	83	2.7	97.1		

Sampling Station ID	Matrix	Collection Date	Collection Time	Digestion Date	Run Date	Time Elapsed From Dig	Holding Time (Days)	uM Instrument Reading	Blank Correction	Corrected uM Reading	Phosphorus Units (ppm, ug/g)	Detection Limit (ppm)	QA/QC Batch ID	QA Data %R	QA Data %RPD	QA Data Matrix %R	QA Data Sample RPD	Notes
M5-664-SWF	SW	09/27/99	1100	10/08/99	10/27/99	11	28	0.220172	-0.07	0.290172	0.0090	0.0006	EPA1027A	83	2.7	97.1		
M5-665-SWF	SW	09/27/99	1710	10/08/99	10/27/99	11	28	0.142434	-0.07	0.212434	0.0066	0.0006	EPA1027A	83	2.7	97.1		
M5-666-SWF	SW	09/27/99	1655	10/08/99	10/27/99	11	28	0.187586	-0.07	0.257586	0.0080	0.0006	EPA1027A	83	2.7	97.1		
M5-667-SWF	SW	09/27/99	1545	10/08/99	10/27/99	11	28	0.247630	-0.07	0.317630	0.0098	0.0006	EPA1027A	83	2.7	97.1		
M5-668-SWF	SW	09/27/99	1207	10/08/99	10/27/99	11	28	0.195497	-0.07	0.265497	0.0082	0.0006	EPA1027A	83	2.7	97.1		
M5-669-SWF	SW	09/27/99	1000	10/08/99	10/27/99	11	28	0.172297	-0.07	0.242297	0.0075	0.0006	EPA1027A	83	2.7	97.1		
M5-670-SWF	SW	09/27/99	850	10/08/99	10/27/99	11	28	0.127174	-0.07	0.197174	0.0061	0.0006	EPA1027A	83	2.7	97.1		
M5-671-SWF	SW	09/26/99	850	10/08/99	10/27/99	12	28	0.130301	-0.07	0.200301	0.0062	0.0006	EPA1027A	83	2.7	97.1		
M5-672-SWF	SW	09/27/99	1100	10/08/99	10/27/99	11	28	0.155700	-0.06	0.215700	0.0067	0.0006	EPA1027B	86.6	5.1	107	-14.1	Averaged Result
M5-673-SWF	SW	09/27/99	1400	10/08/99	10/27/99	11	28	0.199175	-0.06	0.259175	0.0080	0.0006	EPA1027B	86.6	5.1	107		
M5-674-SWF	SW	09/26/99	1025	10/08/99	10/27/99	12	28	0.140264	-0.06	0.200264	0.0062	0.0006	EPA1027B	86.6	5.1	107		
M5-675-SWF	SW	09/26/99	1130	10/08/99	10/27/99	12	28	0.163569	-0.06	0.223569	0.0069	0.0006	EPA1027B	86.6	5.1	107		
M5-676-SWF	SW	09/28/99	925	10/08/99	10/27/99	10	28	0.225253	-0.06	0.285253	0.0088	0.0006	EPA1027B	86.6	5.1	107		
M5-677-SWF	SW	09/26/99	1310	10/08/99	10/27/99	12	28	0.195496	-0.06	0.255496	0.0079	0.0006	EPA1027B	86.6	5.1	107		
M5-678-SWF	SW	09/26/99	1213	10/08/99	10/27/99	12	28	0.187692	-0.06	0.247692	0.0077	0.0006	EPA1027B	86.6	5.1	107		
M5-679-SWF	SW	09/26/99	1335	10/08/99	10/27/99	12	28	0.162378	-0.06	0.222378	0.0069	0.0006	EPA1027B	86.6	5.1	107		
M5-680-SWF	SW	09/27/99	900	10/08/99	10/27/99	11	28	0.137051	-0.06	0.197051	0.0061	0.0006	EPA1027B	86.6	5.1	107		
M5-681-SWF	SW	09/26/99	1410	10/08/99	10/27/99	12	28	0.118766	-0.06	0.178766	0.0055	0.0006	EPA1027B	86.6	5.1	107		
M5-682-SWF	SW	09/26/99	1615	10/08/99	10/27/99	12	28	0.099750	-0.06	0.159750	0.0050	0.0006	EPA1027B	86.6	5.1	107	7.84	Averaged Result
M5-683-SWF	SW	09/26/99	850	10/08/99	10/27/99	12	28	0.115462	-0.06	0.175462	0.0054	0.0006	EPA1027B	86.6	5.1	107		
M5-684-SWF	SW	09/26/99	1530	10/08/99	10/27/99	12	28	0.107606	-0.06	0.167606	0.0052	0.0006	EPA1027B	86.6	5.1	107		
M5-685-SWF	SW	09/26/99	1434	10/08/99	10/27/99	12	28	0.127229	-0.06	0.187229	0.0058	0.0006	EPA1027B	86.6	5.1	107		
M5-686-SWF	SW	09/25/99	915	10/08/99	10/27/99	13	28	0.177588	-0.06	0.237588	0.0074	0.0006	EPA1027B	86.6	5.1	107		
M5-687-SWF	SW	09/25/99	1035	10/08/99	10/27/99	13	28	0.170491	-0.06	0.230491	0.0071	0.0006	EPA1027B	86.6	5.1	107		
M5-688-SWF	SW	09/26/99	1527	10/08/99	10/27/99	12	28	0.114608	-0.06	0.174608	0.0054	0.0006	EPA1027B	86.6	5.1	107		
M5-689-SWF	SW	09/25/99	1205	10/08/99	10/27/99	13	28	0.100074	-0.06	0.160074	0.0050	0.0006	EPA1027B	86.6	5.1	107		
M5-690-SWF	SW	09/25/99	1350	10/08/99	10/27/99	13	28	0.094508	-0.06	0.154508	0.0048	0.0006	EPA1027B	86.6	5.1	107		
M5-691-SWF	SW	09/25/99	1554	10/08/99	10/27/99	13	28	0.107701	-0.06	0.167701	0.0052	0.0006	EPA1027B	86.6	5.1	107		
M5-692-SWF	SW	09/25/99	1200	10/08/99	10/27/99	13	28	0.149260	-0.06	0.209260	0.0065	0.0006	EPA1027B	86.6	5.1	107	6.4	Averaged Result
M5-693-SWF	SW	09/25/99	1139	10/08/99	10/27/99	13	28	0.119933	-0.06	0.179933	0.0056	0.0006	EPA1027B	86.6	5.1	107		
M5-694-SWF	SW	09/25/99	1702	10/08/99	10/27/99	13	28	0.105080	-0.06	0.165080	0.0051	0.0006	EPA1027B	86.6	5.1	107		
M5-695-SWF	SW	09/25/99	1400	10/08/99	10/27/99	13	28	0.095180	-0.06	0.155180	0.0048	0.0006	EPA1027B	86.6	5.1	107		
M5-696-SWF	SW	09/25/99	1510	10/08/99	10/27/99	13	28	0.099830	-0.06	0.159830	0.0050	0.0006	EPA1027B	86.6	5.1	107		
M5-697-SWF	SW	09/26/99	1637	10/08/99	10/27/99	12	28	0.141901	-0.06	0.201901	0.0063	0.0006	EPA1027B	86.6	5.1	107		
M5-698-SWF	SW	09/25/99	1030	10/08/99	10/27/99	13	28	0.101514	-0.06	0.161514	0.0050	0.0006	EPA1027B	86.6	5.1	107		
M5-699-SWF	SW	09/25/99	1635	10/08/99	10/27/99	13	28	0.101414	-0.06	0.161414	0.0050	0.0006	EPA1027B	86.6	5.1	107		
M5-700-SWF	SW	09/25/99	1750	10/08/99	10/27/99	13	28	0.093274	-0.06	0.153274	0.0048	0.0006	EPA1027B	86.6	5.1	107		
M5-701-SWF	SW	09/26/99	1740	10/08/99	10/27/99	12	28	0.108139	-0.06	0.168139	0.0052	0.0006	EPA1027B	86.6	5.1	107		
M5-702-SWF	SW	09/25/99	918	10/08/99	10/27/99	13	28	0.083187	-0.06	0.143187	0.0044	0.0006	EPA1027B	86.6	5.1	107		
M5-703-SWF	SW	09/24/99	1655	10/08/99	10/27/99	14	28	0.111397	-0.06	0.171397	0.0053	0.0006	EPA1027B	86.6	5.1	107		
M5-704-SWF	SW	09/24/99	1615	10/08/99	10/27/99	14	28	0.113856	-0.06	0.173856	0.0054	0.0006	EPA1027B	86.6	5.1	107		
M5-705-SWF	SW	09/24/99	1725	10/08/99	10/27/99	14	28	0.130517	-0.06	0.190517	0.0059	0.0006	EPA1027B	86.6	5.1	107		
M5-706-SWF	SW	09/24/99	1555	10/08/99	10/27/99	14	28	0.089133	-0.06	0.149133	0.0046	0.0006	EPA1027B	86.6	5.1	107		
M5-707-SWF	SW	09/24/99	1500	10/08/99	10/27/99	14	28	0.152922	-0.06	0.212922	0.0066	0.0006	EPA1027B	86.6	5.1	107		
M5-708-SWF	SW	09/24/99	900	10/08/99	10/27/99	14	28	0.108196	-0.06	0.168196	0.0052	0.0006	EPA1027B	86.6	5.1	107		

Sampling Station ID	Matrix	Collection Date	Collection Time	Digestion Date	Run Date	Time Elapsed From Dig	Holding Time (Days)	uM Instrument Reading	Blank Correction	Corrected uM Reading	Phosphorus Units (ppm, ug/g)	Detection Limit (ppm)	QA/QC Batch ID	QA Data %R	QA Data %RPD	QA Data Matrix %R	QA Data Sample RPD	Notes
M5-709-SWF	SW	09/24/99	1330	10/08/99	10/27/99	14	28	0.125360	-0.06	0.185360	0.0057	0.0006	EPA1027B	86.6	5.1	107	8.2	Averaged Result
M5-710-SWF	SW	09/24/99	1430	10/08/99	10/27/99	14	28	0.092072	-0.06	0.152072	0.0047	0.0006	EPA1027B	86.6	5.1	107		
M5-711-SWF	SW	09/24/99	1115	10/08/99	10/27/99	14	28	0.122320	-0.06	0.182320	0.0057	0.0006	EPA1027B	86.6	5.1	107		
M5-712-SWF	SW	09/24/99	905	10/08/99	10/27/99	14	28	0.084478	-0.06	0.144478	0.0045	0.0006	EPA1027B	86.6	5.1	107		
M5-714-SWF	SW	09/23/99	1715	10/08/99	10/27/99	15	28	0.089338	-0.06	0.149338	0.0046	0.0006	EPA1027B	86.6	5.1	107		
M5-715-SWF	SW	09/24/99	1145	10/08/99	10/27/99	14	28	0.072499	-0.06	0.132499	0.0041	0.0006	EPA1027B	86.6	5.1	107		
M5-716-SWF	SW	09/24/99	1310	10/08/99	10/27/99	14	28	0.087589	-0.06	0.147589	0.0046	0.0006	EPA1027B	86.6	5.1	107		
M5-718-SWF	SW	09/24/99	1030	10/08/99	10/27/99	14	28	0.086011	-0.06	0.146011	0.0045	0.0006	EPA1027B	86.6	5.1	107		
M5-719-SWF	SW	09/23/99	1715	10/08/99	10/27/99	15	28	0.103145	-0.06	0.163145	0.0051	0.0006	EPA1027B	86.6	5.1	107		
M5-720-SWF	SW	09/23/99	0	10/08/99	10/27/99	15	28	0.088199	-0.06	0.148199	0.0046	0.0006	EPA1027B	86.6	5.1	107		
M5-722-SWF	SW	09/23/99	1600	10/08/99	10/27/99	15	28	0.084810	-0.06	0.144810	0.0045	0.0006	EPA1027B	86.6	5.1	107	4	Averaged Result
M5-723-SWF	SW	09/23/99	1500	10/08/99	10/27/99	15	28	0.083362	-0.06	0.143362	0.0044	0.0006	EPA1027B	86.6	5.1	107		
M5-724-SWF	SW	09/23/99	1442	10/08/99	10/27/99	15	28	0.098319	-0.07	0.168319	0.0052	0.0006	EPA1027C	83.5	8.6	98.4		
M5-725-SWF	SW	09/23/99	1323	10/08/99	10/27/99	15	28	0.088023	-0.07	0.158023	0.0049	0.0006	EPA1027C	83.5	8.6	98.4		
M5-726-SWF	SW	09/23/99	1230	10/08/99	10/27/99	15	28	0.093085	-0.07	0.163085	0.0051	0.0006	EPA1027C	83.5	8.6	98.4		
M5-727-SWF	SW	09/23/99	1216	10/08/99	10/27/99	15	28	0.087421	-0.07	0.157421	0.0049	0.0006	EPA1027C	83.5	8.6	98.4		
M5-728-SWF	SW	09/23/99	1350	10/08/99	10/27/99	15	28	0.117803	-0.07	0.187803	0.0058	0.0006	EPA1027C	83.5	8.6	98.4		
M5-729-SWF	SW	09/23/99	1027	10/08/99	10/27/99	15	28	0.095400	-0.07	0.165400	0.0051	0.0006	EPA1027C	83.5	8.6	98.4		
M5-730-SWF	SW	09/23/99	1120	10/08/99	10/27/99	15	28	0.119505	-0.07	0.189505	0.0059	0.0006	EPA1027C	83.5	8.6	98.4		
M5-731-SWF	SW	09/22/99	1725	10/08/99	10/27/99	16	28	0.080596	-0.07	0.150596	0.0047	0.0006	EPA1027C	83.5	8.6	98.4		
M5-732-SWF	SW	09/23/99	917	12/09/99	12/21/99	77	28	-0.019733	-0.02	0.000267	ND	0.0006	RR1221B	99.3	4.5	103.5		Sample was RR at Later Date
M5-733-SWF	SW	09/23/99	910	10/08/99	10/27/99	15	28	0.125068	-0.07	0.195068	0.0060	0.0006	EPA1027C	83.5	8.6	98.4		
M5-734-SWF	SW	09/22/99	1540	10/08/99	10/27/99	16	28	0.115092	-0.07	0.185092	0.0057	0.0006	EPA1027C	83.5	8.6	98.4		
M5-735-SWF	SW	09/22/99	1700	10/08/99	10/27/99	16	28	0.116421	-0.07	0.186421	0.0058	0.0006	EPA1027C	83.5	8.6	98.4		
M5-738-SWF	SW	09/22/99	1410	10/08/99	10/27/99	16	28	0.092087	-0.07	0.162087	0.0050	0.0006	EPA1027C	83.5	8.6	98.4		
M5-740-SWF	SW	09/22/99	1245	10/08/99	10/27/99	16	28	0.107290	-0.07	0.177290	0.0055	0.0006	EPA1027C	83.5	8.6	98.4		
M5-741-SWF	SW	09/22/99	1534	10/08/99	10/27/99	16	28	0.107995	-0.07	0.177995	0.0055	0.0006	EPA1027C	83.5	8.6	98.4		
M5-742-SWF	SW	09/22/99	1418	10/08/99	10/27/99	16	28	0.085193	-0.07	0.155193	0.0048	0.0006	EPA1027C	83.5	8.6	98.4		
M5-743-SWF	SW	09/22/99	1130	10/08/99	10/27/99	16	28	0.062130	-0.07	0.132130	0.0041	0.0006	EPA1027C	83.5	8.6	98.4		
M5-744-SWF	SW	09/22/99	1224	10/08/99	10/27/99	16	28	0.061631	-0.07	0.131631	0.0041	0.0006	EPA1027C	83.5	8.6	98.4		
M5-745-SWF	SW	09/22/99	1120	10/08/99	10/27/99	16	28	0.084750	-0.07	0.154750	0.0048	0.0006	EPA1027C	83.5	8.6	98.4	1.5	Averaged Result
M5-746-SWF	SW	09/22/99	948	10/08/99	10/27/99	16	28	0.103167	-0.07	0.173167	0.0054	0.0006	EPA1027C	83.5	8.6	98.4		
M5-747-SWF	SW	09/22/99	942	10/08/99	10/27/99	16	28	0.131585	-0.07	0.201585	0.0062	0.0006	EPA1027C	83.5	8.6	98.4		
M5-823-SWF	SW	09/30/99	0	10/08/99	10/27/99	8	28	0.257252	-0.07	0.327252	0.010	0.0006	EPA1027C	83.5	8.6	98.4		
M5-828-SWF	SW	09/29/99	0	10/08/99	10/27/99	9	28	0.199503	-0.07	0.269503	0.0084	0.0006	EPA1027C	83.5	8.6	98.4		
M5-838-SWF	SW	09/29/99	0	10/08/99	10/27/99	9	28	0.230777	-0.07	0.300777	0.0093	0.0006	EPA1027C	83.5	8.6	98.4		
M5-848-SWF	SW	09/28/99	0	10/08/99	10/27/99	10	28	0.282932	-0.07	0.352932	0.011	0.0006	EPA1027C	83.5	8.6	98.4		
M5-859-SWF	SW	09/27/99	0	10/08/99	10/27/99	11	28	0.207392	-0.07	0.277392	0.0086	0.0006	EPA1027C	83.5	8.6	98.4		
M5-868-SWF	SW	09/27/99	0	10/08/99	10/27/99	11	28	0.202190	-0.07	0.272190	0.0084	0.0006	EPA1027C	83.5	8.6	98.4		
M5-878-SWF	SW	09/26/99	0	10/08/99	10/27/99	12	28	0.161276	-0.07	0.231276	0.0072	0.0006	EPA1027C	83.5	8.6	98.4		
M5-890-SWF	SW	09/25/99	0	10/08/99	10/27/99	13	28	0.101030	-0.07	0.171030	0.0053	0.0006	EPA1027C	83.5	8.6	98.4	8.3	Averaged Result
M5-908-SWF	SW	09/24/99	0	10/08/99	10/27/99	14	28	0.105677	-0.07	0.175677	0.0054	0.0006	EPA1027C	83.5	8.6	98.4		
M5-920-SWF	SW	09/23/99	0	10/08/99	10/27/99	15	28	0.089756	-0.07	0.159756	0.0050	0.0006	EPA1027C	83.5	8.6	98.4		
M5-932-SWF	SW	09/23/99	0	10/08/99	10/27/99	15	28	0.098859	-0.07	0.168859	0.0052	0.0006	EPA1027C	83.5	8.6	98.4		
M5-944-SWF	SW	09/22/99	1224	10/08/99	10/27/99	16	28	0.108243	-0.07	0.178243	0.0055	0.0006	EPA1027C	83.5	8.6	98.4		

Sampling Station ID	Matrix	Collection Date	Collection Time	Digestion Date	Run Date	Time Elapsed From Dig	Holding Time (Days)	uM Instrument Reading	Blank Correction	Corrected uM Reading	Phosphorus Units (ppm, ug/g)	Detection Limit (ppm)	QA/QC Batch ID	QA Data % R	QA Data %RPD	QA Data Matrix %R	QA Data Sample RPD	Notes
QA-042-PEF	QA			10/08/99	11/04/99	36441	28	4.983843	-0.05	5.033843	0.16	0.0006	QAPEF-B	119.0	3.8	91.5		
QA-031-PEF	QA			10/08/99	11/04/99	36441	28	0.092094	-0.05	0.142094	0.0044	0.0006	QAPEF-B	119.0	3.8	91.5		
QA-009-CB1	QA			10/08/99	11/04/99	36441	28	0.037971	-0.05	0.087971	0.0027	0.0006	QAPEF-B	119.0	3.8	91.5		
QA-008-CB1	QA			10/08/99	11/04/99	36441	28	0.006699	-0.05	0.056699	0.0018	0.0006	QAPEF-B	119.0	3.8	91.5		
QA-004-CB1	QA			10/08/99	11/04/99	36441	28	-0.019526	-0.05	0.030474	ND	0.0006	QAPEF-B	119.0	3.8	91.5		
QA-003-CB2	QA			10/08/99	11/04/99	36441	28	-0.032538	-0.05	0.017462	ND	0.0006	QAPEF-B	119.0	3.8	91.5	0	Averaged Result
QA-002-CB1	QA			10/08/99	11/04/99	36441	28	-0.045807	-0.05	0.004193	ND	0.0006	QAPEF-B	119.0	3.8	91.5		
QA-001-CB2	QA			10/08/99	11/04/99	36441	28	-0.041997	-0.05	0.008003	ND	0.0006	QAPEF-B	119.0	3.8	91.5		
QA-006-CB2	QA			10/08/99	11/04/99	36441	28	-0.052202	-0.05	-0.002202	ND	0.0006	QAPEF-B	119.0	3.8	91.5		
QA-008-CB2	QA			10/08/99	11/04/99	36441	28	-0.051369	-0.05	-0.001369	ND	0.0006	QAPEF-B	119.0	3.8	91.5		
QA-003-CB1	QA			10/08/99	11/04/99	36441	28	-0.051713	-0.05	-0.001713	ND	0.0006	QAPEF-B	119.0	3.8	91.5		
QA-001-CB1	QA			10/08/99	11/04/99	36441	28	-0.049857	-0.05	0.000143	ND	0.0006	QAPEF-B	119.0	3.8	91.5		
QA-004-CB1	QA			10/08/99	11/04/99	36441	28	-0.044320	-0.05	0.005680	ND	0.0006	QAPEF-B	119.0	3.8	91.5		
QA-007-CB2	QA			10/08/99	11/04/99	36441	28	-0.022949	-0.05	0.027051	ND	0.0006	QAPEF-B	119.0	3.8	91.5		
QA-006-CB1	QA			10/08/99	11/04/99	36441	28	-0.039655	-0.05	0.010345	ND	0.0006	QAPEF-B	119.0	3.8	91.5		
QA-005-CB2	QA			10/08/99	11/04/99	36441	28	-0.050702	-0.05	-0.000702	ND	0.0006	QAPEF-B	119.0	3.8	91.5	0	Averaged Result
QA-009-CB1	QA			10/08/99	11/04/99	36441	28	-0.075141	-0.05	-0.025141	ND	0.0006	QAPEF-B	119.0	3.8	91.5		
QA-005-CB1	QA			10/08/99	11/04/99	36441	28	-0.064787	-0.05	-0.014787	ND	0.0006	QAPEF-B	119.0	3.8	91.5		
QA-007-CB1	QA			10/08/99	11/04/99	36441	28	-0.086683	-0.05	-0.036683	ND	0.0006	QAPEF-B	119.0	3.8	91.5		
QA-002-CB2	QA			10/08/99	11/04/99	36441	28	-0.098451	-0.05	-0.048451	ND	0.0006	QAPEF-B	119.0	3.8	91.5		

10 % Recalculated Results for Total Nitrogen in Surface Water

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-4-00 Checked by njs 4-13-00

Sample	QC Batch	Qualifier Note	Dilution	Rep 1 uM	Rep 2 uM	Rep 3 uM	Rep 4 uM	Rep 5 uM	Rep 6 uM	Average Replicates uM	Blank Correction uM	Corrected Peak uM	Slope	Average Replicate x Slope	Dilution Correction	Sample Results ppm	Detection Limit /3 times (ppm)	SPK CONC (ppm)	R%	RPD
M5-622-SWF	ANTEK 11-22-99		1	343227	318989	341182				334466		334466	3.46767E-06	1.16	1	1.16	0.03/0.09			
M5-633-SWF	ANTEK 11-22-99		1	511879	844769	318851				558500		558500	3.46767E-06	1.94	1	1.94	0.03/0.09			
M5-643-SWF	ANTEK 11-22-99		1	413455	343769	349515				368913		368913	3.46767E-06	1.28	1	1.28	0.03/0.09			
M5-646-SWF	ANTEK 11-22-99		1	467139	462189	411953				447094		447094	3.46767E-06	1.55	1	1.55	0.03/0.09			
M5-646-SWF-DUP	ANTEK 11-22-99		1	357426	384134	609733				450431		450431	3.46767E-06	1.56	1	1.56	0.03/0.09			-0.744
QC CHECK	ANTEK 11-22-99		1	560256	560542	545656				555485		555485	3.46767E-06	1.93	1	1.93	0.03/0.09	2	96.31	
FINAL QC CHECK	ANTEK 11-22-99		1	511349	689147	NO DATA	532575	505166	516596	550967		550967	3.46767E-06	1.91	1	1.91	0.03/0.09	2	95.53	
CALIBRATION(CCV)	ANTEK 11-22-99		1	599725	617711	597817				605084		605084	3.46767E-06	2.10	1	2.10	0.03/0.09	2	104.91	
UMS	ANTEK 11-22-99		1	224783	235423	201768	190281	200777	195350	208064		208064	3.46767E-06	0.72	1	0.72	0.03/0.09			
MS	ANTEK 11-22-99		1	427292	426493	439335				431040		431040	3.46767E-06	1.49	1	1.49	0.03/0.09	1	77.32	
MSD	ANTEK 11-22-99		1	447712	441435	445373				444840		444840	3.46767E-06	1.54	1	1.54	0.03/0.09	1	82.11	-3.151
M5-653-SWF	ANTEK 11-22-99		1	441081	467484	454801				454455		454455	4.08217E-06	1.86	1	1.86	0.03/0.09			
M5-663-SWF	ANTEK 11-22-99		1	248754	125909	130425				168363		168363	4.08217E-06	0.69	1	0.69	0.03/0.09			
M5-670-SWF	ANTEK 11-22-99		1	132693	178325	189752				166923		166923	4.08217E-06	0.68	1	0.68	0.03/0.09			
M5-670-SWF-DUP	ANTEK 11-22-99		1	138862	184219	190633				171238		171238	4.08217E-06	0.70	1	0.70	0.03/0.09			-2.552
QC CHECK	ANTEK 11-22-99		1	493197	494798	503545				497180		497180	4.08217E-06	2.03	1	2.03	0.03/0.09	2	101.48	
FINAL QC CHECK	ANTEK 11-22-99		1	530841	479427	500823	486774	481913	488754	494755		494755	4.08217E-06	2.02	1	2.02	0.03/0.09	2	100.98	
CALIBRATION(CCV)	ANTEK 11-22-99		1	488747	486099	480706				485184		485184	4.08217E-06	1.98	1	1.98	0.03/0.09	2	99.03	
UMS	ANTEK 11-22-99		1	159247	157095	153871	129351	143166	169985	152119		152119	4.08217E-06	0.62	1	0.62	0.03/0.09			
MS	ANTEK 11-22-99		1	360735	357060	352655				356817		356817	4.08217E-06	1.46	1	1.46	0.03/0.09	1	83.56	
MSD	ANTEK 11-22-99		1	357436	357852	375127				363472		363472	4.08217E-06	1.48	1	1.48	0.03/0.09	1	86.28	-1.848
M5-673-SWF	ANTEK 11-23-99		1	124723	130483	142041				132416		132416	3.85884E-06	0.51	1	0.51	0.03/0.09			
M5-683-SWF	ANTEK 11-23-99		1	86567	111428	157823				118606		118606	3.85884E-06	0.46	1	0.46	0.03/0.09			
M5-693-SWF	ANTEK 11-23-99		1	175772	183734	152063				170523		170523	3.85884E-06	0.66	1	0.66	0.03/0.09			
M5-694-SWF	ANTEK 11-23-99		1	196447	178343	188588				187793		187793	3.85884E-06	0.72	1	0.72	0.03/0.09			
M5-694-SWF-DUP	ANTEK 11-23-99		1	211076	218146	211602				213608		213608	3.85884E-06	0.82	1	0.82	0.03/0.09			-12.863
QC CHECK	ANTEK 11-23-99		1	531665	499298	501055				510673		510673	3.85884E-06	1.97	1	1.97	0.03/0.09	2	98.53	
FINAL QC CHECK	ANTEK 11-23-99		1	537072	509934	517494	518542	506820	533937	520633		520633	3.85884E-06	2.01	1	2.01	0.03/0.09	2	100.45	
CALIBRATION(CCV)	ANTEK 11-23-99		1	513634	516662	504139				511478		511478	3.85884E-06	1.97	1	1.97	0.03/0.09	2	98.69	
UMS	ANTEK 11-23-99		1	154071	148739	140462	155780	137622	140945	146270		146270	3.85884E-06	0.56	1	0.56	0.03/0.09			
MS	ANTEK 11-23-99		1	378548	373538	385616				379234		379234	3.85884E-06	1.46	1	1.46	0.03/0.09	1	89.90	
MSD	ANTEK 11-23-99		1	381315	382807	417639				393920		393920	3.85884E-06	1.52	1	1.52	0.03/0.09	1	95.56	-3.799

10 % Recalculated Results for Total Nitrogen in Surface Water

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-4-00 Checked by njs 4-13-00

Sample	QC Batch	Qualifier Note	Dilution	Rep 1 uM	Rep 2 uM	Rep 3 uM	Rep 4 uM	Rep 5 uM	Rep 6 uM	Average Replicates uM	Blank Correction uM	Corrected Peak uM	Slope	Average Replicate x Slope	Dilution Correction	Sample Results ppm	Detection Limit /3 times (ppm)	SPK CONC (ppm)	R%	RPD
M5-703-SWF	ANTEK 11-23-99		1	198265	209979	200283				202842		202842	3.98802E-06	0.81	1	0.81	0.03/0.09			
M5-714-SWF	ANTEK 11-23-99		1	146542	149006	154812				150120		150120	3.98802E-06	0.60	1	0.60	0.03/0.09			
M5-720-SWF	ANTEK 11-23-99		1	157942	157993	263983				193306		193306	3.98802E-06	0.77	1	0.77	0.03/0.09			
M5-720-SWF-DUP	ANTEK 11-23-99		1	204677	175963	192998				191213		191213	3.98802E-06	0.76	1	0.76	0.03/0.09			1.089
QC CHECK	ANTEK 11-23-99		1	494705	500641	501261				498869		498869	3.98802E-06	1.99	1	1.99	0.03/0.09	2	99.47	
FINAL QC CHECK	ANTEK 11-23-99		1	538788	515388	546990	517572	522517	516741	526333		526333	3.98802E-06	2.10	1	2.10	0.03/0.09	2	104.95	
CALIBRATION (CCV)	ANTEK 11-23-99		1	531113	538721	533640				534491		534491	3.98802E-06	2.13	1	2.13	0.03/0.09	2	106.58	
UMS	ANTEK 11-23-99		1	192383	NO DATA	166266	162009	178392	NO DATA	174763		174763	3.98802E-06	0.70	1	0.70	0.03/0.09			
MS	ANTEK 11-23-99		1	399174	NO DATA	422194				410684		410684	3.98802E-06	1.64	1	1.64	0.03/0.09	1	94.09	
MSD	ANTEK 11-23-99		1	470479	473802	437260				460514		460514	3.98802E-06	1.84	1	1.84	0.03/0.09	1	113.96	-11.439
M5-726-SWF	ANTEK 11-23-99		1	101698	97999	102123				100607		100607	3.97803E-06	0.40	1	0.40	0.03/0.09			
M5-738-SWF	ANTEK 11-23-99		1	55020	53674	58051				55582		55582	3.97803E-06	0.22	1	0.22	0.03/0.09			
M5-828-SWF	ANTEK 11-23-99		1	142608	128039	141499				137382		137382	3.97803E-06	0.55	1	0.55	0.03/0.09			
M5-823-SWF	ANTEK 11-23-99		1	479929	254735	221256				318640		318640	3.97803E-06	1.27	1	1.27	0.03/0.09			
M5-823-SWF-DUP	ANTEK 11-23-99		1	332540	263435	271018				288998		288998	3.97803E-06	1.15	1	1.15	0.03/0.09			9.757
QC CHECK	ANTEK 11-23-99		1	499770	501408	508584				503254		503254	3.97803E-06	2.00	1	2.00	0.03/0.09	2	100.10	
FINAL QC CHECK	ANTEK 11-23-99		1	521009	517753	511477	510048	506203	502547	511506		511506	3.97803E-06	2.03	1	2.03	0.03/0.09	2	101.74	
CALIBRATION (CCV)	ANTEK 11-23-99		1	500661	497732	499122				499172		499172	3.97803E-06	1.99	1	1.99	0.03/0.09	2	99.29	
UMS	ANTEK 11-23-99		1	185143	190928	191269	190971	186138	182724	187862		187862	3.97803E-06	0.75	1	0.75	0.03/0.09			
MS	ANTEK 11-23-99		1	454798	476171	460173				463714		463714	3.97803E-06	1.84	1	1.84	0.03/0.09	1	109.73	
MSD	ANTEK 11-23-99		1	488734	422468	419473				443558		443558	3.97803E-06	1.76	1	1.76	0.03/0.09	1	101.72	4.443
M5-944-SWF	ANTEK 2-24-00	"H"	1	75363	71121	80588				75691		75691	3.75455E-06	0.28	1	0.28	0.03/0.09			
QA-PEF-042	ANTEK 2-24-00		1	91241	88966	84138				88115		88115	3.75455E-06	0.33	1	0.33	0.03/0.09			
QC CHECK	ANTEK 2-24-00		1	557158	541509	516509				538392		538392	3.75455E-06	2.02	1	2.02	0.03/0.09	2	101.07	
FINAL QC CHECK	ANTEK 2-24-00		1	531646	450310	446252	431370	432341	420695	452102		452102	3.75455E-06	1.70	1	1.70	0.03/0.09	2	84.87	
CALIBRATION (CCV)	ANTEK 2-24-00		1	466930	473767	449217				463305		463305	3.75455E-06	1.74	1	1.74	0.03/0.09	2	86.98	
UMS	ANTEK 2-24-00		1	235981	220498	211487	214280	208237	204126	215768		215768	3.75455E-06	0.81	1	0.81	0.03/0.09			
MS	ANTEK 2-24-00	"M"	1	418044	422999	403444				414829		414829	3.75455E-06	1.56	1	1.56	0.03/0.09	1	74.74	
MSD	ANTEK 2-24-00	"M"	1	412190	406787	408984				409320		409320	3.75455E-06	1.54	1	1.54	0.03/0.09	1	72.67	1.337

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

Total Nitrogen in Surface Water by Antek 7000N Analyzer

Analyzed by Florida International University (SERC) for the M5 Sampling Event

Entered by P Meyer 3-16-00 Checked by MWB 3-17-99

Sample ID	Collection Date	Collection Time	Digestion Date	Analysis Date	Holding Time	Time since Digestion	Batch ID	Dilution	Rep 1 uM	Rep 2 uM	Rep 3 uM	Rep 4 uM	Rep 5 uM	Rep 6 uM	Average Replicates uM	Blank Correction uM	Corrected Peak uM	Slope	Sample Results ppm	Detection Limit /3 times (ppm)	SPK CONC (ppm)	R%	RPD	Notes
Calibration Std							ANTEK 2-24-00	1	525701	526548	545812				532687			3.75455E-06	2.0	0.03/0.09				
M5-859-SWF	09/27/99		10/08/99	02/24/00	28	11	ANTEK 2-24-00	1	215269	219742	262595				232535			3.75455E-06	0.87	0.03/0.09				
M5-868-SWF	09/27/99		10/08/99	02/24/00	28	11	ANTEK 2-24-00	1	154271	150371	132374				145672			3.75455E-06	0.55	0.03/0.09				
M5-878-SWF	09/26/99	0	10/08/99	02/24/00	28	12	ANTEK 2-24-00	1	134538	120681	135089				130103			3.75455E-06	0.49	0.03/0.09				
M5-890-SWF	09/25/99	0	10/08/99	02/24/00	28	13	ANTEK 2-24-00	1	144939	121187	139085				135070			3.75455E-06	0.51	0.03/0.09				
M5-908-SWF	09/24/99	0	10/08/99	02/24/00	28	14	ANTEK 2-24-00	1	186405	150770	147939				161705			3.75455E-06	0.61	0.03/0.09				
M5-920-SWF	09/23/99	0	10/08/99	02/24/00	28	15	ANTEK 2-24-00	1	177019	153909	135265				155398			3.75455E-06	0.58	0.03/0.09				
M5-932-SWF	09/23/99	0	10/08/99	02/24/00	28	15	ANTEK 2-24-00	1	90343	93132	92001				91825			3.75455E-06	0.34	0.03/0.09				
M5-944-SWF	09/22/99	1224	10/08/99	02/24/00	28	16	ANTEK 2-24-00	1	75363	71121	80588				75691			3.75455E-06	0.28	0.03/0.09				
QA-PEF-042	09/30/99		10/08/99	02/24/00	28		ANTEK 2-24-00	1	91241	88966	84138				88115			3.75455E-06	0.33	0.03/0.09				
Method Blk							ANTEK 2-24-00	1							#DIV/0!			3.75455E-06	#DIV/0!	0.03/0.09				
QC CHECK			10/08/99	02/24/00			ANTEK 2-24-00	1	557158	541509	516509				538392			3.75455E-06	2.0	0.03/0.09	2	101.1		
FINAL QC CHECK			10/08/99	02/24/00			ANTEK 2-24-00	1	531646	450310	446252	431370	432341	420695	452102			3.75455E-06	1.70	0.03/0.09	2	84.9		
CALIBRATION (CCV)			10/08/99	02/24/00			ANTEK 2-24-00	1	466930	473767	449217				463305			3.75455E-06	1.74	0.03/0.09	2	87.0		
UMS			10/08/99	02/24/00			ANTEK 2-24-00	1	235981	220498	211487	214280	208237	204126	215768			3.75455E-06	0.81	0.03/0.09	0			
MS			10/08/99	02/24/00			ANTEK 2-24-00	1	418044	422999	403444				414829			3.75455E-06	1.56	0.03/0.09	1	74.7		
MSD			10/08/99	02/24/00			ANTEK 2-24-00	1	412190	406787	408984				409320			3.75455E-06	1.54	0.03/0.09	1	72.7	0.702	

Sample ID	Collection Date	Collection Time	Digestion Date	Analysis Date	Holding Time	Time since Digestion	Batch ID	Dilution	Rep 1 uM	Rep 2 uM	Rep 3 uM	Rep 4 uM	Rep 5 uM	Rep 6 uM	Average Replicates uM	Blank Correction uM	Corrected Peak uM	Slope	Sample Results ppm	Detection Limit /3 times (ppm)	SPK CONC (ppm)	R%	RPD	Notes
Calibration Std			10/08/99	11/22/99			ANTEK 11-22-99	1	580137	564196	606086	591585	549470	569066	576757			3.46767E-06	2.00	0.03/0.09				
M5-622-SWF	09/30/99	1125	10/08/99	11/22/99	28	8	ANTEK 11-22-99	1	343227	318989	341182				334466			3.46767E-06	1.2	0.03/0.09				
M5-623-SWF	09/30/99	915	10/08/99	11/22/99	28	8	ANTEK 11-22-99	1	338124	277642	277333				297700			3.46767E-06	1.0	0.03/0.09				
M5-624-SWF	09/30/99	1018	10/08/99	11/22/99	28	8	ANTEK 11-22-99	1	378167	279081	312097				323115			3.46767E-06	1.1	0.03/0.09				
M5-625-SWF	09/30/99	1257	10/08/99	11/22/99	28	8	ANTEK 11-22-99	1	335255	409948	364416				369873			3.46767E-06	1.3	0.03/0.09				
M5-626-SWF	09/30/99	908	10/08/99	11/22/99	28	8	ANTEK 11-22-99	1	621794	581728	663964				622495			3.46767E-06	2.2	0.03/0.09				
M5-627-SWF	09/30/99	1050	10/08/99	11/22/99	28	8	ANTEK 11-22-99	1	642521	467336	476298				528718			3.46767E-06	1.8	0.03/0.09				
M5-628-SWF	09/29/99	1716	10/08/99	11/22/99	28	9	ANTEK 11-22-99	1	265927	206367	209182				227159			3.46767E-06	0.79	0.03/0.09				
M5-630-SWF	09/28/99	1520	10/08/99	11/22/99	28	10	ANTEK 11-22-99	1	835842	213832	223102				424259			3.46767E-06	1.5	0.03/0.09				
M5-631-SWF	09/29/99	1414	10/08/99	11/22/99	28	9	ANTEK 11-22-99	1	357200	237339	320197				304912			3.46767E-06	1.1	0.03/0.09				
M5-632-SWF	09/29/99	1010	10/08/99	11/22/99	28	9	ANTEK 11-22-99	1	365165	445994	912951				574703			3.46767E-06	2.0	0.03/0.09				
M5-633-SWF	09/29/99	1115	10/08/99	11/22/99	28	9	ANTEK 11-22-99	1	511879	844769	318851				558500			3.46767E-06	1.9	0.03/0.09				
M5-634-SWF	09/29/99	1510	10/08/99	11/22/99	28	9	ANTEK 11-22-99	1	361473	745377	442938				516596			3.46767E-06	1.8	0.03/0.09				
M5-635-SWF	09/29/99	1216	10/08/99	11/22/99	28	9	ANTEK 11-22-99	1	905949	481772	268626				552116			3.46767E-06	1.9	0.03/0.09				
M5-636-SWF	09/29/99	1405	10/08/99	11/22/99	28	9	ANTEK 11-22-99	1	372070	423498	461376				418981			3.46767E-06	1.5	0.03/0.09				
M5-637-SWF	09/29/99	1210	10/08/99	11/22/99	28	9	ANTEK 11-22-99	1	253943	259897	381327				298389			3.46767E-06	1.0	0.03/0.09				
M5-638-SWF	09/29/99	1630	10/08/99	11/22/99	28	9	ANTEK 11-22-99	1	236881	423609	400616				353702			3.46767E-06	1.2	0.03/0.09				
M5-639-SWF	09/28/99	1715	10/08/99	11/22/99	28	10	ANTEK 11-22-99	1	327237	365156	257248				316547			3.46767E-06	1.1	0.03/0.09				
M5-640-SWF	09/29/99	1116	10/08/99	11/22/99	28	9	ANTEK 11-22-99	1	188703	138353	144729				157262			3.46767E-06	0.55	0.03/0.09				
M5-641-SWF	09/28/99	1615	10/08/99	11/22/99	28	10	ANTEK 11-22-99	1	310685	302740	258700				290708			3.46767E-06	1.0	0.03/0.09				
M5-642-SWF	09/29/99	1011	10/08/99	11/22/99	28	9	ANTEK 11-22-99	1	461827	698305	525778				561970			3.46767E-06	1.9	0.03/0.09				
M5-643-SWF	09/29/99	910	10/08/99	11/22/99	28	9	ANTEK 11-22-99	1	413455	343769	349515				368913			3.46767E-06	1.3	0.03/0.09				
M5-644-SWF	09/30/99	1145	10/08/99	11/22/99	28	8	ANTEK 11-22-99	1	286634	262451	419779				322955			3.46767E-06	1.1	0.03/0.09				
M5-645-SWF	09/28/99	1447	10/08/99	11/22/99	28	10	ANTEK 11-22-99	1	707285	525432	714747				649155			3.46767E-06	2.3	0.03/0.09				
M5-646-SWF	09/28/99	1515	10/08/99	11/22/99	28	10	ANTEK 11-22-99	1	467139	462189	411953				447094			3.46767E-06	1.6	0.03/0.09			0	Averaged Result
M5-646-SWF-DUP	09/28/99	1515	10/08/99	11/22/99	28	10	ANTEK 11-22-99	1	357426	384134	609733				450431			3.46767E-06	1.6	0.03/0.09				
M5-647-SWF	09/28/99	1102	10/08/99	11/22/99	28	10	ANTEK 11-22-99	1	552856	319623	303044				391841			3.46767E-06	1.4	0.03/0.09				
QC CHECK			10/08/99	11/22/99	28		ANTEK 11-22-99	1	560256	560542	545656				555485			3.46767E-06	1.93	0.03/0.09	2	96.3		
FINAL QC CHECK			10/08/99	11/22/99	28		ANTEK 11-22-99	1	511349	689147	NO DATA	532575	505166	516596	550967			3.46767E-06	1.91	0.03/0.09	2	95.5		
CALIBRATION (CCV)			10/08/99	11/22/99	28		ANTEK 11-22-99	1	599725	617711	597817				605084			3.46767E-06	2.10	0.03/0.09	2	104.9		
UMS			10/08/99	11/22/99	28		ANTEK 11-22-99	1	224783	235423	201768	190281	200777	195350	208064			3.46767E-06	0.72	0.03/0.09	0			
MS			10/08/99	11/22/99	28		ANTEK 11-22-99	1	427292	426493	439335				431040			3.46767E-06	1.49	0.03/0.09	1	77.3		
MSD			10/08/99	11/22/99	28		ANTEK 11-22-99	1	447712	441435	445373				444840			3.46767E-06	1.54	0.03/0.09	1	82.1	-1.501	

Sample ID	Collection Date	Collection Time	Digestion Date	Analysis Date	Holding Time	Time since Digestion	Batch ID	Dilution	Rep 1 uM	Rep 2 uM	Rep 3 uM	Rep 4 uM	Rep 5 uM	Rep 6 uM	Average Replicates uM	Blank Correction uM	Corrected Peak uM	Slope	Sample Results ppm	Detection Limit /3 times (ppm)	SPK CONC (ppm)	R%	RPD	Notes
Calibration Std			10/08/99	11/22/99			ANTEK 11/22/99	1	506725	491602	469167	491126	491089	489906	489936			4.08217E-06	2.00	0.03/0.09				
M5-648-SWF	09/28/99	1300	10/08/99	11/22/99	28	10	ANTEK 11/22/99	1	195415	170765	661397				342526			4.08217E-06	1.40	0.03/0.09				
M5-649-SWF	09/28/99	1620	10/08/99	11/22/99	28	10	ANTEK 11/22/99	1	190073	228829	177650				198851			4.08217E-06	0.81	0.03/0.09				
M5-650-SWF	09/28/99	1158	10/08/99	11/22/99	28	10	ANTEK 11/22/99	1	202719	226317	210854				213297			4.08217E-06	0.87	0.03/0.09				
M5-651-SWF	09/28/99	1410	10/08/99	11/22/99	28	10	ANTEK 11/22/99	1	232284	229024	210486				223931			4.08217E-06	0.91	0.03/0.09				
M5-652-SWF	09/28/99	959	10/08/99	11/22/99	28	10	ANTEK 11/22/99	1	164527	136002	145060				148530			4.08217E-06	0.61	0.03/0.09				
M5-653-SWF	09/28/99	1300	10/08/99	11/22/99	28	10	ANTEK 11/22/99	1	441081	467484	454801				454455			4.08217E-06	1.86	0.03/0.09				
M5-654-SWF	09/28/99	1145	10/08/99	11/22/99	28	10	ANTEK 11/22/99	1	232309	212404	253030				232581			4.08217E-06	0.95	0.03/0.09				
M5-655-SWF	09/28/99	1028	10/08/99	11/22/99	28	10	ANTEK 11/22/99	1	401981	387514	381470				390322			4.08217E-06	1.59	0.03/0.09				
M5-656-SWF	09/28/99	900	10/08/99	11/22/99	28	10	ANTEK 11/22/99	1	158814	118805	125068				134229			4.08217E-06	0.55	0.03/0.09				
M5-657-SWF	09/27/99	1751	10/08/99	11/22/99	28	11	ANTEK 11/22/99	1	257813	259558	318288				278553			4.08217E-06	1.14	0.03/0.09				
M5-658-SWF	09/28/99	1722	10/08/99	11/22/99	28	10	ANTEK 11/22/99	1	197929	226304	309284				244506			4.08217E-06	1.00	0.03/0.09				
M5-659-SWF	09/27/99	1205	10/08/99	11/22/99	28	11	ANTEK 11/22/99	1	235091	224708	225942				228580			4.08217E-06	0.93	0.03/0.09				
M5-660-SWF	09/27/99	1450	10/08/99	11/22/99	28	11	ANTEK 11/22/99	1	256887	250824	209501				239071			4.08217E-06	0.98	0.03/0.09				
M5-661-SWF	09/29/99	857	10/08/99	11/22/99	28	9	ANTEK 11/22/99	1	245546	166289	183237				198357			4.08217E-06	0.81	0.03/0.09				
M5-662-SWF	09/27/99	1610	10/08/99	11/22/99	28	11	ANTEK 11/22/99	1	161244	168397	168970				166204			4.08217E-06	0.68	0.03/0.09				
M5-663-SWF	09/27/99	1330	10/08/99	11/22/99	28	11	ANTEK 11/22/99	1	248754	125909	130425				168363			4.08217E-06	0.69	0.03/0.09				
M5-664-SWF	09/27/99	1100	10/08/99	11/22/99	28	11	ANTEK 11/22/99	1	192693	231429	227007				217043			4.08217E-06	0.89	0.03/0.09				
M5-665-SWF	09/27/99	1710	10/08/99	11/22/99	28	11	ANTEK 11/22/99	1	166592	175533	163906				168677			4.08217E-06	0.69	0.03/0.09				
M5-666-SWF	09/27/99	1655	10/08/99	11/22/99	28	11	ANTEK 11/22/99	1	120440	133857	161569				138622			4.08217E-06	0.57	0.03/0.09				
M5-667-SWF	09/27/99	1545	10/08/99	11/22/99	28	11	ANTEK 11/22/99	1	113845	134726	142422				130331			4.08217E-06	0.53	0.03/0.09				
M5-668-SWF	09/27/99	1207	10/08/99	11/22/99	28	11	ANTEK 11/22/99	1	119877	109636	125466				118326			4.08217E-06	0.48	0.03/0.09				
M5-669-SWF	09/27/99	1000	10/08/99	11/22/99	28	11	ANTEK 11/22/99	1	170769	200535	198617				189974			4.08217E-06	0.78	0.03/0.09				
M5-670-SWF	09/27/99	850	10/08/99	11/22/99	28	11	ANTEK 11/22/99	1	132693	178325	189752				166923			4.08217E-06	0.69	0.03/0.09			-2.9	Averaged Result
QC CHECK			10/08/99	11/22/99	28		ANTEK 11/22/99	1	493197	494798	503545				497180			4.08217E-06	2.03	0.03/0.09	2	101.5		
FINAL QC CHECK			10/08/99	11/22/99	28		ANTEK 11/22/99	1	530841	479427	500823	486774	481913	488754	494755			4.08217E-06	2.02	0.03/0.09	2	101.0		
CALIBRATION (CCV)			10/08/99	11/22/99	28		ANTEK 11/22/99	1	488747	486099	480706				485184			4.08217E-06	1.98	0.03/0.09	2	99.0		
UMS			10/08/99	11/22/99	28		ANTEK 11/22/99	1	159247	157095	153871	129351	143166	169985	152119			4.08217E-06	0.62	0.03/0.09	0			
MS			10/08/99	11/22/99	28		ANTEK 11-22-99	1	360735	357060	352655				356817			4.08217E-06	1.46	0.03/0.09	1	83.6		
MSD			10/08/99	11/22/99	28		ANTEK 11-22-99	1	357436	357852	375127				363472			4.08217E-06	1.48	0.03/0.09	1	86.3	-0.800	

Sample ID	Collection Date	Collection Time	Digestion Date	Analysis Date	Holding Time	Time since Digestion	Batch ID	Dilution	Rep 1 uM	Rep 2 uM	Rep 3 uM	Rep 4 uM	Rep 5 uM	Rep 6 uM	Average Replicates uM	Blank Correction uM	Corrected Peak uM	Slope	Sample Results ppm	Detection Limit /3 times (ppm)	SPK CONC (ppm)	R%	RPD	Notes
Calibration Std			10/08/99	11/23/99			ANTEK 11/23/99	1	500975	497931	509543	607795	492210	501289	518291			3.85884E-06	2.00	0.03/0.09				
M5-671-SWF	09/26/99	850	10/08/99	11/23/99	28	12	ANTEK 11/23/99	1	159878	145674	155948				153833			3.85884E-06	0.59	0.03/0.09				
M5-672-SWF	09/27/99	1100	10/08/99	11/23/99	28	11	ANTEK 11/23/99	1	104355	101760	95662				100592			3.85884E-06	0.39	0.03/0.09				
M5-673-SWF	09/27/99	1400	10/08/99	11/23/99	28	11	ANTEK 11/23/99	1	124723	130483	142041				132416			3.85884E-06	0.51	0.03/0.09				
M5-674-SWF	09/26/99	1025	10/08/99	11/23/99	28	12	ANTEK 11/23/99	1	125753	124615	122624				124331			3.85884E-06	0.48	0.03/0.09				
M5-675-SWF	09/26/99	1130	10/08/99	11/23/99	28	12	ANTEK 11/23/99	1	152390	159548	133916				148618			3.85884E-06	0.57	0.03/0.09				
M5-676-SWF	09/28/99	925	10/08/99	11/23/99	28	10	ANTEK 11/23/99	1	231671	120766	105473				152637			3.85884E-06	0.59	0.03/0.09				
M5-677-SWF	09/26/99	1310	10/08/99	11/23/99	28	12	ANTEK 11/23/99	1	262468	204605	136064				201046			3.85884E-06	0.78	0.03/0.09				
M5-678-SWF	09/26/99	1213	10/08/99	11/23/99	28	12	ANTEK 11/23/99	1	174053	90666	99955				121558			3.85884E-06	0.47	0.03/0.09				
M5-679-SWF	09/26/99	1335	10/08/99	11/23/99	28	12	ANTEK 11/23/99	1	171738	174425	130581				158915			3.85884E-06	0.61	0.03/0.09				
M5-680-SWF	09/27/99	900	10/08/99	11/23/99	28	11	ANTEK 11/23/99	1	129009	209021	139227				159086			3.85884E-06	0.61	0.03/0.09				
M5-681-SWF	09/26/99	1410	10/08/99	11/23/99	28	12	ANTEK 11/23/99	1	155596	177528	200755				177960			3.85884E-06	0.69	0.03/0.09				
M5-682-SWF	09/26/99	1615	10/08/99	11/23/99	28	12	ANTEK 11/23/99	1	125590	149978	155942				143837			3.85884E-06	0.56	0.03/0.09				
M5-683-SWF	09/26/99	850	10/08/99	11/23/99	28	12	ANTEK 11/23/99	1	86567	111428	157823				118606			3.85884E-06	0.46	0.03/0.09				
M5-684-SWF	09/26/99	1530	10/08/99	11/23/99	28	12	ANTEK 11/23/99	1	108359	135766	230429				158185			3.85884E-06	0.61	0.03/0.09				
M5-685-SWF	09/26/99	1434	10/08/99	11/23/99	28	12	ANTEK 11/23/99	1	105053	121757	119290				115367			3.85884E-06	0.45	0.03/0.09				
M5-686-SWF	09/25/99	915	10/08/99	11/23/99	28	13	ANTEK 11/23/99	1	151803	169314	211790				177636			3.85884E-06	0.69	0.03/0.09				
M5-687-SWF	09/25/99	1035	10/08/99	11/23/99	28	13	ANTEK 11/23/99	1	196358	157124	191307				181596			3.85884E-06	0.70	0.03/0.09				
M5-688-SWF	09/26/99	1527	10/08/99	11/23/99	28	12	ANTEK 11/23/99	1	129513	92050	96958				106174			3.85884E-06	0.41	0.03/0.09				
M5-689-SWF	09/25/99	1205	10/08/99	11/23/99	28	13	ANTEK 11/23/99	1	251223	250206	271243				257557			3.85884E-06	0.99	0.03/0.09				
M5-690-SWF	09/25/99	1350	10/08/99	11/23/99	28	13	ANTEK 11/23/99	1	141638	122848	116755				127080			3.85884E-06	0.49	0.03/0.09				
M5-691-SWF	09/25/99	1554	10/08/99	11/23/99	28	13	ANTEK 11/23/99	1	117331	85892	97653				100292			3.85884E-06	0.39	0.03/0.09				
M5-692-SWF	09/25/99	1200	10/08/99	11/23/99	28	13	ANTEK 11/23/99	1	94457	100500	115030				103329			3.85884E-06	0.40	0.03/0.09				
M5-693-SWF	09/25/99	1139	10/08/99	11/23/99	28	13	ANTEK 11/23/99	1	175772	183734	152063				170523			3.85884E-06	0.66	0.03/0.09				
M5-694-SWF	09/25/99	1702	10/08/99	11/23/99	28	13	ANTEK 11/23/99	1	196447	178343	188588				187793			3.85884E-06	0.77	0.03/0.09			-13	Averaged Result
M5-695-SWF	09/25/99	1400	10/08/99	11/23/99	28	13	ANTEK 11/23/99	1	179958	187320	196348				187875			3.85884E-06	0.72	0.03/0.09				
QC CHECK			10/08/99	11/23/99			ANTEK 11/23/99	1	531665	499298	501055				510673			3.85884E-06	1.97	0.03/0.09	2	98.5		
FINAL QC CHECK			10/08/99	11/23/99			ANTEK 11/23/99	1	537072	509934	517494	518542	506820	533937	520633			3.85884E-06	2.01	0.03/0.09	2	100.5		
CALIBRATION (CCV)			10/08/99	11/23/99			ANTEK 11/23/99	1	513634	516662	504139				511478			3.85884E-06	1.97	0.03/0.09	2	98.7		
UMS			10/08/99	11/23/99			ANTEK 11/23/99	1	154071	148739	140462	155780	137622	140945	146270			3.85884E-06	0.56	0.03/0.09	0			
MS			10/08/99	11/23/99			ANTEK 11/23/99	1	378548	373538	385616				379234			3.85884E-06	1.46	0.03/0.09	1	89.9		
MSD			10/08/99	11/23/99			ANTEK 11/23/99	1	381315	382807	417639				393920			3.85884E-06	1.52	0.03/0.09	1	95.6	-1.528	

Sample ID	Collection Date	Collection Time	Digestion Date	Analysis Date	Holding Time	Time since Digestion	Batch ID	Dilution	Rep 1 uM	Rep 2 uM	Rep 3 uM	Rep 4 uM	Rep 5 uM	Rep 6 uM	Average Replicates uM	Blank Correction uM	Corrected Peak uM	Slope	Sample Results ppm	Detection Limit /3 times (ppm)	SPK CONC (ppm)	R%	RPD	Notes
Calibration Std			10/08/99	11/23/99			ANTEK 11/23/99	1	508879	495740	492412	499249	500166	512567	501502			3.98802E-06	2.00	0.03/0.09				
M5-696-SWF	09/25/99	1510	10/08/99	11/23/99	28	13	ANTEK 11/23/99	1	162320	219467	178111				186633			3.98802E-06	0.74	0.03/0.09				
M5-697-SWF	09/26/99	1637	10/08/99	11/23/99	28	12	ANTEK 11/23/99	1	173439	219848	290654				227980			3.98802E-06	0.91	0.03/0.09				
M5-698-SWF	09/25/99	1030	10/08/99	11/23/99	28	13	ANTEK 11/23/99	1	284802	153753	167225				201927			3.98802E-06	0.81	0.03/0.09				
M5-699-SWF	09/25/99	1635	10/08/99	11/23/99	28	13	ANTEK 11/23/99	1	325672	353233	312623				330509			3.98802E-06	1.32	0.03/0.09				
M5-700-SWF	09/25/99	1750	10/08/99	11/23/99	28	13	ANTEK 11/23/99	1	239785	227784	260241				242603			3.98802E-06	0.97	0.03/0.09				
M5-701-SWF	09/26/99	1740	10/08/99	11/23/99	28	12	ANTEK 11/23/99	1	453418	295054	203785				317419			3.98802E-06	1.27	0.03/0.09				
M5-702-SWF	09/25/99	918	10/08/99	11/23/99	28	13	ANTEK 11/23/99	1	704815	266464	235091				402123			3.98802E-06	1.60	0.03/0.09				
M5-703-SWF	09/24/99	1655	10/08/99	11/23/99	28	14	ANTEK 11/23/99	1	198265	209979	200283				202842			3.98802E-06	0.81	0.03/0.09				
M5-704-SWF	09/24/99	1615	10/08/99	11/23/99	28	14	ANTEK 11/23/99	1	158737	200058	163973				174256			3.98802E-06	0.69	0.03/0.09				
M5-705-SWF	09/24/99	1725	10/08/99	11/23/99	28	14	ANTEK 11/23/99	1	145238	174366	172897				164167			3.98802E-06	0.65	0.03/0.09				
M5-706-SWF	09/24/99	1555	10/08/99	11/23/99	28	14	ANTEK 11/23/99	1	159852	160606	146831				155763			3.98802E-06	0.62	0.03/0.09				
M5-707-SWF	09/24/99	1500	10/08/99	11/23/99	28	14	ANTEK 11/23/99	1	137108	195536	218820				183821			3.98802E-06	0.73	0.03/0.09				
M5-708-SWF	09/24/99	900	10/08/99	11/23/99	28	14	ANTEK 11/23/99	1	185514	227273	142298				185028			3.98802E-06	0.74	0.03/0.09				
M5-709-SWF	09/24/99	1330	10/08/99	11/23/99	28	14	ANTEK 11/23/99	1	122877	92577	87658				101037			3.98802E-06	0.40	0.03/0.09				
M5-710-SWF	09/24/99	1430	10/08/99	11/23/99	28	14	ANTEK 11/23/99	1	199087	229275	232242				220201			3.98802E-06	0.88	0.03/0.09				
M5-711-SWF	09/24/99	1115	10/08/99	11/23/99	28	14	ANTEK 11/23/99	1	113147	188271	133577				144998			3.98802E-06	0.58	0.03/0.09				
M5-712-SWF	09/24/99	905	10/08/99	11/23/99	28	14	ANTEK 11/23/99	1	183774	192142	152521				176146			3.98802E-06	0.70	0.03/0.09				
M5-714-SWF	09/23/99	1715	10/08/99	11/23/99	28	15	ANTEK 11/23/99	1	146542	149006	154812				150120			3.98802E-06	0.60	0.03/0.09				
M5-715-SWF	09/24/99	1145	10/08/99	11/23/99	28	14	ANTEK 11/23/99	1	90333	128849	147125				122102			3.98802E-06	0.49	0.03/0.09				
M5-716-SWF	09/24/99	1310	10/08/99	11/23/99	28	14	ANTEK 11/23/99	1	171736	173478	144394				163203			3.98802E-06	0.65	0.03/0.09				
M5-718-SWF	09/24/99	1030	10/08/99	11/23/99	28	14	ANTEK 11/23/99	1	148783	156455	145131				150123			3.98802E-06	0.60	0.03/0.09				
M5-719-SWF	09/23/99	1715	10/08/99	11/23/99	28	15	ANTEK 11/23/99	1	155773	194613	211607				187331			3.98802E-06	0.75	0.03/0.09				
M5-720-SWF	09/23/99	0	10/08/99	11/23/99	28	15	ANTEK 11/23/99	1	157942	157993	263983				193306			3.98802E-06	0.77	0.03/0.09			1.3	Averaged Result
M5-722-SWF	09/23/99	1600	10/08/99	11/23/99	28	15	ANTEK 11/23/99	1	162255	115898	131858				136670			3.98802E-06	0.55	0.03/0.09				
M5-723-SWF	09/23/99	1500	10/08/99	11/23/99	28	15	ANTEK 11/23/99	1	122325	139835	99471				120544			3.98802E-06	0.48	0.03/0.09				
QC CHECK			10/08/99	11/23/99			ANTEK 11/23/99	1	494705	500641	501261				498869			3.98802E-06	1.99	0.03/0.09	2	99.5		
FINAL QC CHECK			10/08/99	11/23/99			ANTEK 11/23/99	1	538788	515388	546990	517572	522517	516741	526333			3.98802E-06	2.10	0.03/0.09	2	105.0		
CALIBRATION (CCV)			10/08/99	11/23/99			ANTEK 11/23/99	1	531113	538721	533640				534491			3.98802E-06	2.13	0.03/0.09	2	106.6		
UMS			10/08/99	11/23/99			ANTEK 11/23/99	1	192383	NO DATA	166266	162009	178392	NO DATA	174763			3.98802E-06	0.70	0.03/0.09	0			
MS			10/08/99	11/23/99			ANTEK 11/23/99	1	399174	NO DATA	422194				410684			3.98802E-06	1.64	0.03/0.09	1	94.1		
MSD			10/08/99	11/23/99			ANTEK 11/23/99	1	470479	473802	437260				460514			3.98802E-06	1.84	0.03/0.09	1	114.0	-4.776	

Sample ID	Collection Date	Collection Time	Digestion Date	Analysis Date	Holding Time	Time since Digestion	Batch ID	Dilution	Rep 1 uM	Rep 2 uM	Rep 3 uM	Rep 4 uM	Rep 5 uM	Rep 6 uM	Average Replicates uM	Blank Correction uM	Corrected Peak uM	Slope	Sample Results ppm	Detection Limit /3 times (ppm)	SPK CONC (ppm)	R%	RPD	Notes
Calibration Std			10/08/99	11/23/99			ANTEK 11/23/99	1	499878	496837	495823	500894	513553	509586	502762			3.97803E-06	2.00	0.03/0.09				
M5-724-SWF	09/23/99	1442	10/08/99	11/23/99	28	15	ANTEK 11/23/99	1	177693	204268	210066				197342			3.97803E-06	0.79	0.03/0.09				
M5-725-SWF	09/23/99	1323	10/08/99	11/23/99	28	15	ANTEK 11/23/99	1	132768	106827	117482				119026			3.97803E-06	0.47	0.03/0.09				
M5-726-SWF	09/23/99	1230	10/08/99	11/23/99	28	15	ANTEK 11/23/99	1	101698	97999	102123				100607			3.97803E-06	0.40	0.03/0.09				
M5-727-SWF	09/23/99	1216	10/08/99	11/23/99	28	15	ANTEK 11/23/99	1	91707	103829	141283				112273			3.97803E-06	0.45	0.03/0.09				
M5-728-SWF	09/23/99	1350	10/08/99	11/23/99	28	15	ANTEK 11/23/99	1	112597	125752	100674				113008			3.97803E-06	0.45	0.03/0.09				
M5-729-SWF	09/23/99	1027	10/08/99	11/23/99	28	15	ANTEK 11/23/99	1	99739	103736	103797				102424			3.97803E-06	0.41	0.03/0.09				
M5-730-SWF	09/23/99	1120	10/08/99	11/23/99	28	15	ANTEK 11/23/99	1	126876	129193	126703				127591			3.97803E-06	0.51	0.03/0.09				
M5-731-SWF	09/22/99	1725	10/08/99	11/23/99	28	16	ANTEK 11/23/99	1	129140	136469	108501				124703			3.97803E-06	0.50	0.03/0.09				
M5-732-SWF	09/23/99	917	10/08/99	11/23/99	28	15	ANTEK 11/23/99	1	127159	88978	95881				104006			3.97803E-06	0.41	0.03/0.09				
M5-733-SWF	09/23/99	910	10/08/99	11/23/99	28	15	ANTEK 11/23/99	1	91575	88923	82010				87503			3.97803E-06	0.35	0.03/0.09				
M5-734-SWF	09/22/99	1540	10/08/99	11/23/99	28	16	ANTEK 11/23/99	1	109118	104863	113483				109155			3.97803E-06	0.43	0.03/0.09				
M5-735-SWF	09/22/99	1700	10/08/99	11/23/99	28	16	ANTEK 11/23/99	1	84652	72088	82188				79643			3.97803E-06	0.32	0.03/0.09				
M5-738-SWF	09/22/99	1410	10/08/99	11/23/99	28	16	ANTEK 11/23/99	1	55020	53674	58051				55582			3.97803E-06	0.22	0.03/0.09				
M5-740-SWF	09/22/99	1245	10/08/99	11/23/99	28	16	ANTEK 11/23/99	1	41659	77333	37119				52037			3.97803E-06	0.21	0.03/0.09				
M5-741-SWF	09/22/99	1534	10/08/99	11/23/99	28	16	ANTEK 11/23/99	1	51466	58015	57144				55542			3.97803E-06	0.22	0.03/0.09				
M5-742-SWF	09/22/99	1418	10/08/99	11/23/99	28	16	ANTEK 11/23/99	1	84603	80795	74001				79800			3.97803E-06	0.32	0.03/0.09				
M5-743-SWF	09/22/99	1130	10/08/99	11/23/99	28	16	ANTEK 11/23/99	1	54352	57829	62004				58062			3.97803E-06	0.23	0.03/0.09				
M5-744-SWF	09/22/99	1224	10/08/99	11/23/99	28	16	ANTEK 11/23/99	1	74632	79046	85463				79714			3.97803E-06	0.32	0.03/0.09				
M5-745-SWF	09/22/99	1120	10/08/99	11/23/99	28	16	ANTEK 11/23/99	1	79714	81919	75869				79167			3.97803E-06	0.31	0.03/0.09				
M5-746-SWF	09/22/99	948	10/08/99	11/23/99	28	16	ANTEK 11/23/99	1	84326	79863	76716				80302			3.97803E-06	0.32	0.03/0.09				
M5-747-SWF	09/22/99	942	10/08/99	11/23/99	28	16	ANTEK 11/23/99	1	97048	108171	101574				102264			3.97803E-06	0.41	0.03/0.09				
M5-823-SWF	09/30/99		10/08/99	11/23/99			ANTEK 11/23/99	1	479929	254735	221256				318640			3.97803E-06	1.21	0.03/0.09			9.9	Averaged Result
M5-828-SWF	09/29/99		10/08/99	11/23/99			ANTEK 11/23/99	1	142608	128039	141499				137382			3.97803E-06	0.55	0.03/0.09				
M5-838-SWF	09/29/99		10/08/99	11/23/99			ANTEK 11/23/99	1	165416	173157	173612				170728			3.97803E-06	0.68	0.03/0.09				
M5-848-SWF	09/28/99		10/08/99	11/23/99			ANTEK 11/23/99	1	206276	221671	205263				211070			3.97803E-06	0.84	0.03/0.09				
QC CHECK			10/08/99	11/23/99			ANTEK 11/23/99	1	499770	501408	508584				503254			3.97803E-06	2.00	0.03/0.09	2	100.1		
FINAL QC CHECK			10/08/99	11/23/99			ANTEK 11/23/99	1	521009	517753	511477	510048	506203	502547	511506			3.97803E-06	2.03	0.03/0.09	2	101.7		
CALIBRATION (CCV)			10/08/99	11/23/99			ANTEK 11/23/99	1	500661	497732	499122				499172			3.97803E-06	1.99	0.03/0.09	2	99.3		
UMS			10/08/99	11/23/99			ANTEK 11/23/99	1	185143	190928	191269	190971	186138	182724	187862			3.97803E-06	0.75	0.03/0.09	0			
MS			10/08/99	11/23/99			ANTEK 11/23/99	1	454798	476171	460173				463714			3.97803E-06	1.84	0.03/0.09	1	109.7		
MSD			10/08/99	11/23/99			ANTEK 11/23/99	1	488734	422468	419473				443558			3.97803E-06	1.76	0.03/0.09	1	101.7	1.896	

10 % Recalculated Results for Total Organic Carbon in Surface Water

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-5-00 Checked by njs 4-13-00

Sample	QC Batch	Qualifier Note	Dilution	Instrument Result ppm	Blank Factor ppm	Sample Results ppm	Detection Limit ppm
M5-622-SWF	10-21-99/TOC-B		1	36.77	0	36.77	0.12
M5-633-SWF	10-21-99/TOC-B		1	21.02	0	21.02	0.12
M5-643-SWF	10-21-99/TOC-B		1	24.94	0	24.94	0.12
M5-653-SWF	10-21-99/TOC-B		1	34.31	0	34.31	0.12
M5-663-SWF	10-21-99/TOC-B		1	19.94	0	19.94	0.12
M5-673-SWF	10-21-99/TOC-B		1	18.90	0	18.90	0.12

Sample	QC Batch	Qualifier Note	Dilution	Instrument Result ppm	Blank Factor ppm	Sample Results ppm	Detection Limit*3 ppm	Spike Conc (ppm)	R%	RPD
Method Blank	10-21-99/TOC-B		1	2.331	2.578	-0.25	0.36	0		
Method Blank	10-21-99/TOC-B		1	2.273	2.578	-0.31	0.36	0		
Method Blank	10-21-99/TOC-B		1	2.933	2.578	0.36	0.36	0		
Method Blank	10-21-99/TOC-B		1	2.565	2.578	-0.01	0.36	0		
Method Blank	10-21-99/TOC-B		1	2.668	2.578	0.09	0.36	0		
Method Blank	10-21-99/TOC-B		1	2.698	2.578	0.12	0.36	0		
CCV 10	10-21-99/TOC-B		1	13.04	2.578	10.46	0.36	10	104.62	
CCV 5	10-21-99/TOC-B		1	8.51	2.578	5.93	0.36	5	118.64	
CCV 10	10-21-99/TOC-B		1	13.80	2.578	11.22	0.36	10	112.22	
CCV 5	10-21-99/TOC-B		1	8.405	2.578	5.83	0.36	5	116.54	
CCV 10	10-21-99/TOC-B		1	13.63	2.578	11.05	0.36	10	110.52	
UMS	10-21-99/TOC-B		1	10.73	2.578	8.15	0.36			
UMS	10-21-99/TOC-B		1	10.91	2.578	8.33	0.36			-2.184
MS	10-21-99/TOC-B		1	19.72	2.578	17.14	0.36	8	112.38	
MS	10-21-99/TOC-B		1	19.41	2.578	16.83	0.36	8	106.25	1.825
627	10-21-99/TOC-B		1	40.63	0	40.63	0.36	0		
627D	10-21-99/TOC-B		1	41.15	0	41.15	0.36	0		-1.272
638	10-21-99/TOC-B		1	22.5	0	22.50	0.36	0		
638D	10-21-99/TOC-B		1	22.55	0	22.55	0.36	0		-0.222
648	10-21-99/TOC-B		1	26.89	0	26.89	0.36	0		
948D	10-21-99/TOC-B		1	26.84	0	26.84	0.36	0		0.186
658	10-21-99/TOC-B		1	26.24	0	26.24	0.36	0		
658D	10-21-99/TOC-B		1	26.51	0	26.51	0.36	0		-1.024
668	10-21-99/TOC-B		1	17.19	0	17.19	0.36	0		
668D	10-21-99/TOC-B		1	17.11	0	17.11	0.36	0		0.466

10 % Recalculated Results for Total Organic Carbon in Surface Water

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-5-00 Checked by njs 4-13-00

Sample	QC Batch	Qualifier Note	Dilution	Instrument Result ppm	Blank Factor ppm	Sample Results ppm	Detection Limit ppm
M5-683-SWF	10-22-99/TOC-B		1	11.55	0	11.55	0.12
M5-693-SWF	10-22-99/TOC-B		1	10.22	0	10.22	0.12
M5-703-SWF	10-22-99/TOC-B		1	14.16	0	14.16	0.12
M5-714-SWF	10-22-99/TOC-B		1	13.99	0	13.99	0.12
M5-726-SWF	10-22-99/TOC-B		1	11.50	0	11.50	0.12

Sample	QC Batch	Qualifier Note	Dilution	Instrument Result ppm	Blank Factor ppm	Sample Results ppm	Detection Limit*3 ppm	Spike Conc (ppm)	R%	RPD
Method Blank	10-22-99/TOC-B		1	1.139	1.24	-0.10	0.36	0		
Method Blank	10-22-99/TOC-B		1	1.158	1.24	-0.08	0.36	0		
Method Blank	10-22-99/TOC-B		1	1.307	1.24	0.07	0.36	0		
Method Blank	10-22-99/TOC-B		1	1.161	1.24	-0.08	0.36	0		
Method Blank	10-22-99/TOC-B		1	1.302	1.24	0.06	0.36	0		
Method Blank	10-22-99/TOC-B		1	1.371	1.24	0.13	0.36	0		
CCV 10	10-22-99/TOC-B		1	11.13	1.24	9.89	0.36	10	98.90	
CCV 5	10-22-99/TOC-B		1	6.364	1.24	5.12	0.36	5	102.48	
CCV 10	10-22-99/TOC-B		1	11.22	1.24	9.98	0.36	10	99.80	
CCV 5	10-22-99/TOC-B		1	6.343	1.24	5.10	0.36	5	102.06	
CCV 10	10-22-99/TOC-B		1	11.18	1.24	9.94	0.36	10	99.40	
UMS	10-22-99/TOC-B		1	8.789	1.24	7.55	0.36			
UMS	10-22-99/TOC-B		1	9.143	1.24	7.90	0.36			-4.582
MS	10-22-99/TOC-B		1	16.7	1.24	15.46	0.36	8	98.89	
MS	10-22-99/TOC-B		1	17	1.24	15.76	0.36	8	98.21	-1.922
686	10-22-99/TOC-B		1	18.58	0	18.58	0.36	0		
686D	10-22-99/TOC-B		1	18.78	0	18.78	0.36	0		-1.071
696	10-22-99/TOC-B		1	17.09	0	17.09	0.36	0		
696D	10-22-99/TOC-B		1	17.01	0	17.01	0.36	0		0.469
706	10-22-99/TOC-B		1	10.64	0	10.64	0.36	0		
706D	10-22-99/TOC-B		1	10.25	0	10.25	0.36	0		3.734
718	10-22-99/TOC-B		1	12.1	0	12.1	0.36	0		
718D	10-22-99/TOC-B		1	11.32	0	11.32	0.36	0		6.661
729	10-22-99/TOC-B		1	9.356	0	9.356	0.36	0		
729D	10-22-99/TOC-B		1	9.092	0	9.092	0.36	0		2.862

10 % Recalculated Results for Total Organic Carbon in Surface Water

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-5-00 Checked by njs 4-13-00

Sample	QC Batch	Qualifier Note	Dilution	Instrument Result ppm	Blank Factor ppm	Sample Results ppm	Detection Limit ppm
M5-738-SWF	10-22-99/T0C-A		1	6.326	0	6.33	0.12
M5-828-SWF	10-22-99/T0C-A		1	18.67	0	18.67	0.12
M5-944-SWF	10-22-99/T0C-A		1	7.77	0	7.77	0.12

Sample	QC Batch	Qualifier Note	Dilution	Instrument Result ppm	Blank Factor ppm	Sample Results ppm	Detection Limit*3 ppm	Spike Conc (ppm)	R%	RPD
Method Blank	10-22-99/T0C-A		1	1.163	1.1	0.06	0.36	0		
Method Blank	10-22-99/T0C-A		1	1.223	1.1	0.12	0.36	0		
Method Blank	10-22-99/T0C-A		1	1.275	1.1	0.18	0.36	0		
Method Blank	10-22-99/T0C-A		1	1.082	1.1	-0.02	0.36	0		
Method Blank	10-22-99/T0C-A		1	1.037	1.1	-0.06	0.36	0		
Method Blank	10-22-99/T0C-A		1	0.822	1.1	-0.28	0.36	0		
CCV 10	10-22-99/T0C-A		1	11.70	1.1	10.60	0.36	10	106.00	
CCV 5	10-22-99/T0C-A		1	6.771	1.1	5.67	0.36	5	113.42	
CCV 10	10-22-99/T0C-A		1	11.85	1.1	10.75	0.36	10	107.50	
CCV 5	10-22-99/T0C-A		1	6.519	1.1	5.42	0.36	5	108.38	
CCV 10	10-22-99/T0C-A		1	11.5	1.1	10.40	0.36	10	104.00	
UMS	10-22-99/T0C-A		1	8.889	1.1	7.79	0.36			
UMS	10-22-99/T0C-A		1	9.033	1.1	7.93	0.36			-1.832
MS	10-22-99/T0C-A		1	18	1.1	16.90	0.36	8	113.89	
MS	10-22-99/T0C-A		1	17.9	1.1	16.80	0.36	8	110.84	0.593
740	10-22-99/T0C-A		1	5.777	0	5.78	0.36	0		
740D	10-22-99/T0C-A		1	6.133	0	6.13	0.36	0		-5.978
838	10-22-99/T0C-A		1	22.06	0	22.06	0.36	0		
838D	10-22-99/T0C-A		1	21.88	0	21.88	0.36	0		0.819
QA-042-PEF	10-22-99/T0C-A		1	0.82	0	0.82	0.36	0		
QA-042-PEF-DUP	10-22-99/T0C-A		1	0.838	0	0.84	0.36	0		-2.171
QA-003-CB1	10-22-99/T0C-A		1	0.883	0	0.883	0.36	0		
QA-003-CB1-DUP	10-22-99/T0C-A		1	0.881	0	0.881	0.36	0		0.227

SERC Lab / EPA REMAP results

Total Organic Carbon (TOC) Water Analysis by EPA Method 415.1 (modified)

: Analysis not performed

: Analysis not required

: Average of two

Data Entered by: PMEYER 3-16-00

Data Entry Checked by: njs 3-17-00

Sampling Station ID	Matrix	Collection Date	Collection Time	Digestion Date	Run Date	Holding Time (Days)	Time Elapsed From Dig	TOC Units (ppm)	QA/QC Batch ID	QA Data % R	QA Data %RPD	QA Data Matrix %R	QA Data Sample RPD	Notes
M5-622-SWF	SW	09/30/99	1125	10/21/99	10/21/99	28	21	36.77	10-21-99/TOC-B	118	2	109		
M5-623-SWF	SW	09/30/99	915	10/21/99	10/21/99	28	21	22.28	10-21-99/TOC-B	118	2	109		
M5-624-SWF	SW	09/30/99	1018	10/21/99	10/21/99	28	21	27.22	10-21-99/TOC-B	118	2	109		
M5-625-SWF	SW	09/30/99	1257	10/21/99	10/21/99	28	21	28.27	10-21-99/TOC-B	118	2	109		
M5-626-SWF	SW	09/30/99	908	10/21/99	10/21/99	28	21	22.65	10-21-99/TOC-B	118	2	109		
M5-627-SWF	SW	09/30/99	1050	10/21/99	10/21/99	28	21	40.89	10-21-99/TOC-B	118	2	109	-1.27	Averaged Result
M5-628-SWF	SW	09/29/99	1716	10/21/99	10/21/99	28	22	19.60	10-21-99/TOC-B	118	2	109		
M5-631-SWF	SW	09/29/99	1414	10/21/99	10/21/99	28	22	17.69	10-21-99/TOC-B	118	2	109		
M5-632-SWF	SW	09/29/99	1010	10/21/99	10/21/99	28	22	17.99	10-21-99/TOC-B	118	2	109		
M5-633-SWF	SW	09/29/99	1115	10/21/99	10/21/99	28	22	21.02	10-21-99/TOC-B	118	2	109		
M5-635-SWF	SW	09/29/99	1216	10/21/99	10/21/99	28	22	17.36	10-21-99/TOC-B	118	2	109		
M5-637-SWF	SW	09/29/99	1210	10/21/99	10/21/99	28	22	20.24	10-21-99/TOC-B	118	2	109		
M5-638-SWF	SW	09/29/99	1630	10/21/99	10/21/99	28	22	22.53	10-21-99/TOC-B	118	2	109	0.22	Averaged Result
M5-639-SWF	SW	09/28/99	1715	10/21/99	10/21/99	28	23	25.40	10-21-99/TOC-B	118	2	109		
M5-640-SWF	SW	09/29/99	1116	10/21/99	10/21/99	28	22	16.23	10-21-99/TOC-B	118	2	109		
M5-641-SWF	SW	09/28/99	1615	10/21/99	10/21/99	28	23	26.20	10-21-99/TOC-B	118	2	109		
M5-642-SWF	SW	09/29/99	1011	10/21/99	10/21/99	28	22	39.12	10-21-99/TOC-B	118	2	109		
M5-643-SWF	SW	09/29/99	910	10/21/99	10/21/99	28	22	24.94	10-21-99/TOC-B	118	2	109		
M5-644-SWF	SW	09/30/99	1145	10/21/99	10/21/99	28	21	29.06	10-21-99/TOC-B	118	2	109		
M5-646-SWF	SW	09/28/99	1515	10/21/99	10/21/99	28	23	33.42	10-21-99/TOC-B	118	2	109		
M5-647-SWF	SW	09/28/99	1102	10/21/99	10/21/99	28	23	37.49	10-21-99/TOC-B	118	2	109		
M5-648-SWF	SW	09/28/99	1300	10/21/99	10/21/99	28	23	26.86	10-21-99/TOC-B	118	2	109	0.19	Averaged Result
M5-649-SWF	SW	09/28/99	1620	10/21/99	10/21/99	28	23	27.75	10-21-99/TOC-B	118	2	109		
M5-650-SWF	SW	09/28/99	1158	10/21/99	10/21/99	28	23	30.02	10-21-99/TOC-B	118	2	109		
M5-653-SWF	SW	09/28/99	1300	10/21/99	10/21/99	28	23	34.31	10-21-99/TOC-B	118	2	109		
M5-654-SWF	SW	09/28/99	1145	10/21/99	10/21/99	28	23	27.46	10-21-99/TOC-B	118	2	109		
M5-655-SWF	SW	09/28/99	1028	10/21/99	10/21/99	28	23	35.39	10-21-99/TOC-B	118	2	109		
M5-656-SWF	SW	09/28/99	900	10/21/99	10/21/99	28	23	18.45	10-21-99/TOC-B	118	2	109		
M5-657-SWF	SW	09/27/99	1751	10/21/99	10/21/99	28	24	33.66	10-21-99/TOC-B	118	2	109		
M5-658-SWF	SW	09/28/99	1722	10/21/99	10/21/99	28	23	26.38	10-21-99/TOC-B	118	2	109	-1.02	Averaged Result
M5-659-SWF	SW	09/27/99	1205	10/21/99	10/21/99	28	24	26.98	10-21-99/TOC-B	118	2	109		
M5-660-SWF	SW	09/27/99	1450	10/21/99	10/21/99	28	24	29.14	10-21-99/TOC-B	118	2	109		
M5-661-SWF	SW	09/29/99	857	10/21/99	10/21/99	28	22	22.07	10-21-99/TOC-B	118	2	109		
M5-662-SWF	SW	09/27/99	1610	10/21/99	10/21/99	28	24	25.77	10-21-99/TOC-B	118	2	109		
M5-663-SWF	SW	09/27/99	1330	10/21/99	10/21/99	28	24	19.94	10-21-99/TOC-B	118	2	109		
M5-664-SWF	SW	09/27/99	1100	10/21/99	10/21/99	28	24	27.47	10-21-99/TOC-B	118	2	109		
M5-665-SWF	SW	09/27/99	1710	10/21/99	10/21/99	28	24	23.95	10-21-99/TOC-B	118	2	109		
M5-666-SWF	SW	09/27/99	1655	10/21/99	10/21/99	28	24	20.49	10-21-99/TOC-B	118	2	109		
M5-667-SWF	SW	09/27/99	1545	10/21/99	10/21/99	28	24	18.52	10-21-99/TOC-B	118	2	109		

M5-668-SWF	SW	09/27/99	1207	10/21/99	10/21/99	28	24	17.15	10-21-99/TOC-B	118	2	109	0.47	Averaged Result
M5-669-SWF	SW	09/27/99	1000	10/21/99	10/21/99	28	24	26.09	10-21-99/TOC-B	118	2	109		
M5-670-SWF	SW	09/27/99	850	10/21/99	10/21/99	28	24	20.14	10-21-99/TOC-B	118	2	109		
M5-672-SWF	SW	09/27/99	1100	10/21/99	10/21/99	28	24	15.43	10-21-99/TOC-B	118	2	109		
M5-673-SWF	SW	09/27/99	1400	10/21/99	10/21/99	28	24	18.9	10-21-99/TOC-B	118	2	109		
M5-674-SWF	SW	09/26/99	1025	10/21/99	10/21/99	28	25	17.46	10-21-99/TOC-B	118	2	109		
M5-675-SWF	SW	09/26/99	1130	10/21/99	10/21/99	28	25	22.51	10-21-99/TOC-B	118	2	109		
M5-676-SWF	SW	09/28/99	925	10/21/99	10/21/99	28	23	18.05	10-21-99/TOC-B	118	2	109		
M5-677-SWF	SW	09/26/99	1310	10/22/99	10/22/99	28	26	18.51	10-22-99/TOC-B	102	2	99		
M5-678-SWF	SW	09/26/99	1213	10/22/99	10/22/99	28	26	13.81	10-22-99/TOC-B	102	2	99		
M5-679-SWF	SW	09/26/99	1335	10/22/99	10/22/99	28	26	16.7	10-22-99/TOC-B	102	2	99		
M5-680-SWF	SW	09/27/99	900	10/22/99	10/22/99	28	25	15.68	10-22-99/TOC-B	102	2	99		
M5-681-SWF	SW	09/26/99	1410	10/22/99	10/22/99	28	26	18.45	10-22-99/TOC-B	102	2	99		
M5-683-SWF	SW	09/26/99	850	10/22/99	10/22/99	28	26	11.55	10-22-99/TOC-B	102	2	99		
M5-684-SWF	SW	09/26/99	1530	10/22/99	10/22/99	28	26	18.08	10-22-99/TOC-B	102	2	99		
M5-685-SWF	SW	09/26/99	1434	10/22/99	10/22/99	28	26	12.59	10-22-99/TOC-B	102	2	99		
M5-686-SWF	SW	09/25/99	915	10/22/99	10/22/99	28	27	18.68	10-22-99/TOC-B	102	2	99	-1.07	Averaged Result
M5-687-SWF	SW	09/25/99	1035	10/22/99	10/22/99	28	27	18.74	10-22-99/TOC-B	102	2	99		
M5-688-SWF	SW	09/26/99	1527	10/22/99	10/22/99	28	26	11.32	10-22-99/TOC-B	102	2	99		
M5-689-SWF	SW	09/25/99	1205	10/22/99	10/22/99	28	27	17.71	10-22-99/TOC-B	102	2	99		
M5-690-SWF	SW	09/25/99	1350	10/22/99	10/22/99	28	27	13.11	10-22-99/TOC-B	102	2	99		
M5-691-SWF	SW	09/25/99	1554	10/22/99	10/22/99	28	27	11.67	10-22-99/TOC-B	102	2	99		
M5-692-SWF	SW	09/25/99	1200	10/22/99	10/22/99	28	27	12.17	10-22-99/TOC-B	102	2	99		
M5-693-SWF	SW	09/25/99	1139	10/22/99	10/22/99	28	27	10.22	10-22-99/TOC-B	102	2	99		
M5-694-SWF	SW	09/25/99	1702	10/22/99	10/22/99	28	27	12.39	10-22-99/TOC-B	102	2	99		
M5-695-SWF	SW	09/25/99	1400	10/22/99	10/22/99	28	27	16.21	10-22-99/TOC-B	102	2	99		
M5-697-SWF	SW	09/26/99	1637	10/22/99	10/22/99	28	26	18.89	10-22-99/TOC-B	102	2	99		
M5-699-SWF	SW	09/25/99	1635	10/22/99	10/22/99	28	27	16.88	10-22-99/TOC-B	102	2	99		
M5-700-SWF	SW	09/25/99	1750	10/22/99	10/22/99	28	27	12.27	10-22-99/TOC-B	102	2	99		
M5-701-SWF	SW	09/26/99	1740	10/22/99	10/22/99	28	26	12.46	10-22-99/TOC-B	102	2	99		
M5-702-SWF	SW	09/25/99	918	10/22/99	10/22/99	28	27	16.9	10-22-99/TOC-B	102	2	99		
M5-703-SWF	SW	09/24/99	1655	10/22/99	10/22/99	28	28	14.16	10-22-99/TOC-B	102	2	99		
M5-704-SWF	SW	09/24/99	1615	10/22/99	10/22/99	28	28	9.518	10-22-99/TOC-B	102	2	99		
M5-705-SWF	SW	09/24/99	1725	10/22/99	10/22/99	28	28	10.09	10-22-99/TOC-B	102	2	99		
M5-706-SWF	SW	09/24/99	1555	10/22/99	10/22/99	28	28	10.45	10-22-99/TOC-B	102	2	99	3.73	Averaged Result
M5-707-SWF	SW	09/24/99	1500	10/22/99	10/22/99	28	28	11	10-22-99/TOC-B	102	2	99		
M5-708-SWF	SW	09/24/99	900	10/22/99	10/22/99	28	28	14.46	10-22-99/TOC-B	102	2	99		
M5-709-SWF	SW	09/24/99	1330	10/22/99	10/22/99	28	28	6.433	10-22-99/TOC-B	102	2	99		
M5-711-SWF	SW	09/24/99	1115	10/22/99	10/22/99	28	28	10.66	10-22-99/TOC-B	102	2	99		
M5-712-SWF	SW	09/24/99	905	10/22/99	10/22/99	28	28	11.34	10-22-99/TOC-B	102	2	99		
M5-714-SWF	SW	09/23/99	1715	10/22/99	10/22/99	28	29	13.99	10-22-99/TOC-B	102	2	99		
M5-715-SWF	SW	09/24/99	1145	10/22/99	10/22/99	28	28	8.607	10-22-99/TOC-B	102	2	99		
M5-716-SWF	SW	09/24/99	1310	10/22/99	10/22/99	28	28	14.86	10-22-99/TOC-B	102	2	99		
M5-718-SWF	SW	09/24/99	1030	10/22/99	10/22/99	28	28	11.71	10-22-99/TOC-B	102	2	99	6.66	Averaged Result
M5-720-SWF	SW	09/23/99	0	10/22/99	10/22/99	28	29	15.61	10-22-99/TOC-B	102	2	99		

M5-722-SWF	SW	09/23/99	1600	10/22/99	10/22/99	28	29	10.69	10-22-99/TOC-B	102	2	99		
M5-723-SWF	SW	09/23/99	1500	10/22/99	10/22/99	28	29	8.841	10-22-99/TOC-B	102	2	99		
M5-724-SWF	SW	09/23/99	1442	10/22/99	10/22/99	28	29	14.08	10-22-99/TOC-B	102	2	99		
M5-725-SWF	SW	09/23/99	1323	10/22/99	10/22/99	28	29	10.02	10-22-99/TOC-B	102	2	99		
M5-726-SWF	SW	09/23/99	1230	10/22/99	10/22/99	28	29	11.5	10-22-99/TOC-B	102	2	99		
M5-727-SWF	SW	09/23/99	1216	10/22/99	10/22/99	28	29	9.453	10-22-99/TOC-B	102	2	99		
M5-728-SWF	SW	09/23/99	1350	10/22/99	10/22/99	28	29	11	10-22-99/TOC-B	102	2	99		
M5-729-SWF	SW	09/23/99	1027	10/22/99	10/22/99	28	29	9.22	10-22-99/TOC-B	102	2	99	2.86	Averaged Result
M5-730-SWF	SW	09/23/99	1120	10/22/99	10/22/99	28	29	11.77	10-22-99/TOC-B	102	2	99		
M5-731-SWF	SW	09/22/99	1725	10/22/99	10/22/99	28	30	11.64	10-22-99/TOC-B	102	2	99		
M5-732-SWF	SW	09/23/99	917	10/22/99	10/22/99	28	29	8.877	10-22-99/TOC-B	102	2	99		
M5-733-SWF	SW	09/23/99	910	10/22/99	10/22/99	28	29	8.521	10-22-99/TOC-B	102	2	99		
M5-734-SWF	SW	09/22/99	1540	10/22/99	10/22/99	28	30	10.67	10-22-99/T0C-A	111	1	112		
M5-735-SWF	SW	09/22/99	1700	10/22/99	10/22/99	28	30	9.589	10-22-99/T0C-A	111	1	112		
M5-738-SWF	SW	09/22/99	1410	10/22/99	10/22/99	28	30	6.326	10-22-99/T0C-A	111	1	112		
M5-740-SWF	SW	09/22/99	1245	10/22/99	10/22/99	28	30	5.96	10-22-99/T0C-A	111	1	112	-5.97	Averaged Result
M5-741-SWF	SW	09/22/99	1534	10/22/99	10/22/99	28	30	8.7	10-22-99/T0C-A	111	1	112		
M5-742-SWF	SW	09/22/99	1418	10/22/99	10/22/99	28	30	8.733	10-22-99/T0C-A	111	1	112		
M5-743-SWF	SW	09/22/99	1130	10/22/99	10/22/99	28	30	5.541	10-22-99/T0C-A	111	1	112		
M5-745-SWF	SW	09/22/99	1120	10/22/99	10/22/99	28	30	9.547	10-22-99/T0C-A	111	1	112		
M5-746-SWF	SW	09/22/99	948	10/22/99	10/22/99	28	30	8.579	10-22-99/T0C-A	111	1	112		
M5-747-SWF	SW	09/22/99	942	10/22/99	10/22/99	28	30	11.02	10-22-99/T0C-A	111	1	112		
M5-823-SWF	SW	09/30/99		10/22/99	10/22/99	28	22	20.76	10-22-99/T0C-A	111	1	112		
M5-828-SWF	SW	09/29/99		10/22/99	10/22/99	28	23	18.67	10-22-99/T0C-A	111	1	112		
M5-838-SWF	SW	09/29/99		10/22/99	10/22/99	28	23	21.97	10-22-99/T0C-A	111	1	112	0.82	Averaged Result
M5-848-SWF	SW	09/28/99		10/22/99	10/22/99	28	24	26.26	10-22-99/T0C-A	111	1	112		
M5-859-SWF	SW	09/27/99		10/22/99	10/22/99	28	25	25.24	10-22-99/T0C-A	111	1	112		
M5-868-SWF	SW	09/27/99		10/22/99	10/22/99	28	25	15.95	10-22-99/T0C-A	111	1	112		
M5-878-SWF	SW	09/26/99		10/22/99	10/22/99	28	26	14.76	10-22-99/T0C-A	111	1	112		
M5-890-SWF	SW	09/25/99		10/22/99	10/22/99	28	27	13.02	10-22-99/T0C-A	111	1	112		
M5-908-SWF	SW	09/24/99		10/22/99	10/22/99	28	28	15.23	10-22-99/T0C-A	111	1	112		
M5-920-SWF	SW	09/23/99		10/22/99	10/22/99	28	29	15.89	10-22-99/T0C-A	111	1	112		
M5-932-SWF	SW	09/23/99		10/22/99	10/22/99	28	29	8.945	10-22-99/T0C-A	111	1	112		
M5-944-SWF	SW	09/22/99	1224	10/22/99	10/22/99	28	30	7.77	10-22-99/T0C-A	111	1	112		
QA-001-CB1	WATER	09/22/99		10/22/99	10/22/99	28	30	0.75	10-22-99/T0C-A	111	1	112		
QA-001-CB2	WATER	09/22/99		10/22/99	10/22/99	28	30	0.765	10-22-99/T0C-A	111	1	112		
QA-002-CB1	WATER	09/22/99		10/22/99	10/22/99	28	30	0.713	10-22-99/T0C-A	111	1	112		
QA-002-CB2	WATER	09/22/99		10/23/99	10/23/99	28	31	0.92	10-23-99/TOC-A	108	1	101	24.60	Averaged Result
QA-003-CB1	WATER	09/22/99		10/22/99	10/22/99	28	30	0.882	10-22-99/T0C-A	111	1	112	0.23	Averaged Result
QA-003-CB2	WATER	09/22/99		10/22/99	10/22/99	28	30	0.753	10-22-99/T0C-A	111	1	112		
QA-004-CB1	WATER	09/22/99		10/22/99	10/22/99	28	30	0.814	10-22-99/T0C-A	111	1	112		
QA-004-CB2	WATER	09/22/99		10/22/99	10/22/99	28	30	0.758	10-22-99/T0C-A	111	1	112		
QA-005-CB1	WATER	09/22/99		10/22/99	10/22/99	28	30	0.91	10-22-99/T0C-A	111	1	112		
QA-005-CB2	WATER	09/22/99		10/22/99	10/22/99	28	30	0.787	10-22-99/T0C-A	111	1	112		
QA-006-CB1	WATER	09/22/99		10/22/99	10/22/99	28	30	0.755	10-22-99/T0C-A	111	1	112		

QA-006-CB2	WATER	09/22/99		10/22/99	10/22/99	28	30	0.786	10-22-99/T0C-A	111	1	112		
QA-007-CB1	WATER	09/22/99		10/22/99	10/22/99	28	30	0.742	10-22-99/T0C-A	111	1	112		
QA-007-CB2	WATER	09/22/99		10/22/99	10/22/99	28	30	0.739	10-22-99/T0C-A	111	1	112		
QA-008-CB1	WATER	09/22/99		10/22/99	10/22/99	28	30	0.764	10-22-99/T0C-A	111	1	112		
QA-008-CB2	WATER	09/22/99		10/22/99	10/22/99	28	30	0.815	10-22-99/T0C-A	111	1	112		
QA-009-CB1	WATER	09/22/99		10/22/99	10/22/99	28	30	0.941	10-22-99/T0C-A	111	1	112		
QA-009-CB2	WATER	09/22/99		10/22/99	10/22/99	28	30	0.75	10-22-99/T0C-A	111	1	112		
QA-027-PEF	PE	09/22/99		10/21/99	10/21/99	28	29	46.52	10-21-99/TOC-B	118	2	109		
QA-031-PEF	WATER	09/22/99		10/22/99	10/22/99	28	30	0.797	10-22-99/T0C-A	111	1	112		
QA-032-PEF	PE	09/22/99		10/21/99	10/21/99	28	29	12.6	10-21-99/TOC-B	118	2	109		
QA-042-PEF	WATER	09/22/99		10/22/99	10/22/99	28	30	0.83	10-22-99/T0C-A	111	1	112	-2.17	Averaged Result
QA-043-PEF	PE	09/22/99		10/21/99	10/21/99	28	29	6.78	10-21-99/TOC-B	118	2	109		
QA-044-PEF	PE	09/22/99		10/21/99	10/21/99	28	29	2.204	10-21-99/TOC-B	118	2	109		
QA-630-SWF	SW	09/22/99		10/21/99	10/21/99	28	29	21.20	10-21-99/TOC-B	118	2	109		
QA-634-SWF	SW	09/22/99		10/21/99	10/21/99	28	29	29.86	10-21-99/TOC-B	118	2	109		
QA-636-SWF	SW	09/22/99		10/21/99	10/21/99	28	29	38.62	10-21-99/TOC-B	118	2	109		
QA-645-SWF	SW	09/22/99		10/21/99	10/21/99	28	29	42.34	10-21-99/TOC-B	118	2	109		
QA-651-SWF	SW	09/22/99		10/21/99	10/21/99	28	29	30.65	10-21-99/TOC-B	118	2	109		
QA-652-SWF	SW	09/22/99		10/21/99	10/21/99	28	29	21.46	10-21-99/TOC-B	118	2	109		
QA-671-SWF	SW	09/22/99		10/21/99	10/21/99	28	29	19.43	10-21-99/TOC-B	118	2	109		
QA-682-SWF	SW	09/22/99		10/22/99	10/22/99	28	30	14.36	10-22-99/TOC-B	102	2	99		
QA-696-SWF	SW	09/22/99		10/22/99	10/22/99	28	30	17.05	10-22-99/TOC-B	102	2	99	0.41	Averaged Result
QA-698-SWF	SW	09/22/99		10/22/99	10/22/99	28	30	10.91	10-22-99/TOC-B	102	2	99		
QA-710-SWF	SW	09/22/99		10/22/99	10/22/99	28	30	14.72	10-22-99/TOC-B	102	2	99		
QA-719-SWF	SW	09/22/99		10/22/99	10/22/99	28	30	13.83	10-22-99/TOC-B	102	2	99		
QA-744-SWF	SW	09/22/99		10/22/99	10/22/99	28	30	7.704	10-22-99/T0C-A	111	1	112		

10 % Recalculated Results for Total Mercury in Surface Water

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-6-00 Checked by njs 4-13-00

Sample	QC Batch	Qualifier Note	Dilution	Instrument Reading Peak Height Rep 1,2,3	Method Reagant Blank Peak Hieght	Corrected Reading Peak Height Rep 1,2,3	Y ratio	Hg Concentration ppt	Averaged Result ppt	DETECTION LIMIT/*3 ppt	SPK CONC (ppt)	R%	Standard Deviation	Relative Standard Deviation
M5-622-SWF-1	HG19JF1		1	106.3	0	106.30	15.2	7.19		0.3/0.9				
	"		1	109.9	0	109.90	15.2	7.44		0.3/0.9				
	"		1	111.7	0	111.70	15.2	7.56		0.3/0.9				
M5-622-SWF-2	"		1	114.6	0	114.60	15.2	7.76		0.3/0.9				
	"		1	112.7	0	112.70	15.2	7.63		0.3/0.9				
	"		1	112.3	0	112.30	15.2	7.60		0.3/0.9				
M5-622-SWF-3	"		1	108.4	0	108.40	15.2	7.34		0.3/0.9				
	"		1	107.9	0	107.90	15.2	7.30		0.3/0.9				
	"		1	107.9	0	107.90	15.2	7.30	7.46	0.3/0.9			0.19	2.5
M5-622-SWF-1-S	"		1	130.4	0	130.40	15.2	8.83		0.3/0.9	2			
	"		1	133.0	0	133.00	15.2	9.00		0.3/0.9	2			
	"		1	129.8	0	129.80	15.2	8.79	8.87	0.3/0.9	2	70.65	0.12	1.3
Instrument Blank	"		1	0.65		0.65	15.2	0.04		0.3/0.9				
CCV-1	"		1	78.3	0	78.30	15.2	5.30		0.3/0.9	5	105.99		
CCV-2	"		1	77.9	0	77.90	15.2	5.27		0.3/0.9	5	105.45		
CCV-3	"		1	81.5	0	81.50	15.2	5.52		0.3/0.9	5	110.33		
CCV-4	"		1	80.4	0	80.40	15.2	5.44		0.3/0.9	5	108.84		
CCV-5	"		1	72.2	0	72.20	15.2	4.89		0.3/0.9	5	97.74		
CCV-6	"		1	76.9	0	76.90	15.2	5.20		0.3/0.9	5	104.10		
CCV-7	"		1	81.1	0	81.10	15.2	5.49		0.3/0.9	5	109.78		
CCV-8	"		1	77.9	0	77.90	15.2	5.27	5.30	0.3/0.9	5	105.45	0.20	3.8

10 % Recalculated Results for Total Mercury in Surface Water

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-6-00 Checked by njs 4-13-00

Sample	QC Batch	Qualifier Note	Dilution	Instrument Reading Peak Height Rep 1,2,3	Method Reagant Blank Peak Hieght	Corrected Reading Peak Height Rep 1,2,3	Y ratio	Hg Concentration ppt	Averaged Result ppt	DETECTION LIMIT/*3 ppt	SPK CONC (ppm)	R%	Standard Deviation	Relative Standard Deviation
M5-633-SWF-1	HG13JF1		1	52.4	4.01	48.39	17.8	2.80		0.3/0.9				
	"		1	51.0	4.01	46.99	17.8	2.72		0.3/0.9				
	"		1	49.7	4.01	45.69	17.8	2.64		0.3/0.9				
M5-633-SWF-2	"		1	48.7	4.01	44.69	17.8	2.58		0.3/0.9				
	"		1	51.4	4.01	47.39	17.8	2.74		0.3/0.9				
	"		1	48.0	4.01	43.99	17.8	2.54		0.3/0.9				
M5-633-SWF-3	"		1	43.7	4.01	39.69	17.8	2.29		0.3/0.9				
	"		1	46.8	4.01	42.79	17.8	2.47		0.3/0.9				
	"		1	41.2	4.01	37.19	17.8	2.15	2.55	0.3/0.9			0.21	8.4
M5-633-SWF-1-S	"		1	59.3	4.01	55.29	17.8	3.20		0.3/0.9	1			
	"		1	61.3	4.01	57.29	17.8	3.31		0.3/0.9	1			
	"		1	60.9	4.01	56.89	17.8	3.29	3.26	0.3/0.9	1	71.67	0.06	1.9
Instrument Blank	"		1	1.55		1.55	17.8	0.09		0.3/0.9				
CCV-1	"		1	90.8	4.01	86.79	17.8	5.02		0.3/0.9	5	100.33		
CCV-2	"		1	90.8	4.01	86.79	17.8	5.02		0.3/0.9	5	100.33		
CCV-3	"		1	87.4	4.01	83.39	17.8	4.82		0.3/0.9	5	96.40		
CCV-4	"		1	88.0	4.01	83.99	17.8	4.85		0.3/0.9	5	97.09		
CCV-5	"		1	81.9	4.01	77.89	17.8	4.50		0.3/0.9	5	90.04		
CCV-6	"		1	86.4	4.01	82.39	17.8	4.76		0.3/0.9	5	95.24		
CCV-7	"		1	81.7	4.01	77.69	17.8	4.49		0.3/0.9	5	89.81		
CCV-8	"		1	85.0	4.01	80.99	17.8	4.68		0.3/0.9	5	93.62		
M5-944-SWF-1	HG13JF1		1	32.6	4.01	28.59	15.1	1.95		0.3/0.9				
	"		1	27.7	4.01	23.69	15.1	1.61		0.3/0.9				
	"		1	19.7	4.01	15.69	15.1			0.3/0.9				
M5-944-SWF-2	"		1	25.1	4.01	21.09	15.1	1.44		0.3/0.9				
	"		1	29.3	4.01	25.29	15.1	1.72		0.3/0.9				
	"		1	26.1	4.01	22.09	15.1	1.51		0.3/0.9				
M5-944-SWF-3	"		1	29.1	4.01	25.09	15.1	1.71		0.3/0.9				
	"		1	30.7	4.01	26.69	15.1	1.82		0.3/0.9				
	"		1	30.3	4.01	26.29	15.1	1.79	1.69	0.3/0.9			0.17	9.9
Instrument Blank	"		1	2.95		2.95	15.1	0.20		0.3/0.9				
CCV-1	"		1	85.0	4.01	80.99	15.1	5.52		0.3/0.9	5	110.36		
CCV-2	"		1	86.1	4.01	82.09	15.1	5.59		0.3/0.9	5	111.86		
CCV-3	"		1	76.8	4.01	72.79	15.1	4.96		0.3/0.9	5	99.19		
CCV-4	"		1	76.3	4.01	72.29	15.1	4.93		0.3/0.9	5	98.51		
CCV-5	"		1	82.1	4.01	78.09	15.1	5.32		0.3/0.9	5	106.41		
CCV-6	"		1	84.4	4.01	80.39	15.1	5.48		0.3/0.9	5	109.54		
CCV-7	"		1	79.4	4.01	75.39	15.1	5.14		0.3/0.9	5	102.73		
CCV-8	"		1	80.9	4.01	76.89	15.1	5.24		0.3/0.9	5	104.77		
CCV-9	"		1	77.7	4.01	73.69	15.1	5.02		0.3/0.9	5	100.41		
CCV-10	"		1	76.6	4.01	72.59	15.1	4.95		0.3/0.9	5	98.91		
CCV-11	"		1	84.8	4.01	80.79	15.1	5.50		0.3/0.9	5	110.09		
CCV-12	"		1	87.0	4.01	82.99	15.1	5.65	5.27	0.3/0.9	5	113.09	0.27	5.2

10 % Recalculated Results for Total Mercury in Surface Water

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-6-00 Checked by njs 4-13-00

Sample	QC Batch	Qualifier Note	Dilution	Instrument Reading Peak Height Rep 1,2,3	Method Reagant Blank Peak Hieght	Corrected Reading Peak Height Rep 1,2,3	Y ratio	Hg Concentration ppt	Averaged Result ppt	DETECTION LIMIT/*3 ppt	SPK CONC (ppm)	R%	Standard Deviation	Relative Standard Deviation
M5-643-SWF-1	HG07JF1		1	18.4	2.78	15.62	12.0	1.34		0.3/0.9				
	"		1	16.4	2.78	13.62	12.0	1.17		0.3/0.9				
	"		1	13.6	2.78	10.82	12.0	0.93		0.3/0.9				
M5-643-SWF-2	"		1	14.1	2.78	11.32	12.0	0.97		0.3/0.9				
	"		1	15.7	2.78	12.92	12.0	1.11		0.3/0.9				
	"		1	16.8	2.78	14.02	12.0	1.20		0.3/0.9				
M5-643-SWF-3	"		1	19.3	2.78	16.52	12.0	1.42		0.3/0.9				
	"		1	18.4	2.78	15.62	12.0	1.34		0.3/0.9				
	"		1	15.9	2.78	13.12	12.0	1.12	1.18	0.3/0.9			0.17	14.2
M5-677-SWF-1	"		1	15.5	2.78	12.72	14.6	0.90		0.3/0.9				
	"		1	20.2	2.78	17.42	14.6	1.23		0.3/0.9				
	"		1	14.3	2.78	11.52	14.6	0.81		0.3/0.9				
M5-677-SWF-2	"		1	11.7	2.78	8.92	14.6	0.63		0.3/0.9				
	"		1	16.9	2.78	14.12	14.6	0.99		0.3/0.9				
	"		1	14.9	2.78	12.12	14.6	0.85		0.3/0.9				
M5-677-SWF-3	"		1	15.8	2.78	13.02	14.6	0.92		0.3/0.9				
	"		1	19.5	2.78	16.72	14.6	1.18		0.3/0.9				
	"		1	17.0	2.78	14.22	14.6	1.00	0.95	0.3/0.9			0.18	19.4
QA-677-SWF-1-S	"		1	42.6	2.78	39.82	14.6	2.81		0.3/0.9	2			
	"		1	40.8	2.78	38.02	14.6	2.68		0.3/0.9	2			
	"		1	45.2	2.78	42.42	14.6	2.99	2.82	0.3/0.9	2	93.95	0.16	5.5
Instrument Blank	"		1	4.85		4.85	12.0	0.42		0.3/0.9				
CCV-1	"		1	60.3	2.78	57.52	12.0	4.93		0.3/0.9	5	98.63		
CCV-2	"		1	61.2	2.78	58.42	12.0	5.01		0.3/0.9	5	100.17		
CCV-3	"		1	61.9	2.78	59.12	12.0	5.07		0.3/0.9	5	101.37		
CCV-4	"		1	61.3	2.78	58.52	12.0	5.02		0.3/0.9	5	100.34		
CCV-5	"		1	62.5	2.78	59.72	12.0	5.12		0.3/0.9	5	102.40		
CCV-6	"		1	60.6	2.78	57.82	12.0	4.96		0.3/0.9	5	99.14		
CCV-7	"		1	60.9	2.78	58.12	12.0	4.98		0.3/0.9	5	99.66		
CCV-8	"		1	57.7	2.78	54.92	12.0	4.71		0.3/0.9	5	94.17		
CCV-9	"		1	55.8	2.78	53.02	12.0	4.55		0.3/0.9	5	90.91		
CCV-10	"		1	60.0	2.78	57.22	12.0	4.91		0.3/0.9	5	98.11		
CCV-11	"		1	65.2	2.78	62.42	12.0	5.35		0.3/0.9	5	107.03		
CCV-12	"		1	60.6	2.78	57.82	12.0	4.96	4.96	0.3/0.9	5	99.14	0.20	4.0

10 % Recalculated Results for Total Mercury in Surface Water

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-6-00 Checked by njs 4-13-00

Sample	QC Batch	Qualifier Note	Dilution	Instrument Reading Peak Height Rep 1,2,3	Method Reagant Blank Peak Hieght	Corrected Reading Peak Height Rep 1,2,3	Y ratio	Hg Concentration ppt	Averaged Result ppt	DETECTION LIMIT/*3 ppt	SPK CONC (ppm)	R%	Standard Deviation	Relative Standard Deviation
M5-653-SWF-1	HG15JF1		1	9.5	0.08	9.42	16.1	0.60		0.3/0.9				
	"		1	11.9	0.08	11.82	16.1	0.76		0.3/0.9				
	"		1	10.6	0.08	10.52	16.1	0.67		0.3/0.9				
M5-653-SWF-2	"		1	10.2	0.08	10.12	16.1	0.65		0.3/0.9				
	"		1	11.4	0.08	11.32	16.1	0.72		0.3/0.9				
	"		1	14.1	0.08	14.02	16.1			0.3/0.9				
M5-653-SWF-3	"		1	9.6	0.08	9.52	16.1	0.61		0.3/0.9				
	"		1	9.5	0.08	9.42	16.1	0.60		0.3/0.9				
	"		1	11.9	0.08	11.82	16.1	0.76	0.67	0.3/0.9			0.07	9.9
M5-632-SWF-1	"		1	15.2	0.08	15.12	16.1	0.97		0.3/0.9				
	"		1	13.7	0.08	13.62	16.1	0.87		0.3/0.9				
	"		1	14.5	0.08	14.42	16.1	0.92		0.3/0.9				
M5-632-SWF-2	"		1	10.2	0.08	10.12	16.1	0.65		0.3/0.9				
	"		1	15.4	0.08	15.32	16.1	0.98		0.3/0.9				
	"		1	6.5	0.08	6.42	16.1			0.3/0.9				
M5-632-SWF-3	"		1	11.7	0.08	11.62	16.1	0.74		0.3/0.9				
	"		1	12.2	0.08	12.12	16.1	0.77		0.3/0.9				
	"		1	12.7	0.08	12.62	16.1	0.81	0.84	0.3/0.9			0.12	13.9
QA-632-SWF-1-S	"	"M"	1	53.7	0.08	53.62	16.1	3.43		0.3/0.9	2			
	"	"M"	1	54.4	0.08	54.32	16.1	3.47		0.3/0.9	2			
	"	"M"	1	55.6	0.08	55.52	16.1	3.55	3.48	0.3/0.9	2	132.17	0.06	1.8
Instrument Blank	"		1	0.55		0.55	16.1	0.04		0.3/0.9				
CCV-1	"		1	81.1	0.08	81.02	16.1	5.18		0.3/0.9	5	103.54		
CCV-2	"		1	83.6	0.08	83.52	16.1	5.34		0.3/0.9	5	106.74		
CCV-3	"		1	80.5	0.08	80.42	16.1	5.14		0.3/0.9	5	102.78		
CCV-4	"		1	81.3	0.08	81.22	16.1	5.19		0.3/0.9	5	103.80		
CCV-5	"		1	84.6	0.08	84.52	16.1	5.40		0.3/0.9	5	108.02		
CCV-6	"		1	81.8	0.08	81.72	16.1	5.22		0.3/0.9	5	104.44		
CCV-7	"		1	87.1	0.08	87.02	16.1	5.56		0.3/0.9	5	111.21		
CCV-8	"		1	85.8	0.08	85.72	16.1	5.48		0.3/0.9	5	109.55		
CCV-9	"		1	87.6	0.08	87.52	16.1	5.59		0.3/0.9	5	111.85		
CCV-10	"		1	84.1	0.08	84.02	16.1	5.37		0.3/0.9	5	107.38		
CCV-11	"		1	81.4	0.08	81.32	16.1	5.20		0.3/0.9	5	103.93		
CCV-12	"		1	84.8	0.08	84.72	16.1	5.41		0.3/0.9	5	108.27		
CCV-13	"		1	81.7	0.08	81.62	16.1	5.22		0.3/0.9	5	104.31		
CCV-14	"		1	80.7	0.08	80.62	16.1	5.15		0.3/0.9	5	103.03		
CCV-15	"		1	84.1	0.08	84.02	16.1	5.37		0.3/0.9	5	107.38		
CCV-16	"		1	74.4	0.08	74.32	16.1	4.75		0.3/0.9	5	94.98		
CCV-17	"		1	80.5	0.08	80.42	16.1	5.14		0.3/0.9	5	102.78		
CCV-18	"		1	80.4	0.08	80.32	16.1	5.13		0.3/0.9	5	102.65		
CCV-19	"		1	83.3	0.08	83.22	16.1	5.32		0.3/0.9	5	106.36		
CCV-20	"		1	78.5	0.08	78.42	16.1	5.01		0.3/0.9	5	100.22		
CCV-21	"		1	77.5	0.08	77.42	16.1	4.95		0.3/0.9	5	98.94		
CCV-22	"		1	79.3	0.08	79.22	16.1	5.06	5.24	0.3/0.9	5	101.24	0.20	3.8

10 % Recalculated Results for Total Mercury in Surface Water

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-6-00 Checked by njs 4-13-00

Sample	QC Batch	Qualifier Note	Dilution	Instrument Reading Peak Height Rep 1,2,3	Method Reagant Blank Peak Hieght	Corrected Reading Peak Height Rep 1,2,3	Y ratio	Hg Concentration ppt	Averaged Result ppt	DETECTION LIMIT/*3 ppt	SPK CONC (ppm)	R%	Standard Deviation	Relative Standard Deviation
M5-663-SWF-1	HG30F1		1	29.30	5.29	24.01	16.6	1.49		0.3/0.9				
	"		1	30.40	5.29	25.11	16.6	1.56		0.3/0.9				
	"		1	29.40	5.29	24.11	16.6	1.49		0.3/0.9				
M5-663-SWF-2	"		1	30.30	5.29	25.01	16.6	1.55		0.3/0.9				
	"		1	30.10	5.29	24.81	16.6	1.54		0.3/0.9				
	"		1	28.00	5.29	22.71	16.6	1.41		0.3/0.9				
M5-663-SWF-3	"		1	33.70	5.29	28.41	16.6			0.3/0.9				
	"		1	30.40	5.29	25.11	16.6	1.56		0.3/0.9				
	"		1	29.50	5.29	24.21	16.6	1.50	1.51	0.3/0.9			0.05	3.4
M5-663-SWF-1-S	"		1	50.60	5.29	45.31	16.6	2.81		0.3/0.9	2			
	"		1	58.30	5.29	53.01	16.6	3.29		0.3/0.9	2			
	"		1	55.30	5.29	50.01	16.6	3.10	3.06	0.3/0.9	2	77.65	0.24	7.85
Instrument Blank	"		1	2.53		2.53	16.6	0.16		0.3/0.9				
CCV-1	"		1	80.30	5.29	75.01	16.6	4.65		0.3/0.9	5	92.98		
CCV-2	"		1	84.00	5.29	78.71	16.6	4.88		0.3/0.9	5	97.56		
CCV-3	"		1	95.20	5.29	89.91	16.6	5.57		0.3/0.9	5	111.45		
CCV-4	"		1	86.00	5.29	80.71	16.6	5.00		0.3/0.9	5	100.04		
CCV-5	"		1	70.60	5.29	65.31	16.6	4.05		0.3/0.9	5	80.95		
CCV-6	"		1	71.60	5.29	66.31	16.6	4.11		0.3/0.9	5	82.19		
CCV-7	"		1	74.90	5.29	69.61	16.6	4.31		0.3/0.9	5	86.28		
CCV-8	"		1	70.60	5.29	65.31	16.6	4.05		0.3/0.9	5	80.95		
CCV-9	"		1	72.10	5.29	66.81	16.6	4.14		0.3/0.9	5	82.81		
CCV-10	"		1	68.30	5.29	63.01	16.6	3.91		0.3/0.9	5	0.00		
CCV-11	"		1	76.00	5.29	70.71	16.6	4.38		0.3/0.9	5	87.65		
CCV-12	"		1	70.30	5.29	65.01	16.6	4.03	4.36	0.3/0.9	5	87.10	0.53	12.1

10 % Recalculated Results for Total Mercury in Surface Water

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-6-00 Checked by njs 4-13-00

Sample	QC Batch	Qualifier Note	Dilution	Instrument Reading Peak Height Rep 1,2,3	Method Reagant Blank Peak Hieght	Corrected Reading Peak Height Rep 1,2,3	Y ratio	Hg Concentration ppt	Averaged Result ppt	DETECTION LIMIT/*3 ppt	SPK CONC (ppm)	R%	Standard Deviation	Relative Standard Deviation
M5-673-SWF-1	HG29IF1		1	26.5	8.25	18.25	12.8	1.47		0.3/0.9				
	"		1	28.6	8.25	20.35	12.8	1.64		0.3/0.9				
	"		1	28.5	8.25	20.25	12.8	1.63		0.3/0.9				
M5-673-SWF-2	"		1	26.1	8.25	17.85	12.8	1.43		0.3/0.9				
	"		1	30.6	8.25	22.35	12.8	1.80		0.3/0.9				
	"		1	30.8	8.25	22.55	12.8	1.81		0.3/0.9				
M5-673-SWF-3	"		1	23.6	8.25	15.35	12.8	1.23		0.3/0.9				
	"		1	31.6	8.25	23.35	12.8	1.88		0.3/0.9				
	"		1	27.4	8.25	19.15	12.8	1.54	1.60	0.3/0.9			0.21	13.0
M5-683-SWF-1	"		1	21.60	8.25	13.35	12.8	1.07		0.3/0.9				
	"		1	15.80	8.25	7.55	12.8	0.61		0.3/0.9				
	"		1	19.00	8.25	10.75	12.8	0.86		0.3/0.9				
M5-683-SWF-2	"		1	22.80	8.25	14.55	12.8	1.17		0.3/0.9				
	"		1	21.00	8.25	12.75	12.8	1.02		0.3/0.9				
	"		1	24.40	8.25	16.15	12.8	1.30		0.3/0.9				
M5-683-SWF-3	"		1	23.60	8.25	15.35	12.8	1.23		0.3/0.9				
	"		1	16.90	8.25	8.65	12.8	0.70		0.3/0.9				
	"		1	22.80	8.25	14.55	12.8	1.17	1.01	0.3/0.9			0.24	23.9
M5-678-SWF-1	"		1	21.4	8.25	13.15	17.1	0.79		0.3/0.9				
	"		1	20.9	8.25	12.65	17.1	0.76		0.3/0.9				
	"		1	18.7	8.25	10.45	17.1	0.63		0.3/0.9				
M5-678-SWF-2	"		1	20.7	8.25	12.45	17.1	0.75		0.3/0.9				
	"		1	20.0	8.25	11.75	17.1	0.71		0.3/0.9				
	"		1	19.9	8.25	11.65	17.1	0.70		0.3/0.9				
M5-678-SWF-3	"		1	20.4	8.25	12.15	17.1	0.73		0.3/0.9				
	"		1	26.1	8.25	17.85	17.1			0.3/0.9				
	"		1	21.7	8.25	13.45	17.1	0.81	0.73	0.3/0.9			0.06	7.8
QA-678-SWF-1-S	"		1	50.7	8.25	42.45	17.1	2.55		0.3/0.9	2			
	"		1	45.4	8.25	37.15	17.1	2.24		0.3/0.9	2			
	"		1	46.0	8.25	37.75	17.1	2.27	2.35	0.3/0.9	2	80.93	0.17	7.4
Instrument Blank	"		1	1.36		1.36	17.1	0.08		0.3/0.9				
CCV-1	"		1	81.9	8.25	73.65	17.1	4.43		0.3/0.9	5	88.62		
CCV-2	"		1	80.1	8.25	71.85	17.1	4.32		0.3/0.9	5	86.46		
CCV-3	"		1	75.8	8.25	67.55	17.1	4.06		0.3/0.9	5	81.28		
CCV-4	"		1	74.2	8.25	65.95	17.1	3.97	4.20	0.3/0.9	5	79.36	0.22	5.2
Instrument Blank	"		1	3.75		3.75	12.8	0.30		0.3/0.9				
CCV-1	"		1	81.9	8.25	73.65	12.8	5.92		0.3/0.9	5	118.39		
CCV-2	"		1	80.1	8.25	71.85	12.8	5.77		0.3/0.9	5	115.50		
CCV-3	"		1	63.1	8.25	54.85	12.8	4.41		0.3/0.9	5	88.17		
CCV-4	"		1	68	8.25	59.75	12.8	4.80		0.3/0.9	5	96.05		
CCV-5	"		1	62.6	8.25	54.35	12.8	4.37		0.3/0.9	5	87.37		
CCV-6	"		1	61.2	8.25	52.95	12.8	4.26		0.3/0.9	5	85.12		
CCV-7	"		1	66.4	8.25	58.15	12.8	4.67		0.3/0.9	5	93.48		
CCV-8	"		1	64.3	8.25	56.05	12.8	4.51		0.3/0.9	5	90.10		
CCV-9	"		1	71.7	8.25	63.45	12.8	5.10		0.3/0.9	5	102.00		
CCV-10	"		1	69.1	8.25	60.85	12.8	4.89		0.3/0.9	5	97.82		
CCV-11	"		1	71.7	8.25	63.45	12.8	5.10		0.3/0.9	5	102.00		
CCV-12	"		1	71.9	8.25	63.65	12.8	5.12		0.3/0.9	5	102.32		
CCV-13	"		1	70.8	8.25	62.55	12.8	5.03		0.3/0.9	5	100.55		
CCV-14	"		1	70.6	8.25	62.35	12.8	5.01		0.3/0.9	5	100.23		
CCV-15	"		1	70.5	8.25	62.25	12.8	5.00		0.3/0.9	5	100.07		
CCV-16	"		1	73.5	8.25	65.25	12.8	5.24		0.3/0.9	5	104.89		
CCV-17	"		1	66.4	8.25	58.15	12.8	4.67		0.3/0.9	5	93.48		
CCV-18	"		1	65.8	8.25	57.55	12.8	4.63		0.3/0.9	5	92.51		
CCV-19	"		1	72	8.25	63.75	12.8	5.12		0.3/0.9	5	102.48		
CCV-20	"		1	70.4	8.25	62.15	12.8	5.00	4.93	0.3/0.9	5	99.91	0.42	8.6

10 % Recalculated Results for Total Mercury in Surface Water

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-6-00 Checked by njs 4-13-00

Sample	QC Batch	Qualifier Note	Dilution	Instrument Reading Peak Height Rep 1,2,3	Method Reagant Blank Peak Hieght	Corrected Reading Peak Height Rep 1,2,3	Y ratio	Hg Concentration ppt	Averaged Result ppt	DETECTION LIMIT/*3 ppt	SPK CONC (ppm)	R%	Standard Deviation	Relative Standard Deviation
M5-693-SWF-1	HG18JF1		1	22.10	1.08	21.02	17.3	1.25		0.3/0.9				
	"		1	22.8	1.08	21.72	17.3	1.29		0.3/0.9				
	"		1	24.5	1.08	23.42	17.3	1.39		0.3/0.9				
M5-693-SWF-2	"		1	22.00	1.08	20.92	17.3	1.24		0.3/0.9				
	"		1	23	1.08	21.92	17.3	1.30		0.3/0.9				
	"		1	23.8	1.08	22.72	17.3	1.35		0.3/0.9				
M5-693-SWF-3	"		1	25.00	1.08	23.92	17.3	1.42		0.3/0.9				
	"		1	25.1	1.08	24.02	17.3	1.43		0.3/0.9				
	"		1	27.4	1.08	26.32	17.3	1.57	1.36	0.3/0.9			0.10	7.6
M5-703-SWF-1	"		1	26.10	1.08	25.02	17.3	1.49		0.3/0.9				
	"		1	25.9	1.08	24.82	17.3	1.48		0.3/0.9				
	"		1	25.5	1.08	24.42	17.3	1.45		0.3/0.9				
M5-703-SWF-2	"		1	24.10	1.08	23.02	17.3	1.37		0.3/0.9				
	"		1	20.9	1.08	19.82	17.3	1.18		0.3/0.9				
	"		1	22.4	1.08	21.32	17.3	1.27		0.3/0.9				
M5-703-SWF-3	"		1	22.60	1.08	21.52	17.3	1.28		0.3/0.9				
	"		1	22.7	1.08	21.62	17.3	1.29		0.3/0.9				
	"		1	22.1	1.08	21.02	17.3	1.25	1.34	0.3/0.9			0.11	8.3
M5-626-SWF-1	"		1	30.2	1.08	29.12	17.3	1.73		0.3/0.9				
	"		1	28.9	1.08	27.82	17.3	1.65		0.3/0.9				
	"		1	32.5	1.08	31.42	17.3			0.3/0.9				
M5-626-SWF-2	"		1	27.4	1.08	26.32	17.3	1.57		0.3/0.9				
	"		1	28.4	1.08	27.32	17.3	1.62		0.3/0.9				
	"		1	27.4	1.08	26.32	17.3	1.57		0.3/0.9				
M5-626-SWF-3	"		1	26.8	1.08	25.72	17.3	1.53		0.3/0.9				
	"		1	26.9	1.08	25.82	17.3	1.54		0.3/0.9				
	"		1	26.1	1.08	25.02	17.3	1.49	1.59	0.3/0.9			0.08	5.0
QA-626-SWF-1-S	"		1	58.7	1.08	57.62	17.3	3.43		0.3/0.9	2			
	"		1	58.1	1.08	57.02	17.3	3.39		0.3/0.9	2			
	"		1	53.6	1.08	52.52	17.3	3.12	3.31	0.3/0.9	2	86.34	0.17	5.0
Instrument Blank	"		1	1.75		1.75	17.3	0.10		0.3/0.9				
CCV-1	"		1	93.3	1.08	92.22	17.3	5.48		0.3/0.9	5	109.68		
CCV-2	"		1	94.3	1.08	93.22	17.3	5.54		0.3/0.9	5	110.87		
CCV-3	"		1	89.7	1.08	88.62	17.3	5.27		0.3/0.9	5	105.40		
CCV-4	"		1	85.2	1.08	84.12	17.3	5.00		0.3/0.9	5	100.05		
CCV-5	"		1	90.9	1.08	89.82	17.3	5.34		0.3/0.9	5	106.83		
CCV-6	"		1	92	1.08	90.92	17.3	5.41		0.3/0.9	5	108.14		
CCV-7	"		1	88.5	1.08	87.42	17.3	5.20		0.3/0.9	5	103.97		
CCV-8	"		1	85.1	1.08	84.02	17.3	5.00		0.3/0.9	5	99.93		
CCV-9	"		1	85.4	1.08	84.32	17.3	5.01		0.3/0.9	5	100.29		
CCV-10	"		1	87.2	1.08	86.12	17.3	5.12		0.3/0.9	5	102.43		
CCV-11	"		1	85.5	1.08	84.42	17.3	5.02		0.3/0.9	5	100.41		
CCV-12	"		1	95.3	1.08	94.22	17.3	5.60		0.3/0.9	5	112.06		
CCV-13	"		1	94.8	1.08	93.72	17.3	5.57		0.3/0.9	5	111.47		
CCV-14	"		1	90	1.08	88.92	17.3	5.29		0.3/0.9	5	105.76		
CCV-15	"		1	92.3	1.08	91.22	17.3	5.42		0.3/0.9	5	108.49		
CCV-16	"		1	95.3	1.08	94.22	17.3	5.60		0.3/0.9	5	112.06		
CCV-17	"		1	92.9	1.08	91.82	17.3	5.46		0.3/0.9	5	109.21		
CCV-18	"		1	91.1	1.08	90.02	17.3	5.35		0.3/0.9	5	107.07		
CCV-19	"		1	89.3	1.08	88.22	17.3	5.25		0.3/0.9	5	104.93		
CCV-20	"		1	86.1	1.08	85.02	17.3	5.06		0.3/0.9	5	101.12		
CCV-21	"		1	83.80	1.08	82.72	17.3	4.92	5.28	0.3/0.9	5	98.38	0.22	4.2

10 % Recalculated Results for Total Mercury in Surface Water

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-6-00 Checked by njs 4-13-00

Sample	QC Batch	Qualifier Note	Dilution	Instrument Reading Peak Height Rep 1,2,3	Method Reagant Blank Peak Hieght	Corrected Reading Peak Height Rep 1,2,3	Y ratio	Hg Concentration ppt	Averaged Result ppt	DETECTION LIMIT/*3 ppt	SPK CONC (ppm)	R%	Standard Deviation	Relative Standard Deviation
M5-714-SWF-1	HG27IF1		1	19.7	0.00	19.70	12.1	1.67		0.3/0.9				
	"		1	15.5	0.00	15.50	12.1	1.32		0.3/0.9				
	"		1	15.1	0.00	15.10	12.1	1.28		0.3/0.9				
M5-714-SWF-2	"		1	16.9	0.00	16.90	12.1	1.44		0.3/0.9				
	"		1	16.5	0.00	16.50	12.1	1.40		0.3/0.9				
	"		1	15.0	0.00	15.00	12.1	1.28		0.3/0.9				
M5-714-SWF-3	"		1	17.0	0.00	17.00	12.1	1.45		0.3/0.9				
	"		1	18.0	0.00	18.00	12.1	1.53		0.3/0.9				
	"		1	16.9	0.00	16.90	12.1	1.44	1.42	0.3/0.9			0.13	8.9
M5-735-SWF-1	"		1	17.9	0.00	17.90	13.9	1.32		0.3/0.9				
	"		1	22.0	0.00	22.00	13.9	1.63		0.3/0.9				
	"		1	24.3	0.00	24.30	13.9	1.80		0.3/0.9				
M5-735-SWF-2	"		1	21.7	0.00	21.70	13.9	1.61		0.3/0.9				
	"		1	22.0	0.00	22.00	13.9	1.63		0.3/0.9				
	"		1	23.9	0.00	23.90	13.9	1.77		0.3/0.9				
M5-735-SWF-3	"		1	27.3	0.00	27.30	13.9	2.02		0.3/0.9				
	"		1	27.7	0.00	27.70	13.9	2.05		0.3/0.9				
	"		1	28.0	0.00	28.00	13.9	2.07	1.77	0.3/0.9			0.25	14.1
QA-735-SWF-1-S	"		1	43.5	0.00	43.50	13.9	3.22		0.3/0.9	2			
	"		1	45.4	0.00	45.40	13.9	3.36		0.3/0.9	2			
	"		1	42.6	0.00	42.60	13.9	3.15	3.24	0.3/0.9	2	73.89	0.11	3.3
Instrument Blank	"		1	1.01		1.01	12.1	0.09		0.3/0.9				
CCV-1	"		1	50.1	0.00	50.1	12.1	4.26		0.3/0.9	5	85.19		
CCV-2	"		1	56.0	0.00	56	12.1	4.76		0.3/0.9	5	95.23		
CCV-3	"		1	58.1	0.00	58.1	12.1	4.94		0.3/0.9	5	98.80		
CCV-4	"		1	58.3	0.00	58.3	12.1	4.96		0.3/0.9	5	99.14		
CCV-5	"		1	55.4	0.00	55.4	12.1	4.71		0.3/0.9	5	94.21		
CCV-6	"		1	58.6	0.00	58.6	12.1	4.98		0.3/0.9	5	99.65		
CCV-7	"		1	55.5	0.00	55.5	12.1	4.72		0.3/0.9	5	94.38		
CCV-8	"		1	53.1	0.00	53.1	12.1	4.51		0.3/0.9	5	90.30		
CCV-9	"		1	55.0	0.00	55	12.1	4.68		0.3/0.9	5	93.53		
CCV-10	"		1	54.0	0.00	54	12.1	4.59		0.3/0.9	5	91.83		
CCV-11	"		1	55.2	0.00	55.2	12.1	4.69		0.3/0.9	5	93.87		
CCV-12	"		1	56.2	0.00	56.2	12.1	4.78		0.3/0.9	5	95.57		
CCV-13	"		1	54.5	0.00	54.5	12.1	4.63		0.3/0.9	5	92.68		
CCV-14	"		1	53.8	0.00	53.8	12.1	4.57		0.3/0.9	5	91.49		
CCV-15	"		1	56.5	0.00	56.5	12.1	4.80		0.3/0.9	5	96.08		
CCV-16	"		1	55.9	0.00	55.9	12.1	4.75		0.3/0.9	5	95.06		
CCV-17	"		1	55.5	0.00	55.5	12.1	4.72		0.3/0.9	5	94.38		
CCV-18	"		1	55.9	0.00	55.9	12.1	4.75	4.71	0.3/0.9	5	95.06	0.17	3.6

10 % Recalculated Results for Total Mercury in Surface Water

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-6-00 Checked by njs 4-13-00

Sample	QC Batch	Qualifier Note	Dilution	Instrument Reading Peak Height Rep 1,2,3	Method Reagant Blank Peak Hieght	Corrected Reading Peak Height Rep 1,2,3	Y ratio	Hg Concentration ppt	Averaged Result ppt	DETECTION LIMIT/*3 ppt	SPK CONC (ppm)	R%	Standard Deviation	Relative Standard Deviation
M5-726-SWF-1	HG08JF1		1	10.5	2.20	8.30	12.5	0.68		0.3/0.9				
	"		1	11.7	2.20	9.50	12.5	0.78		0.3/0.9				
	"		1	9.8	2.20	7.60	12.5	0.63		0.3/0.9				
M5-726-SWF-2	"		1	13.4	2.20	11.20	12.5	0.92		0.3/0.9				
	"		1	8.3	2.20	6.10	12.5	0.50		0.3/0.9				
	"		1	10.3	2.20	8.10	12.5	0.67		0.3/0.9				
M5-726-SWF-3	"		1	11.1	2.20	8.90	12.5	0.73		0.3/0.9				
	"		1	5.7	2.20	3.50	12.5	0.29		0.3/0.9				
	"		1	8.3	2.20	6.10	12.5	0.50	0.63	0.3/0.9			0.18	29.1
M5-726-SWF-1-S	"		1	33.8	2.20	31.60	12.5	2.60		0.3/0.9	2			
	"		1	35.5	2.20	33.30	12.5	2.74		0.3/0.9	2			
	"		1	35.3	2.20	33.10	12.5	2.72	2.69	0.3/0.9	2	102.74	0.08	2.8
Instrument Blank	"		1	0.40		0.40	12.5	0.03		0.3/0.9				
CCV-1	"		1	62.4	2.20	60.2	12.5	4.95		0.3/0.9	5	99.09		
CCV-2	"		1	62.9	2.20	60.7	12.5	5.00		0.3/0.9	5	99.92		
CCV-3	"		1	60.9	2.20	58.7	12.5	4.83		0.3/0.9	5	96.62		
CCV-4	"		1	59.3	2.20	57.1	12.5	4.70		0.3/0.9	5	93.99		
CCV-5	"		1	58.7	2.20	56.5	12.5	4.65		0.3/0.9	5	93.00		
CCV-6	"		1	60.3	2.20	58.1	12.5	4.78		0.3/0.9	5	95.64		
CCV-7	"		1	55.0	2.20	52.8	12.5	4.35		0.3/0.9	5	86.91		
CCV-8	"		1	60.5	2.20	58.3	12.5	4.80		0.3/0.9	5	95.97		
CCV-9	"		1	59.1	2.20	56.9	12.5	4.68		0.3/0.9	5	93.66		
CCV-10	"		1	60.9	2.20	58.7	12.5	4.83		0.3/0.9	5	96.62		
CCV-11	"		1	60.5	2.20	58.3	12.5	4.80		0.3/0.9	5	95.97		
CCV-12	"		1	56.2	2.20	54	12.5	4.44		0.3/0.9	5	88.89		
CCV-13	"		1	61.8	2.20	59.6	12.5	4.91		0.3/0.9	5	98.11		
CCV-14	"		1	60.6	2.20	58.4	12.5	4.81		0.3/0.9	5	96.13		
CCV-15	"		1	60.8	2.20	58.6	12.5	4.82		0.3/0.9	5	96.46		
CCV-16	"		1	58.7	2.20	56.5	12.5	4.65		0.3/0.9	5	93.00		
CCV-17	"		1	69.9	2.20	67.7	12.5	5.57		0.3/0.9	5	111.44		
CCV-18	"		1	63.3	2.20	61.1	12.5	5.03	4.81	0.3/0.9	5	100.58	0.26	5.3

10 % Recalculated Results for Total Mercury in Surface Water

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-6-00 Checked by njs 4-13-00

Sample	QC Batch	Qualifier Note	Dilution	Instrument Reading Peak Height Rep 1,2,3	Method Reagant Blank Peak Hieght	Corrected Reading Peak Height Rep 1,2,3	Y ratio	Hg Concentration ppt	Averaged Result ppt	DETECTION LIMIT/*3 ppt	SPK CONC (ppm)	R%	Standard Deviation	Relative Standard Deviation
M5-738-SWF-1	HG05JF1		1	33.8	5.32	28.48	10.9	2.69		0.3/0.9				
	"		1	35.3	5.32	29.98	10.9	2.83		0.3/0.9				
	"		1	33.1	5.32	27.78	10.9	2.62		0.3/0.9				
M5-738-SWF-2	"		1	36.1	5.32	30.78	10.9	2.91		0.3/0.9				
	"		1	37.3	5.32	31.98	10.9	3.02		0.3/0.9				
	"		1	35.8	5.32	30.48	10.9	2.88		0.3/0.9				
M5-738-SWF-3	"		1	38.9	5.32	33.58	10.9	3.17		0.3/0.9				
	"		1	36.0	5.32	30.68	10.9	2.90		0.3/0.9				
	"		1	34.9	5.32	29.58	10.9	2.79	2.87	0.3/0.9			0.16	5.7
M5-828-SWF-1	"		1	28.6	5.32	23.28	10.9	2.20		0.3/0.9				
	"		1	31.0	5.32	25.68	10.9	2.42		0.3/0.9				
	"		1	34.9	5.32	29.58	10.9	2.79		0.3/0.9				
M5-828-SWF-2	"		1	33.5	5.32	28.18	10.9	2.66		0.3/0.9				
	"		1	36.7	5.32	31.38	10.9	2.96		0.3/0.9				
	"		1	27.9	5.32	22.58	10.9	2.13		0.3/0.9				
M5-828-SWF-3	"		1	30.2	5.32	24.88	10.9	2.35		0.3/0.9				
	"		1	33.5	5.32	28.18	10.9	2.66		0.3/0.9				
	"		1	32.2	5.32	26.88	10.9	2.54	2.52	0.3/0.9			0.27	10.9
M5-718-SWF-1	"		1	10.1	5.32	4.78	12.3	0.40		0.3/0.9				
	"		1	11.9	5.32	6.58	12.3	0.55		0.3/0.9				
	"		1	11.9	5.32	6.58	12.3	0.55		0.3/0.9				
M5-718-SWF-2	"		1	7.4	5.32	2.08	12.3	0.17		0.3/0.9				
	"		1	11.0	5.32	5.68	12.3	0.48		0.3/0.9				
	"		1	11.7	5.32	6.38	12.3	0.53		0.3/0.9				
M5-718-SWF-3	"		1	11.6	5.32	6.28	12.3	0.53		0.3/0.9				
	"		1	11.0	5.32	5.68	12.3	0.48		0.3/0.9				
	"		1	15.5	5.32	10.18	12.3	0.85	0.50	0.3/0.9			0.18	34.9
QA-718-SWF-1-S	"		1	32.1	5.32	26.78	12.3	2.24		0.3/0.9	2			
	"		1	32.9	5.32	27.58	12.3	2.31		0.3/0.9	2			
	"		1	29.6	5.32	24.28	12.3	2.03	2.19	0.3/0.9	2	84.43	0.14	6.6
Instrument Blank	"		1	0.7		0.70	10.9	0.07		0.3/0.9				
CCV-1	"		1	93.3	5.32	87.98	10.9	8.30		0.3/0.9	5	166.08		
CCV-2	"		1	94.3	5.32	88.98	10.9	8.40		0.3/0.9	5	167.97		
CCV-3	"		1	89.7	5.32	84.38	10.9	7.96		0.3/0.9	5	159.28		
CCV-4	"		1	85.2	5.32	79.88	10.9	7.54		0.3/0.9	5	150.79		
CCV-5	"		1	90.9	5.32	85.58	10.9	8.08		0.3/0.9	5	161.55		
CCV-6	"		1	92.0	5.32	86.68	10.9	8.18		0.3/0.9	5	163.63		
CCV-7	"		1	88.5	5.32	83.18	10.9	7.85		0.3/0.9	5	157.02		
CCV-8	"		1	85.1	5.32	79.78	10.9	7.53		0.3/0.9	5	150.60		
CCV-9	"		1	85.4	5.32	80.08	10.9	7.56		0.3/0.9	5	151.17		
CCV-10	"		1	85.4	5.32	80.08	10.9	7.56		0.3/0.9	5	151.17		
CCV-11	"		1	87.2	5.32	81.88	10.9	7.73		0.3/0.9	5	154.57		
CCV-12	"		1	83.8	5.32	78.48	10.9	7.41	7.84	0.3/0.9	5	148.15	0.34	4.3

SERC Mercury Lab / EPA REMAP results
Total Mercury Analysis

: Analysis not performed
: Analysis not required
: Averaged Results

Data Entered by: PMEYER ; 3/14/00
Data Entry Checked by: MWB ; 3/15/00

Sampling Station ID	Matrix	Analysis Method	Collection Date	Collection Time	Digestion Date	Run Date	Holding Time (Days)	Time Elapsed From Dig	Total Hg Units (ppt)	QA/QC Batch ID	QA Data % R	QA Data %RSD	Matrix %R	Notes
M5-622SWF	SW		09/30/99	11:25	10/18/99	10/19/99	28	18	7.45	HG19JF1	0	2.51		
M5-623SWF	SW		09/30/99	9:15	10/18/99	10/19/99	28	18	2.16	HG19JF1	0	4.52		
M5-624SWF	SW		09/30/99	10:18	10/14/99	10/14/99	28	14	2.59	HG14JF1	102.58	4.49		
M5-625SWF	SW		09/30/99	12:57	10/19/99	10/20/99	28	19	2.66	HG20JF1	0	6.98		
M5-626SWF	SW		09/30/99	9:08	10/15/99	10/18/99	28	15	1.59	HG18JF1	104.46	4.98		
M5-627SWF	SW		09/30/99	10:50	10/15/99	10/18/99	28	15	0.88	HG18JF1	104.46	9.21		
M5-628SWF	SW		09/29/99	17:16	10/01/99	10/04/99	28	2	2.80	HG04JF1	95	7.15		
M5-631SWF	SW		09/29/99	14:14	10/01/99	10/04/99	28	2	3.06	HG04JF1	95	7.00		
M5-632SWF	SW		09/29/99	10:10	10/15/99	10/15/99	28	16	0.84	HG15JF1	100.35	13.90		
M5-633SWF	SW		09/29/99	11:15	10/12/99	10/13/99	28	13	2.55	HG13JF1	98.65	8.39		
M5-635SWF	SW		09/29/99	12:16	10/14/99	10/14/99	28	15	1.63	HG14JF1	102.58	13.94		
M5-637SWF	SW		09/29/99	12:10	10/06/99	10/07/99	28	7	1.16	HG07JF1	100	12.64		
M5-638SWF	SW		09/29/99	16:30	10/01/99	10/04/99	28	2	2.26	HG04JF1	95	11.85		
M5-639SWF	SW		09/28/99	17:15	10/15/99	10/18/99	28	17	2.8	HG18JF1	104.46	3.62		
M5-640SWF	SW		09/29/99	11:16	10/15/99	10/15/99	28	16	2.03	HG15JF1	100.35	7.19		
M5-641SWF	SW		09/28/99	16:15	10/01/99	10/04/99	28	3	1.04	HG04JF1	95	21.83		
M5-642SWF	SW		09/29/99	10:11	10/07/99	10/08/99	28	8	1.53	HG08JF1	109	7.80		
M5-643SWF	SW		09/29/99	9:10	10/06/99	10/07/99	28	7	1.18	HG07JF1	0	14.18		
M5-644SWF	SW		09/30/99	11:45	10/18/99	10/19/99	28	18	2.84	HG19JF1	0	6.54		
M5-646SWF	SW		09/28/99	15:15	10/14/99	10/14/99	28	16	1.4	HG14JF1	102.58	8.40		
M5-647SWF	SW		09/28/99	11:02	10/06/99	10/07/99	28	8	1.28	HG07JF1	0	15.60		
M5-648SWF	SW		09/28/99	13:00	10/15/99	10/15/99	28	17	3.89	HG15JF1	100.35	4.14		
M5-649SWF	SW		09/28/99	16:20	09/29/99	09/30/99	28	1	2.45	HG30F1	92.13	9.06		
M5-650SWF	SW		09/28/99	11:58	10/15/99	10/15/99	28	17	1.61	HG15JF1	100.35	9.64		
M5-651SWF	SW		09/28/99	14:10	09/29/99	09/30/99	28	1	1.14	HG30F1	92.13	9.25		
M5-653SWF	SW		09/28/99	13:00	10/15/99	10/15/99	28	17	0.66	HG15JF1	100.35	9.92		
M5-654SWF	SW		09/28/99	11:45	10/15/99	10/18/99	28	17	1.27	HG18JF1	104.46	12.53		
M5-655SWF	SW		09/28/99	10:28	10/14/99	10/14/99	28	16	0.98	HG14JF1	102.58	18.75		
M5-656SWF	SW		09/28/99	9:00	10/15/99	10/18/99	28	17	1.4	HG18JF1	104.46	10.08		
M5-657SWF	SW		09/27/99	17:51	10/04/99	10/05/99	28	7	1.29	HG05JF1	119	6.80		
M5-658SWF	SW		09/28/99	17:22	10/01/99	10/04/99	28	3	1.99	HG04JF1	95	7.00		
M5-659SWF	SW		09/27/99	12:05	10/15/99	10/15/99	28	18	1.31	HG15JF1	100.35	10.53		
M5-660SWF	SW		09/27/99	14:50	09/29/99	09/30/99	28	2	0.53	HG30F1	92.13	3.35		
M5-661SWF	SW		09/29/99	8:57	10/01/99	10/04/99	28	2	2.52	HG04JF1	95	10.03		
M5-662SWF	SW		09/27/99	16:10	10/15/99	10/18/99	28	18	3.56	HG18JF1	104.46	2.96		
M5-663SWF	SW		09/27/99	13:30	09/29/99	09/30/99	28	2	1.47	HG30F1	92.13	3.35		
M5-664SWF	SW		09/27/99	11:00	10/06/99	10/07/99	28	9	1.58	HG07JF1	0	13.25		
M5-665SWF	SW		09/27/99	17:10	09/28/99	09/29/99	28	1	2.16	HG29IF1	129.83	7.01		
M5-666SWF	SW		09/27/99	16:55	09/29/99	09/30/99	28	2	0.68	HG30F1	92.13	14.80		
M5-667SWF	SW		09/26/99	13:10	10/08/99	10/11/99	28	12	0.64	BK11JF1	96.31	25.74		
M5-668SWF	SW		09/27/99	13:07	10/06/99	10/07/99	28	9	0.97	HG07JF1	0	11.89		
M5-669SWF	SW		09/27/99	10:00	10/15/99	10/18/99	28	18	1.66	HG18JF1	104.46	7.04		
M5-670SWF	SW		09/27/99	8:50	10/12/99	10/13/99	28	15	1.64	HG13JF1	98.65	3.15		
M5-672SWF	SW		09/27/99	11:00	09/29/99	10/01/99	28	2	1.60	HG01JF1	101	6.47		
M5-673SWF	SW		09/27/99	14:00	09/28/99	09/29/99	28	1	1.61	HG29IF1	129.83	12.96		
M5-674SWF	SW		09/26/99	10:25	10/01/99	10/04/99	28	5	<MDL	HG04JF1	95	0.00		
M5-675SWF	SW		09/26/99	11:30	10/15/99	10/15/99	28	19	0.92	HG15JF1	100.35	13.48		
M5-676SWF	SW		09/28/99	9:25	10/14/99	10/14/99	28	16	2.08	HG14JF1	102.58	10.12		
M5-677SWF	SW		09/26/99	13:10	10/06/99	10/07/99	28	10	0.95	HG07JF1	100	19.40		
M5-678SWF	SW		09/26/99	12:13	09/28/99	09/29/99	28	2	0.74	HG29IF1	96.97	7.77		
M5-679SWF	SW		09/26/99	13:35	10/06/99	10/07/99	28	10	1.00	HG07JF1	0	10.78		
M5-680SWF	SW		09/27/99	9:00	09/29/99	10/01/99	28	2	0.92	HG01JF1	101	16.11		
M5-681SWF	SW		09/26/99	14:10	10/14/99	10/14/99	28	18	1.2	HG14JF1	102.58	10.93		

Sampling Station ID	Matrix	Analysis Method	Collection Date	Collection Time	Digestion Date	Run Date	Holding Time (Days)	Time Elapsed From Dig	Total Hg Units (ppt)	QA/QC Batch ID	QA Data % R	QA Data %RSD	Matrix %R	Notes
M5-683SWF	SW		09/26/99	8:50	09/28/99	09/29/99	28	2	1.02	HG29IF1	129.83	23.91		
M5-684SWF	SW		09/26/99	15:30	10/07/99	10/08/99	28	11	1.11	HG08JF1	109	10.59		
M5-685SWF	SW		09/26/99	14:34	10/15/99	10/18/99	28	19	1.03	HG18JF1	104.46	11.18		
M5-686SWF	SW		09/25/99	9:15	09/27/99	09/28/99	28	2	1.16	HG28IF1	98.01	18.46		
M5-687SWF	SW		09/25/99	10:35	09/28/99	09/29/99	28	3	1.66	HG29IF1	129.83	7.01		
M5-688SWF	SW		09/26/99	15:27	10/06/99	10/07/99	28	10	0.76	HG07JF1	0	22.80		
M5-689SWF	SW		09/25/99	12:05	10/04/99	10/05/99	28	9	0.55	HG05JF1	119	26.71		
M5-690SWF	SW		09/25/99	13:50	10/07/99	10/08/99	28	12	1	HG08JF1	109	25.30		
M5-691SWF	SW		09/25/99	15:54	10/07/99	10/08/99	28	12	0.89	HG08JF1	109	7.48		
M5-692SWF	SW		09/25/99	12:00	09/28/99	09/29/99	28	3	0.91	HG29IF1	129.83	15.16		
M5-693SWF	SW		09/25/99	11:39	10/15/99	10/18/99	28	20	1.36	HG18JF1	104.46	7.58		
M5-694SWF	SW		09/25/99	17:02	09/27/99	09/28/99	28	2	1.62	HG28IF1	98.01	10.69		
M5-695SWF	SW		09/25/99	14:02	09/28/99	09/29/99	28	3	1.59	HG29IF1	129.83	9.98		
M5-697SWF	SW		09/26/99	16:37	09/27/99	09/28/99	28	1	2..16	HG28IF1	98.01	9.24		
M5-698SWF	SW		09/25/99	10:30	09/29/99	10/01/99	28	4	0.81	HG01JF1	101	20.00		
M5-699SWF	SW		09/25/99	16:35	09/28/99	09/29/99	28	3	1.75	HG29IF1	129.83	11.10		
M5-700SWF	SW		09/25/99	17:50	09/27/99	09/28/99	28	2	2.31	HG28IF1	98.01	18.46		
M5-701SWF	SW		09/26/99	17:40	09/28/99	09/29/99	28	2	0.85	HG29IF1	129.83	7.01		
M5-702SWF	SW		09/25/99	9:18	09/28/99	09/29/99	28	3	0.85	HG29IF1	129.83	10.92		
M5-703SWF	SW		09/24/99	16:55	10/15/99	10/18/99	28	21	1.34	HG18JF1	104.46	8.34		
M5-704SWF	SW		09/24/99	16:15	10/14/99	10/14/99	28	20	1.19	HG14JF1	102.58	26.15		
M5-705SWF	SW		09/24/99	17:25	10/08/99	10/11/99	28	14	0.65	BK11JF1	96.31	29.55		
M5-706SWF	SW		09/24/99	15:55	10/01/99	10/04/99	28	7	0.83	HG04JF1	95	18.03		
M5-707SWF	SW		09/24/99	15:00	10/04/99	10/05/99	28	10	1.39	HG05JF1	119	13.57		
M5-708SWF	SW		09/24/99	9:00	10/08/99	10/11/99	28	14	0.65	BK11JF1	96.31	31.11		
M5-709SWF	SW		09/24/99	13:30	09/27/99	09/28/99	28	3	2.14	HG28IF1	98.01	10.57		
M5-711SWF	SW		09/24/99	11:15	10/08/99	10/11/99	28	14	1	BK11JF1	96.31	14.28		
M5-712SWF	SW		09/24/99	9:05	09/27/99	09/28/99	28	3	2.51	HG28IF1	98.01	13.82		
M5-714SWF	SW		09/23/99	17:15	09/26/99	09/27/99	28	3	1.43	HG27IF1	96.31	8.91		
M5-715SWF	SW		09/24/99	11:45	09/27/99	09/28/99	28	3	2.99	HG28IF1	98.01	4.45		
M5-716SWF	SW		09/24/99	13:10	09/27/99	09/28/99	28	3	2.2	HG28IF1	98.01	18.46		
M5-718SWF	SW		09/24/99	10:30	10/04/99	10/05/99	28	10	0.50	HG05JF1	105	34.92		
M5-720SWF	SW		09/23/99	16:00	09/26/99	09/27/99	28	3	1.45	HG27IF1	96.31	10.55		
M5-722SWF	SW		09/23/99	16:00	09/26/99	09/27/99	28	3	1.26	HG27IF1	96.31	12.31		
M5-723SWF	SW		09/23/99	15:00	10/07/99	10/08/99	28	14	0.91	HG08JF1	109	13.86		
M5-724SWF	SW		09/23/99	14:42	09/26/99	09/27/99	28	3	1.51	HG27IF1	96.31	8.91		
M5-725SWF	SW		09/23/99	13:23	10/12/99	10/13/99	28	19	0.71	HG13JF1	98.65	8.90		
M5-726SWF	SW		09/23/99	12:30	10/07/99	10/08/99	28	14	0.63	HG08JF1	109	29.12		
M5-727SWF	SW		09/23/99	12:16	10/07/99	10/08/99	28	14	1.06	HG08JF1	109	19.25		
M5-728SWF	SW		09/23/99	13:50	10/04/99	10/05/99	28	11	0.76	HG05JF1	119	6.80		
M5-729SWF	SW		09/23/99	10:27	10/14/99	10/14/99	28	21	1.02	HG14JF1	102.58	18.31		
M5-730SWF	SW		09/23/99	11:20	09/26/99	09/27/99	28	3	1.2	HG27IF1	96.31	18.81		
M5-731SWF	SW		09/22/99	17:25	10/08/99	10/11/99	28	16	0.78	BK11JF1	96.31	17.88		
M5-732SWF	SW		09/23/99	9:17	09/26/99	09/27/99	28	3	1.29	HG27IF1	96.31	12.40		
M5-733SWF	SW		09/23/99	9:10	10/08/99	10/11/99	28	15	1.49	BK11JF1	96.31	20.72		
M5-734SWF	SW		09/22/99	15:40	10/07/99	10/08/99	28	15	1.49	HG08JF1	109	14.38		
M5-735SWF	SW		09/22/99		09/26/99	09/27/99	28	4	1.77	HG27IF1	96.31	12.05		
M5-738SWF	SW		09/22/99	14:10	10/04/99	10/05/99	28	12	2.87	HG05JF1	119	5.73		
M5-740SWF	SW		09/22/99	12:45	10/12/99	10/13/99	28	20	2.78	HG13JF1	98.65	4.44		
M5-741SWF	SW		09/22/99	15:34	10/08/99	10/11/99	28	16	2.31	BK11JF1	96.31	6.60		
M5-742SWF	SW		09/22/99	14:18	09/26/99	09/27/99	28	4	1.64	HG27IF1	96.31	9.58		
M5-743SWF	SW		09/22/99	11:30	09/26/99	09/27/99	28	4	2.19	HG27IF1	96.31	8.91		
M5-745SWF	SW		09/22/99	11:20	10/08/99	10/11/99	28	16	1.44	BK11JF1	96.31	9.14		
M5-746SWF	SW		09/22/99	9:48	10/08/99	10/11/99	28	16	1.88	BK11JF1	96.31	8.27		
M5-747SWF	SW		09/22/99	9:42	09/26/99	09/27/99	28	4	3.09	HG27IF1	96.31	9.33		

Sampling Station ID	Matrix	Analysis Method	Collection Date	Collection Time	Digestion Date	Run Date	Holding Time (Days)	Time Elapsed From Dig	Total Hg Units (ppt)	QA/QC Batch ID	QA Data % R	QA Data %RSD	QA Data Matrix %R	Notes
M5-823SWF	SW		09/30/99	9:15	10/19/99	10/20/99	28	19	3.06	HG20JF1	0	14.27		
M5-828SWF	SW		09/29/99	17:16	10/04/99	10/05/99	28	5	2.53	HG05JF1	119	10.87		
M5-838SWF	SW		09/29/99	16:30	10/15/99	10/15/99	28	16	1.86	HG15JF1	100.35	7.21		
M5-848SWF	SW		09/28/99	13:00	09/29/99	10/01/99	28	1	3.81	HG01JF1	101	6.47		
M5-859SWF	SW		09/27/99	12:05	10/15/99	10/15/99	28	18	1.43	HG15JF1	100.35	14.03		
M5-868SWF	SW		09/27/99	13:07	09/29/99	10/01/99	28	2	0.79	HG01JF1	101	16.41		
M5-878SWF	SW		09/26/99	12:13	09/27/99	09/28/99	28	1	1.96	HG28IF1	98.01	12.49		
M5-890SWF	SW		09/25/99	13:50	10/15/99	10/15/99	28	20	1	HG15JF1	100.35	19.04		
M5-908SWF	SW		09/24/99	9:00	09/27/99	09/28/99	28	3	1.72	HG28IF1	98.01	9.78		
M5-920SWF	SW		09/23/99	16:00	10/12/99	10/13/99	28	19	1.14	HG13JF1	98.65	8.08		
M5-932SWF	SW		09/23/99	9:17	10/12/99	10/13/99	28	19	0.65	HG13JF1	98.65	27.72		
M5-944SWF	SW		09/22/99	12:24	10/12/99	10/13/99	28	20	1.69	HG13JF1	98.65	9.95		
QA-630-SWF	SW				10/18/99	10/19/99	28		2.09	HG19JF1	97.14	1.30	70.55	
QA-634-SWF	SW				10/18/99	10/19/99	28		2.08	HG19JF1	97.14	1.30	70.55	
QA-636-SWF	SW				10/18/99	10/19/99	28		2.32	HG19JF1	97.14	1.30	70.55	
QA-645-SWF	SW				10/19/99	10/20/99	28		3.42	HG20JF1	90.85	4.15	94.21	
QA-652-SWF	SW				10/18/99	10/19/99	28		0.8	HG19JF1	97.14	1.30	70.55	
QA-671-SWF	SW				10/19/99	10/20/99	28		1.54	HG20JF1	90.85	4.15	94.21	
QA-682-SWF	SW				10/19/99	10/20/99	28		1.32	HG20JF1	90.85	4.15	94.21	
QA-696-SWF	SW				10/19/99	10/20/99	28		1.5	HG20JF1	90.85	4.15	94.21	
QA-710-SWF	SW				10/18/99	10/19/99	28		0.7	HG19JF1	97.14	1.30	70.55	
QA-719-SWF	SW				10/18/99	10/19/99	28		1.16	HG19JF1	97.14	1.30	70.55	
QA-744-SWF	SW				10/19/99	10/20/99	28		1.58	HG20JF1	90.85	4.15	94.21	
QA-001-CB1	SW				10/05/99	10/06/99	28		ND	HG06JF1	105.74	7.05	119.09	
QA-001-CB2	SW				10/05/99	10/06/99	28		ND	HG06JF1	105.74	7.05	119.09	
QA-002-CB1	SW				10/05/99	10/06/99	28		ND	HG06JF1	105.74	7.05	119.09	
QA-002-CB2	SW				10/05/99	10/06/99	28		ND	HG06JF1	105.74	7.05	119.09	
QA-003-CB1	SW				10/05/99	10/06/99	28		ND	HG06JF1	105.74	7.05	119.09	
QA-003-CB2	SW				10/05/99	10/06/99	28		1.94	HG06JF1	105.74	7.05	119.09	
QA-004-CB1	SW				10/05/99	10/06/99	28		ND	HG06JF1	105.74	7.05	119.09	
QA-004-CB2	SW				10/11/99	10/12/99	28		ND	HG12JF1	97.19	3.21	111.1	
QA-005-CB1	SW				10/11/99	10/12/99	28		ND	HG12JF1	97.19	3.21	111.1	
QA-005-CB2	SW				10/11/99	10/12/99	28		ND	HG12JF1	97.19	3.21	111.1	
QA-006-CB1	SW				10/11/99	10/12/99	28		ND	HG12JF1	97.19	3.21	111.1	
QA-006-CB2	SW				10/11/99	10/12/99	28		ND	HG12JF1	97.19	3.21	111.1	
QA-007-CB1	SW				10/11/99	10/12/99	28		ND	HG12JF1	97.19	3.21	111.1	
QA-007-CB2	SW				10/11/99	10/12/99	28		ND	HG12JF1	97.19	3.21	111.1	
QA-008-CB1	SW				10/11/99	10/12/99	28		ND	HG12JF1	97.19	3.21	111.1	
QA-008-CB2	SW				10/11/99	10/12/99	28		ND	HG12JF1	97.19	3.21	111.1	

September 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-622-SWF Surface Water

Laboratory Records	**Total P**	**Total N**	**TOC**	**Total Hg**	**MeHg**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	09/30/99	09/30/99	09/30/99	09/30/99	09/30/99
Digestion Date	10/08/99	10/08/99	10/21/99	10/18/99	
Analysis Date	10/27/99	11/22/99	10/21/99	10/19/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle is
Total Volume	10ml	Injection Vial	4 ml	1000 ml	the Main Lab
Sample Volume Analyzed	sample aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1	1	1	
Results	0.009	1.16	36.77	7.45	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	AS	CB	SB	JL	
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	Yes	Yes	Yes	Yes	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	EPA1027A	ANTEK 11-22-99	10-21-99/TOC-B	HG19JF1	File ID
Method Blanks	NA	NA	NA	NA	
Instrument Blanks	**	NR	All < MDL	< MDL	
Duplicates (RPD)	All <20 RPD	< 20 RPD	All <20 RPD	<20 RSD	
Matrix Spike Recoveries (75-125)	All Good	All Good	All Good	Good	
Blank Spike/CCV Recoveries	6 of 7 CCV Good	All Good	All Good	All Good	
Detection Range	0.0006 and > ppm	0.03 and >	0.12 and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.9966	NA	0.998	0.9975	
QC Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
QC Limits Met	**	"B (NR)"			

Notes ** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

The Battelle lab is the primary lab for MeHg in Surface water.

Station ID M4-622-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted, Refer to Laboratory and SESD SOP				
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	X	X	X	X	
Sample Run Logs	X	X	X	X	
Instrument Raw Data	X	X	X	X	
Bench Sheets	X	X	X	X	
Sample Preparation Logs	X	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	X	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	**	"B (NR)"			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-633-SWF Surface Water

Laboratory Records	**Total P**	**Total N**	**TOC**	**Total Hg**	**MeHg**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	09/29/99	09/29/99	09/29/99	09/29/99	09/29/99
Digestion Date	10/08/99	10/08/99	10/21/99	10/12/99	
Analysis Date	10/27/99	11/22/99	10/21/99	10/18/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle is
Total Volume	10ml	Injection Vial	4 ml	1000 ml	the Main Lab
Sample Volume Analyzed	sample aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1	1	1	
Results	0.031	1.94	21.02	2.55	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	AS	CB	SB	JL	
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	Yes	Yes	Yes	Yes	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	EPA1027A	ANTEK 11-22-99	10-21-99/TOC-B	HG13JF1	File ID
Method Blanks	NA	NA	NA	NA	
Instrument Blanks	**	NR	All < MDL	< MDL	
Duplicates (RPD)	All <20 RPD	< 20 RPD	All <20 RPD	<20 RSD	
Matrix Spike Recoveries (75-125)	All Good	All Good	All Good	Good	
Blank Spike/CCV Recoveries	6 of 7 CCV Good	All Good	All Good	All Good	
Detection Range	0.0006 and > ppm	0.03 and >	0.12 and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.9966	NA	0.998	0.9994	
QC Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
QC Limits Met	**	"B (NR)"			

Notes

** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

The Battelle lab is the primary lab for MeHg in Surface water.

Station ID M5-633-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted, Refer to Laboratory and SESD SOP				
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	X	X	X	X	
Sample Run Logs	X	X	X	X	
Instrument Raw Data	X	X	X	X	
Bench Sheets	X	X	X	X	
Sample Preparation Logs	X	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	X	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	**	"B (NR)"			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

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"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-643-SWF Surface Water

Laboratory Records	**Total P**	**Total N**	**TOC**	**Total Hg**	**MeHg**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	09/29/99	09/29/99	09/29/99	09/29/99	09/29/99
Digestion Date	10/08/99	10/08/99	10/21/99	10/06/99	
Analysis Date	10/27/99	11/22/99	10/21/99	10/07/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle is
Total Volume	10ml	Injection Vial	4 ml	1000 ml	the Main Lab
Sample Volume Analyzed	sample aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1	1	1	
Results	0.033	1.28	24.94	1.18	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	AS	CB	SB	JL	
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	Yes	Yes	Yes	Yes	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	EPA1027A	ANTEK 11-22-99	10-21-99/TOC-B	HG07JF1	File ID
Method Blanks	NA	NA	NA	NA	
Instrument Blanks	**	NR	All < MDL	< MDL	
Duplicates (RPD)	All <20 RPD	< 20 RPD	All <20 RPD	<20 RSD	
Matrix Spike Recoveries (75-125)	All Good	All Good	All Good	Good	
Blank Spike/CCV Recoveries	6 of 7 CCV Good	All Good	All Good	All Good	
Detection Range	0.0006 and > ppm	0.03 and >	0.12 and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.9966	NA	0.998	0.9984	
QC Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
QC Limits Met	**	"B (NR)"			

Notes

** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

The Battelle lab is the primary lab for MeHg in Surface water.

Station ID M5-643-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted, Refer to Laboratory and SESD SOP				
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	X	X	X	X	
Sample Run Logs	X	X	X	X	
Instrument Raw Data	X	X	X	X	
Bench Sheets	X	X	X	X	
Sample Preparation Logs	X	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	X	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	**	"B (NR)"			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September 1999 Samples for Critical Parameters Analyzed by SERC With the 10% Full QA/QC Review

Station ID M5-653-SWF Surface Water

Laboratory Records	**Total P**	**Total N**	**TOC**	**Total Hg**	**MeHg**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	09/28/99	09/30/99	09/30/99	09/30/99	09/30/99
Digestion Date	10/08/99	10/08/99	10/21/99	10/15/99	
Analysis Date	10/27/99	11/22/99	10/21/99	10/15/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle is
Total Volume	10ml	Injection Vial	4 ml	1000 ml	the Main Lab
Sample Volume Analyzed	sample aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1	1	1	
Results	0.074	1.86	34.31	0.66	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	AS	CB	SB	JL	
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	Yes	Yes	Yes	Yes	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	EPA1027A	ANTEK 11-22-99	10-21-99/TOC-B	HG15JF1	File ID
Method Blanks	NA	NA	NA	NA	
Instrument Blanks	**	NR	All < MDL	< MDL	
Duplicates (RPD)	All <20 RPD	< 20 RPD	All <20 RPD	<20 RSD	
Matrix Spike Recoveries (75-125)	All Good	All Good	All Good	132.17%	
Blank Spike/CCV Recoveries	6 of 7 CCV Good	All Good	All Good	All Good	
Detection Range	0.0006 and > ppm	0.03 and >	0.12 and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.9966	NA	0.998	0.9975	
QC Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
QC Limits Met	**	"B (NR)"		"M"	

Notes

** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

The Battelle lab is the primary lab for MeHg in Surface water.

Station ID M5-653-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted, Refer to Laboratory and SESD SOP				
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	X	X	X	X	
Sample Run Logs	X	X	X	X	
Instrument Raw Data	X	X	X	X	
Bench Sheets	X	X	X	X	
Sample Preparation Logs	X	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	X	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	**	"B (NR)"		"M"	

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-663-SWF Surface Water

Laboratory Records	**Total P**	**Total N**	**TOC**	**Total Hg**	**MeHg**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	09/27/99	09/27/99	09/27/99	09/27/99	09/27/99
Digestion Date	10/08/99	10/08/99	10/21/99	09/29/99	
Analysis Date	10/27/99	11/22/99	10/21/99	09/30/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle is
Total Volume	10ml	Injection Vial	4 ml	1000 ml	the Main Lab
Sample Volume Analyzed	sample aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1	1	1	
Results	0.0078	0.69	19.94	1.51	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	AS	CB	SB	JL	
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	Yes	Yes	Yes	Yes	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	EPA1027A	ANTEK 11-20-99	10-21-99/TOC-B	HG30F1	File ID
Method Blanks	NA	NA	NA	NA	
Instrument Blanks	**	NR	All < MDL	< MDL	
Duplicates (RPD)	All <20 RPD	< 20 RPD	All <20 RPD	<20 RSD	
Matrix Spike Recoveries (75-125)	All Good	All Good	All Good	Good	
Blank Spike/CCV Recoveries	6 of 7 CCV Good	All Good	All Good	All Good	
Detection Range	0.0006 and > ppm	0.03 and >	0.12 and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.9966	NA	0.998	0.9986	
QC Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
QC Limits Met	**	"B (NR)"			

Notes ** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

The Battelle lab is the primary lab for MeHg in Surface water.

Station ID M5-663-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted, Refer to Laboratory and SESD SOP				
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	X	X	X	X	
Sample Run Logs	X	X	X	X	
Instrument Raw Data	X	X	X	X	
Bench Sheets	X	X	X	X	
Sample Preparation Logs	X	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	X	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	**	"B (NR)"			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-673-SWF Surface Water

Laboratory Records	**Total P**	**Total N**	**TOC**	**Total Hg**	**MeHg**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	09/27/99	09/27/99	09/27/99	09/27/99	09/27/99
Digestion Date	10/08/99	10/08/99	10/21/99	09/28/99	
Analysis Date	10/27/99	11/23/99	10/21/99	09/29/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle is
Total Volume	10ml	Injection Vial	4 ml	1000 ml	the Main Lab
Sample Volume Analyzed	sample aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1	1	1	
Results	0.008	0.51	18.9	1.6	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	AS	CB	SB	JL	
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	Yes	Yes	Yes	Yes	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	EPA1027B	ANTEK 11-23-99	10-21-99/TOC-B	HG29IF1	File ID
Method Blanks	NA	NA	NA	NA	
Instrument Blanks	NR	NR	All < MDL	< MDL	
Duplicates (RPD)	<20 RPD	< 20 RPD	All <20 RPD	<20 RSD	
Matrix Spike Recoveries (75-125)	All Good	All Good	All Good	Good	
Blank Spike/CCV Recoveries	All Good	All Good	All Good	All Good	
Detection Range	0.0006 and > ppm	0.03 and >	0.12 and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.9964	NA	0.998	0.9992	
QC Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
QC Limits Met	"B (NR)"	"B (NR)"			

Notes

** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

The Battelle lab is the primary lab for MeHg in Surface water.

Station ID M5-673-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted, Refer to Laboratory and SESD SOP				
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	X	X	X	X	
Sample Run Logs	X	X	X	X	
Instrument Raw Data	X	X	X	X	
Bench Sheets	X	X	X	X	
Sample Preparation Logs	X	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	X	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	"B (NR)"	"B (NR)"			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-683-SWF Surface Water

Laboratory Records	**Total P**	**Total N**	**TOC**	**Total Hg**	**MeHg**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	09/26/99	09/26/99	09/26/99	09/26/99	09/26/99
Digestion Date	10/08/99	10/08/99	10/22/99	09/28/99	
Analysis Date	10/27/99	11/22/99	10/22/99	09/29/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle is
Total Volume	10ml	Injection Vial	4 ml	1000 ml	the Main Lab
Sample Volume Analyzed	sample aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1	1	1	
Results	0.0054	1.16	11.55	1.01	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	AS	CB	SB	JL	
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	Yes	Yes	Yes	Yes	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	EPA1027B	ANTEK 11-23-99	10-22-99/TOC-B	HG29IF1	File ID
Method Blanks	NA	NA	NA	NA	
Instrument Blanks	NR	NR	All < MDL	< MDL	
Duplicates (RPD)	<20 RPD	< 20 RPD	All <20 RPD	<20 RSD	
Matrix Spike Recoveries (75-125)	All Good	All Good	All Good	Good	
Blank Spike/CCV Recoveries	All Good	All Good	All Good	All Good	
Detection Range	0.0006 and > ppm	0.03 and >	0.12 and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.9964	NA	1.0000	0.9992	
QC Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
QC Limits Met	"B (NR)"	"B (NR)"			

Notes

** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

The Battelle lab is the primary lab for MeHg in Surface water.

Station ID M5-683-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted, Refer to Laboratory and SESD SOP				
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	X	X	X	X	
Sample Run Logs	X	X	X	X	
Instrument Raw Data	X	X	X	X	
Bench Sheets	X	X	X	X	
Sample Preparation Logs	X	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	X	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	"B (NR)"	"B (NR)"			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-693-SWF Surface Water

Laboratory Records	**Total P**	**Total N**	**TOC**	**Total Hg**	**MeHg**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	09/25/99	09/25/99	09/25/99	09/25/99	09/25/99
Digestion Date	10/08/99	10/08/99	10/22/99	10/15/99	
Analysis Date	10/27/99	11/23/99	10/22/99	10/18/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle is
Total Volume	10ml	Injection Vial	4 ml	1000 ml	the Main Lab
Sample Volume Analyzed	sample aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1	1	1	
Results	0.0056	0.66	10.22	1.36	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	AS	CB	SB	JL	
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	Yes	Yes	Yes	Yes	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	EPA1027B	ANTEK 11-23-99	10-22-99/TOC-B	HG18JF1	File ID
Method Blanks	NA	NA	NA	NA	
Instrument Blanks	NR	NR	All < MDL	< MDL	
Duplicates (RPD)	<20 RPD	< 20 RPD	All <20 RPD	<20 RSD	
Matrix Spike Recoveries (75-125)	All Good	All Good	All Good	Good	
Blank Spike/CCV Recoveries	All Good	All Good	All Good	All Good	
Detection Range	0.0006 and > ppm	0.03 and >	0.12 and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.9964	NA	1.0000	0.9993	
QC Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
QC Limits Met	"B (NR)"	"B (NR)"			

Notes

** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

The Battelle lab is the primary lab for MeHg in Surface water.

Station ID M5-693-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted, Refer to Laboratory and SESD SOP				
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	X	X	X	X	
Sample Run Logs	X	X	X	X	
Instrument Raw Data	X	X	X	X	
Bench Sheets	X	X	X	X	
Sample Preparation Logs	X	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	X	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	"B (NR)"	"B (NR)"			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-703-SWF Surface Water

Laboratory Records	**Total P**	**Total N**	**TOC**	**Total Hg**	**MeHg**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	09/24/99	09/24/99	09/24/99	09/24/99	09/24/99
Digestion Date	10/08/99	10/08/99	10/22/99	10/15/99	
Analysis Date	10/27/99	11/23/99	10/22/99	10/18/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle is
Total Volume	10ml	Injection Vial	4 ml	1000 ml	the Main Lab
Sample Volume Analyzed	sample aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1	1	1	
Results	0.0053	0.81	14.16	1.34	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	AS	CB	SB	JL	
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	Yes	Yes	Yes	Yes	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	EPA1027B	ANTEK 11-23-99	10-22-99/TOC-B	HG18JF1	File ID
Method Blanks	NA	NA	NA	NA	
Instrument Blanks	NR	NR	All < MDL	< MDL	
Duplicates (RPD)	<20 RPD	< 20 RPD	All <20 RPD	<20 RSD	
Matrix Spike Recoveries (75-125)	All Good	All Good	All Good	Good	
Blank Spike/CCV Recoveries	All Good	All Good	All Good	All Good	
Detection Range	0.0006 and > ppm	0.03 and >	0.12 and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.9964	NA	1.0000	0.9993	
QC Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
QC Limits Met	"B (NR)"	"B (NR)"			

Notes

** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

The Battelle lab is the primary lab for MeHg in Surface water.

Station ID M5-703-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted, Refer to Laboratory and SESD SOP				
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	X	X	X	X	
Sample Run Logs	X	X	X	X	
Instrument Raw Data	X	X	X	X	
Bench Sheets	X	X	X	X	
Sample Preparation Logs	X	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	X	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	"B (NR)"	"B (NR)"			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-714-SWF Surface Water

Laboratory Records	**Total P**	**Total N**	**TOC**	**Total Hg**	**MeHg**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	09/23/99	09/23/99	09/23/99	09/23/99	09/23/99
Digestion Date	10/08/99	10/08/99	10/22/99	09/26/99	
Analysis Date	10/27/99	11/23/99	10/22/99	09/27/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle is
Total Volume	10ml	Injection Vial	4 ml	1000 ml	the Main Lab
Sample Volume Analyzed	sample aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1	1	1	
Results	0.0046	0.60	13.99	1.42	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	AS	CB	SB	JL	
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	Yes	Yes	No	Yes	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	EPA1027B	ANTEK 11-23-99	10-22-99/TOC-B	HG27JF1	File ID
Method Blanks	NA	NA	NA	NA	
Instrument Blanks	NR	NR	All < MDL	< MDL	
Duplicates (RPD)	<20 RPD	< 20 RPD	All <20 RPD	<20 RSD	
Matrix Spike Recoveries (75-125)	All Good	All Good	All Good	Good	
Blank Spike/CCV Recoveries	All Good	All Good	All Good	All Good	
Detection Range	0.0006 and > ppm	0.03 and >	0.12 and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.9964	NA	1.0000	0.9983	
QC Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
QC Limits Met	"B (NR)"	"B (NR)"	"H"		

Notes ** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

The Battelle lab is the primary lab for MeHg in Surface water.

Station ID M5-714-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted, Refer to Laboratory and SESD SOP				
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	X	X	X	X	
Sample Run Logs	X	X	X	X	
Instrument Raw Data	X	X	X	X	
Bench Sheets	X	X	X	X	
Sample Preparation Logs	X	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	X	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	"B (NR)"	"B (NR)"	"H"		

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-726-SWF Surface Water

Laboratory Records	**Total P**	**Total N**	**TOC**	**Total Hg**	**MeHg**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	09/23/99	09/23/99	09/23/99	09/23/99	09/23/99
Digestion Date	10/08/99	10/08/99	10/22/99	10/07/99	
Analysis Date	10/27/99	11/23/99	10/22/99	10/08/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle is
Total Volume	10ml	Injection Vial	4 ml	1000 ml	the Main Lab
Sample Volume Analyzed	sample aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1	1	1	
Results	0.0051	0.40	11.5	0.63	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	AS	CB	SB	JL	
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	Yes	Yes	No	Yes	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	EPA1027C	ANTEK 11-23-99	10-22-99/TOC-B	HG08JF1	File ID
Method Blanks	NA	NA	NA	NA	
Instrument Blanks	**	NR	All < MDL	< MDL	
Duplicates (RPD)	All <20 RPD	< 20 RPD	All <20 RPD	<20 RSD	
Matrix Spike Recoveries (75-125)	All Good	All Good	All Good	Good	
Blank Spike/CCV Recoveries	All Good	All Good	All Good	All Good	
Detection Range	0.0006 and > ppm	0.03 and >	0.12 and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.9957	NA	1.0000	0.9996	
QC Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
QC Limits Met	**	"B (NR)"	"H"		

Notes

** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

The Battelle lab is the primary lab for MeHg in Surface water.

Station ID M5-726-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted, Refer to Laboratory and SESD SOP				
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	X	X	X	X	
Sample Run Logs	X	X	X	X	
Instrument Raw Data	X	X	X	X	
Bench Sheets	X	X	X	X	
Sample Preparation Logs	X	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	X	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	**	"B (NR)"	"H"		

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-738-SWF Surface Water

Laboratory Records	**Total P**	**Total N**	**TOC**	**Total Hg**	**MeHg**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	09/22/99	09/22/99	09/22/99	09/22/99	09/22/99
Digestion Date	10/08/99	10/08/99	10/22/99	10/04/99	
Analysis Date	10/27/99	11/23/99	10/22/99	10/05/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle is
Total Volume	10ml	Injection Vial	4 ml	1000 ml	the Main Lab
Sample Volume Analyzed	sample aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1	1	1	
Results	0.005	0.22	6.33	2.87	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	AS	CB	SB	JL	
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	Yes	Yes	No	Yes	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	EPA1027C	ANTEK 11-23-99	10-22-99/TOC-B	HG05JF1	File ID
Method Blanks	NA	NA	NA	NA	
Instrument Blanks	**	NR	All < MDL	< MDL	
Duplicates (RPD)	All <20 RPD	< 20 RPD	All <20 RPD	<20 RSD	
Matrix Spike Recoveries (75-125)	All Good	All Good	All Good	Good	
Blank Spike/CCV Recoveries	All Good	All Good	All Good	All Good	
Detection Range	0.0006 and > ppm	0.03 and >	0.12 and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.9957	NA	0.999	0.999	
QC Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
QC Limits Met	**	"B (NR)"	"H"		

Notes

** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

The Battelle lab is the primary lab for MeHg in Surface water.

Station ID M5-738-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted, Refer to Laboratory and SESD SOP				
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	X	X	X	X	
Sample Run Logs	X	X	X	X	
Instrument Raw Data	X	X	X	X	
Bench Sheets	X	X	X	X	
Sample Preparation Logs	X	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	X	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	**	"B (NR)"	"H"		

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-828-SWF Surface Water

Laboratory Records	**Total P**	**Total N**	**TOC**	**Total Hg**	**MeHg**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	09/28/99	09/28/99	09/28/99	09/28/99	09/28/99
Digestion Date	10/08/99	10/08/99	10/22/99	10/04/99	
Analysis Date	10/27/99	11/23/99	10/22/99	10/05/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle is
Total Volume	10ml	Injection Vial	4 ml	1000 ml	the Main Lab
Sample Volume Analyzed	sample aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1	1	1	
Results	0.0084	0.55	18.67	2.52	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	AS	CB	SB	JL	
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	Yes	Yes	Yes	Yes	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	EPA1027C	ANTEK 11-23-99	10-22-99/TOC-B	HG05JF1	File ID
Method Blanks	NA	NA	NA	NA	
Instrument Blanks	**	NR	All < MDL	< MDL	
Duplicates (RPD)	All <20 RPD	< 20 RPD	All <20 RPD	<20 RSD	
Matrix Spike Recoveries (75-125)	All Good	All Good	All Good	Good	
Blank Spike/CCV Recoveries	All Good	All Good	All Good	All Good	
Detection Range	0.0006 and > ppm	0.03 and >	0.12 and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.9957	NA	0.999	0.999	
QC Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
QC Limits Met	**	"B (NR)"			

Notes

** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

The Battelle lab is the primary lab for MeHg in Surface water.

Station ID M5-828-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted, Refer to Laboratory and SESD SOP				
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	X	X	X	X	
Sample Run Logs	X	X	X	X	
Instrument Raw Data	X	X	X	X	
Bench Sheets	X	X	X	X	
Sample Preparation Logs	X	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	X	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	**	"B (NR)"			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-944-SWF Surface Water

Laboratory Records

	Total P	Total N	TOC	Total Hg	MeHg
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	FW = SW	FW = SW	FW = SW	FW = SW	FW = SW
Collection Date	09/22/99	09/22/99	09/22/99	09/22/99	09/22/99
Digestion Date	10/08/99	02/24/00	10/22/99	10/12/99	
Analysis Date	10/27/99	02/24/00	10/22/99	10/13/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume	5 ml	Injection Vial	NA	1000 ml	Battelle is
Total Volume	10ml	Injection Vial	4 ml	1000 ml	the Main Lab
Sample Volume Analyzed	sample aliquot	sample aliquot	sample aliquot	sample aliquot	
Dilution	1	1	1	1	
Results	0.0055	0.28	7.77	1.69	
Measuring Unit	ppm	ppm	ppm	ppt	ppt
EPA Method	EPA 365.1	Antek	EPA415.1	EPA 1631	EPA 1630
Analyst	AS	CB	SB	JL	
Method Detection limit	0.0003ppm (0.01umol/L 97)	0.03 ppm	0.12 ppm	0.3 ppt	0.02 ppt
Data Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	Yes	No	No	Yes	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID	EPA1027C	ANTEK 2-24-00	10-22-99/TOC-B	HG13JF1	File ID
Method Blanks	NA	NA	NA	NA	
Instrument Blanks	**	NR	All < MDL	< MDL	
Duplicates (RPD)	All <20 RPD	< 20 RPD	All <20 RPD	<20 RSD	
Matrix Spike Recoveries (75-125)	All Good	74%	All Good	Good	
Blank Spike/CCV Recoveries	All Good	All Good	All Good	All Good	
Detection Range	0.0006 and > ppm	0.03 and >	0.12 and >	0.3 ppt and >	
Correlation Coefficient (>0.995)	0.9957	NA	0.999	0.9994	
QC Report (Verified)					
Data Entry Checked by Another	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
QC Limits Met	**	"B (NR)", "M", "H"	"H"		

Notes

** The blanks are reported above MDL, Lab water was used as a blank but, not in the sample digestion process. Procedure has been corrected to demonstrate samples uncontaminated.

The Battelle lab is the primary lab for MeHg in Surface water.

Station ID M5-944-SWF Surface Water

	Total P	Total N	TOC	Total Hg	MeHg
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted, Refer to Laboratory and SESD SOP				
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	Status Sheet/Run Tracking Log	In Hg lab Area	
Log-in Procedures	Internal COC	Internal COC	Internal COC	None	
Raw Data (Attached)					
Sample Work Sheets	X	X	X	X	
Sample Run Logs	X	X	X	X	
Instrument Raw Data	X	X	X	X	
Bench Sheets	X	X	X	X	
Sample Preparation Logs	X	X	X	X	
Raw Data (Verified)					
Sample ID Transferred	X	X	X	X	
All Calculation Checked	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Measuring Unit	uM	uM	ppm	ppt	
PE Results (Attached)					
Organization	South Florida Water Management District (SFWMD)			FDEP (9-99)	Never compared
Performance (Pass/Fail)	Spring 99 (60%R)	Fall 98 (68.2%R) Fail	Fall 98 (95%R) Pass	Fail	NA
Validation Criteria					
Applied Qualifiers	**	"B (NR)", "M", "H"	"H"		

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

10% Recalculated Results for Methylmercury in Surface Water Analyzed by Battelle Marine Sciences Laboratory for the September 1999 Wet Season (M5)

Entered by mwb / njs 02/04/00 Checked by: njs 4-13-00

Sampling Station ID	Battelle MSL Code	Data Qualifier Note	QC Batch	Instrument Peak Height	Distilled Sample Volume(ml)	Volume Analyzed (ml)	Distillation Correction Factor	Y intercept	Slope (factor)	Blank Correction Factor	Hg Concentration ng/L	DETECTION LIMIT ng/L	TRUE CONC	%R	Relative Percent Difference
M5-622-SWB	1405-115		101399MEB	12772	49.402	49.402	0.890	492	60.6	0	4.61	0.0226			
M5-622-SWB (QA-DUP)	1405-115		101399MEB	13196	49.767	49.767	0.890	492	60.6	0	4.73	0.0224			2.66
M5-635-SWB	1405-125		101399MEB	814	50.816	50.816	0.890	492	60.6	0	0.12	0.0219			
Method Blank	BLK101299		101399MEB	304	50.721	50.721	0.890	492	60.6	0	-0.07	0.022			
Instrument Blank		The Instrument Blanks were performed but, not reported													
M5-623-SWB (QA)	1405-116		101399MEB	3215	49.838	49.838	0.890	492	60.6	0	1.01	0.0224			
M5-623-SWB (QA MS)	1405-116-MS		101399MEB	13100	50.830	50.830	0.890	492	60.6	0	4.60	0.0219	3.43	104.5	
M5-623-SWB (QA MSD)	1405-116-MSD		101399MEB	13576	50.125	50.125	0.890	492	60.6	0	4.84	0.0222	3.48	110.0	5.10
M5-633-SWB (QA)	1405-124		101399MEB	990	50.657	50.657	0.890	492	60.6	0	0.18	0.022			
M5-633-SWB (QA MS)	1405-124-MS		101399MEB	9571	50.015	50.015	0.890	492	60.6	0	3.37	0.0223	3.49	91.2	
M5-633-SWB (QA MSD)	1405-124-MSD		101399MEB	9881	50.733	50.733	0.890	492	60.6	0	3.43	0.022	3.44	94.5	1.93
Dorm-2 (QA-SRM)			101399MEB			0.025					4.21		4.47	94.2	
Std 150 (QA ICV)			101399MEB			0.025					82.5		87.3	94.5	
Std 134 (QA SVS)			101399MEB			0.040					2.74		3.12	87.8	
Std 135 (QA CCV)			101399MEB			0.2					290.0		321	90.3	
Std 135 (QA CCV)			101399MEB			0.100					168.5		161	104.7	
Std 150 (QA CCV)			101399MEB			0.025					77.9		87.3	89.2	
M5-647-SWB	1405-135		101499MEB	1111	49.826	49.826	0.788	475	54	0	0.30	0.0253			
Method Blank	BLK101399		101499MEB	550	50.428	50.428	0.788	475	54	0	0.03	0.0250			
Instrument Blank		The Instrument Blanks were performed but, not reported													
M5-642-SWB (QA)	1405-131		101499MEB	2853	50.525	50.525	0.788	475	54	0	1.11	0.0249			
M5-642-SWB (QA-DUP)	1405-131-DUP		101499MEB	3198	50.757	50.757	0.788	475	54	0	1.26	0.0248			13.07
M5-643-SWB (QA)	1405-132		101499MEB	1505	50.666	50.666	0.788	475	54	0	0.48	0.0248			
M5-643-SWB (QA MS)	1405-132-MS		101499MEB	8275	50.78	50.78	0.788	475	54	0	3.61	0.0248	3.44	91.0	
M5-643-SWB (QA MSD)	1405-132-MSD		101499MEB	8651	49.660	49.660	0.788	475	54	0	3.87	0.0254	3.51	96.6	6.94
M5-656-SWB (QA)	1405-142		101499MEB	990	49.418	49.418	0.788	475	54	0	0.245	0.0255			
M5-656-SWB (QA MS)	1405-142-MS		101499MEB	9063	49.859	49.859	0.788	475	54	0	4.05	0.0252	3.5	108.7	
M5-656-SWB (QA MSD)	1405-142-MSD		101499MEB	8674	50.325	50.325	0.788	475	54	0	3.83	0.025	3.44	104.2	-5.56
DORM-2 (SRM)			101499MEB			0.025					4.11		4.47	91.9	
Std 150 (ICV)			101499MEB			0.025					86.9		87.3	99.5	
Std 134 (SVS)		"M"	101499MEB			0.04					0.000		3.12	0.0	
Std 150 (CCV)			101499MEB			0.025					79.8		87.3	91.4	
Std 134 (SVS)			101499MEB			0.04					3.43		3.12	109.9	
Std 135 (CCV)			101499MEB			0.1					157		161.2	97.4	
Std 150 (CCV)			101499MEB			0.025					88.4		87.3	101.3	
Std 134 (SVS)		"M"	101499MEB			0.04					1.89		3.12	60.6	
Std 134 (SVS)		"M"	101499MEB			0.04					0.000		3.12	0.0	
DORM-2 (SRM)			101499MEB			0.025					4.79		4.47	107.2	
Std 135 (CCV)			101499MEB			0.1					173.9		161.2	107.9	
Std 150 (CCV)			101499MEB			0.025					89.2		87.3	102.2	
Std 150 (CCV)			101499MEB			0.025					81.0		87.3	92.8	
Std 150 (CCV)			101499MEB			0.025					94.8		87.3	108.6	

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

10 % Recalculated Results for Methylmercury in Surface Water Analyzed by Battelle Marine Sciences Laboratory for the September 1999 Wet Season (M5)

Entered by mwb / njs 02/04/00 Checked by: njs 4-13-00

Sampling Station ID	Battelle MSL Code	Data Qualifier Note	QC Batch	Instrument Peak Height	Distilled Sample Volume(ml)	Volume Analyzed (ml)	Distillation Correction Factor	Y intercept	Slope (factor)	Blank Correction Factor	Hg Concentration ng/L	DETECTION LIMIT ng/L	TRUE CONC	%R	Relative Percent Difference
M5-659-SWB	1405-145		101599MEB	783	50.281	50.281	0.838	280	49	0	0.24	0.0235			
M5-669-SWB	1405-155		101599MEB	989	50.405	50.405	0.838	280	49	0	0.34	0.0235			
Method Blank	BLK101499		101599MEB	0	50.462	50.462	0.838	280	49	0	-0.14	0.0235			
Instrument Blank		The Instrument Blanks were performed but, not reported													
M5-657-SWB (QA)	1405-143		101599MEB	474	50.556	50.556	0.838	280	49	0	0.09	0.0234			
M5-657-SWB (QA-DUP)	1405-143-DUP		101599MEB	435	49.713	49.713	0.838	280	49	0	0.08	0.0238			-20.69
M5-661-SWB (QA)	1405-147		101599MEB	993	50.489	50.489	0.838	280	49	0	0.34	0.0234			
M5-661-SWB (QA MS)	1405-147-MS		101599MEB	8434	50.745	50.745	0.838	280	49	0	3.91	0.0233	3.44	103.8	
M5-661-SWB (QA MSD)	1405-147-MSD		101599MEB	7993	50.401	50.401	0.838	280	49	0	3.73	0.0235	3.46	97.8	-4.88
M5-668-SWB (QA)	1405-154		101599MEB	939	50.871	50.871	0.838	280	49	0	0.32	0.0233			
M5-668-SWB (QA MS)	1405-154-MS		101599MEB	8969	50.332	50.332	0.838	280	49	0	4.20	0.0235	3.47	112.1	
M5-668-SWB (QA MSD)	1405-154-MSD		101599MEB	7107	50.577	50.577	0.838	280	49	0	3.29	0.0234	3.45	86.1	-24.48
Dorm-2 (SRM)			101599MEB			0.025					4.32		4.47	96.6	
Std 150 (ICV)			101599MEB			0.025					90.6		87.3	103.8	
Std 134 (SVS)		"M"	101599MEB			0.040					1.37		3.12	43.9	
Std 134 (SVS)			101599MEB			0.040					3.22		3.12	103.2	
Std 150 (CCV)			101599MEB			0.025					90.1		87.3	103.2	
Std 150 (CCV)			101599MEB			0.025					95.4		87.3	109.3	
Std 135 (CCV)			101599MEB			0.050					83.8		80.6	104.0	
M5-674-SWB	1405-1		093099MEB	1551	50.333	50.333	0.928	455	69.6	0	0.34	0.0212			
M5-687-SWB	1405-11		093099MEB	2838	50.878	50.878	0.928	455	69.6	0	0.73	0.021			
Method Blank	BLK092999		093099MEB	360	49.904	49.904	0.928	455	69.6	0	-0.03	0.0214			
Instrument Blank		The Instrument Blanks were performed but, not reported													
M5-684-SWB (QA)	1405-8		093099MEB	1100	50.042	50.042	0.928	455	69.6	0	0.20	0.0214			
M5-684-SWB (QA-DUP)	1405-8-DUP		093099MEB	1150	49.334	49.334	0.928	455	69.6	0	0.22	0.0217			8.89
M5-675-SWB (QA)	1405-2		093099MEB	871	50.331	50.331	0.928	455	69.6	0	0.13	0.0212			
M5-675-SWB (QA MS)	1405-2-MS		093099MEB	12438	49.97	49.97	0.928	455	69.6	0	3.71	0.0214	3.51	102.1	
M5-675-SWB (QA MSD)	1405-2-MSD		093099MEB	12031	49.853	49.853	0.928	455	69.6	0	3.60	0.0214	3.48	99.6	-3.22
M5-685-SWB (QA)	1405-9		093099MEB	607	50.496	50.496	0.928	455	69.6	0	0.05	0.0212			
M5-685-SWB (QA MS)	1405-9-MS		093099MEB	12073	49.97	49.97	0.928	455	69.6	0	3.60	0.0214	3.49	101.8	
M5-685-SWB (QA MSD)	1405-9-MSD		093099MEB	11462	49.853	49.853	0.928	455	69.6	0	3.42	0.0214	3.5	96.3	-5.17
Dorm-2 (ICV)			093099MEB			0.025					4.11		4.47	91.9	
Std 150 (ICV)			093099MEB			0.050					173.7		174.5	99.5	
Std 134 (SVS)		"M"	093099MEB			0.100					5.19		7.79	66.6	
Std 134 (SVS)		"M"	093099MEB			0.040					1.08		3.12	34.6	
Std 150 (CCV)			093099MEB			0.050					170		174.5	97.4	
Std 134 (SVS)			093099MEB			0.040					2.36		3.12	75.6	
Std 134 (SVS)			093099MEB			0.100					7.92		7.79	101.7	
Std 150 (CCV)			093099MEB			0.050					180.8		174.5	103.6	
Std 150 (CCV)			093099MEB			0.05					174		174.5	99.7	

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

10 % Recalculated Results for Methylmercury in Surface Water Analyzed by Battelle Marine Sciences Laboratory for the September 1999 Wet Season (M5)

Entered by mwb / njs 02/04/00 Checked by: njs 4-13-00

Sampling Station ID	Battelle MSL Code	Data Qualifier Note	QC Batch	Instrument Peak Height	Distilled Sample Volume(ml)	Volume Analyzed (ml)	Distillation Correction Factor	Y intercept	Slope (factor)	Blank Correction Factor	Hg Concentration ng/L	DETECTION LIMIT ng/L	TRUE CONC	%R	Relative Percent Difference
M5-699-SWB	1405-21		100199MEB	1313	50.686	50.686	0.937	490	69.3	0	0.25	0.0209			
M5-699-SWB (QA-DUP)	1405-21-DUP		100199MEB	1305	49.514	49.514	0.937	490	69.3	0	0.25	0.0214			1.36
Method Blank	BLK093099		100199MEB	0	50.745	50.745	0.937	490	69.3	0	-0.1487	0.0209			
Instrument Blank		The Instrument Blanks were performed but, not reported													
M5-693-SWB (QA)	1405-17		100199MEB	798	50.083	50.083	0.937	490	69.3	0	0.09	0.0211			
M5-693-SWB (QA MS)	1405-17-MS		100199MEB	12026	49.923	49.923	0.937	490	69.3	0	3.56	0.0212	3.51	98.7	
M5-693-SWB (QA MSD)	1405-17-MSD		100199MEB	11747	50.018	50.018	0.937	490	69.3	0	3.47	0.0212	3.48	96.9	-2.64
M5-703-SWB (QA)	1405-25		100199MEB	1333	49.631	49.631	0.937	490	69.3	0	0.26	0.0213			
M5-703-SWB (QA MS)	1405-25-MS		100199MEB	12023	50.19	50.19	0.937	490	69.3	0	3.54	0.0211	3.49	93.9	
M5-703-SWB (QA MSD)	1405-25-MSD		100199MEB	13908	50.062	50.062	0.937	490	69.3	0	4.13	0.0211	3.5	110.5	15.36
Dorm-2 (QA CCV)			100199MEB			0.025					4.20		4.47	94.0	
Std 134 (QA CCV)			100199MEB			0.040					2.71		3.12	87.0	
Std 150 (QA CCV)			100199MEB			0.05					165.2		174.5	94.7	
Std 150 (QA CCV)			100199MEB			0.025					88.9		87.25	101.9	
Std 150 (QA CCV)			100199MEB			0.025					102.4		87.25	117.4	
Std 135 (QA CCV)			100199MEB			0.050					85.5		80.6	106.1	
Std 135 (QA CCV)			100199MEB			0.05					85.2		80.6	105.7	
M5-709-SWB	1405-31		100599MEB	650	49.733	49.733	0.842	332	68.3	0	0.11	0.0237			
M5-724-SWB	1405-41		100599MEB	646	49.353	49.353	0.842	332	68.3	0	0.11	0.0239			
Method Blank	BLK100499		100599MEB	0	50.825	50.825	0.842	332	68.3	0	-0.11	0.0232			
Instrument Blank		The Instrument Blanks were performed but, not reported													
M5-720-SWB (QA)	1405-38		100599MEB	1177	49.97	49.97	0.842	332	68.3	0	0.29	0.0236			
M5-720-SWB (QA-DUP)	1405-38-DUP		100599MEB	1144	50.399	50.399	0.842	332	68.3	0	0.28	0.0234			-4.84
M5-707-SWB (QA)	1405-29		100599MEB	759	49.956	49.956	0.842	332	68.3	0	0.15	0.0236			
M5-707-SWB (QA MS)	1405-29-MS		100599MEB	11609	49.747	49.747	0.842	332	68.3	0	3.94	0.0237	3.51	108.1	
M5-707-SWB (QA MSD)	1405-29-MSD		100599MEB	12339	49.529	49.529	0.842	332	68.3	0	4.22	0.0238	3.52	115.5	6.71
M5-712-SWB (QA)	1405-33		100599MEB	597	49.446	49.446	0.842	332	68.3	0	0.09	0.0238			
M5-712-SWB (QA MS)	1405-33-MS		100599MEB	9925	50.461	50.461	0.842	332	68.3	0	3.31	0.0233	3.46	92.8	
M5-712-SWB (QA MSD)	1405-33-MSD		100599MEB	8998	50.369	50.369	0.842	332	68.3	0	2.99	0.0234	3.46	83.8	-9.97
Dorm-2 (SRM)			100599MEB			0.025					4.22		4.47	94.4	
Std 150 (ICV)			100599MEB			0.100					332		349	95.1	
Std 134 (SVS)			100599MEB			0.040					3.66		3.12	117.3	
Std 134 (SVS)			100599MEB			0.040					3.32		3.12	106.4	
Std 150 (CCV)			100599MEB			0.025					85.7		87.3	98.2	
Std 150 (CCV)			100599MEB			0.025					95.7		87.3	109.6	
Std 150 (CCV)			100599MEB			0.025					94.0		87.3	107.7	
Std 150 (CCV)			100599MEB			0.025					86.0		87.3	98.5	

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

10 % Recalculated Results for Methylmercury in Surface Water Analyzed by Battelle Marine Sciences Laboratory for the September 1999 Wet Season (M5)

Entered by mwb / njs 02/04/00 Checked by: njs 4-13-00

Sampling Station ID	Battelle MSL Code	Data Qualifier Note	QC Batch	Instrument Peak Height	Distilled Sample Volume(ml)	Volume Analyzed (ml)	Distillation Correction Factor	Y intercept	Slope (factor)	Blank Correction Factor	Hg Concentration ng/L	DETECTION LIMIT ng/L	TRUE CONC	%R	Relative Percent Difference
M5-734-SWB	1405-51		100699MEB	646	49.353	49.353	0.841	305	67.3	0	0.12	0.0235			
Method Blank	BLK100599		100699MEB	130	49.082	49.082	0.841	305	67.3	0	-0.06	0.024			
Instrument Blank		The Instrument Blanks were performed but, not reported													
M5-726-SWB (QA)	1405-43	"DQO"	100699MEB	595	49.411	49.411	0.841	305	67.3	0	0.10	0.0239			
M5-726-SWB (QA-DUP)	1405-43-DUP	"DQO"	100699MEB	717	49.481	49.481	0.841	305	67.3	0	0.15	0.0238			34.62
M5-727-SWB (QA)	1405-44		100699MEB	521	50.22	50.22	0.841	305	67.3	0	0.08	0.0235			
M5-727-SWB (QA MS)	1405-44-MS		100699MEB	10222	50.291	50.291	0.841	305	67.3	0	3.48	0.0235	3.47	98.2	
M5-727-SWB (QA MSD)	1405-44-MSD		100699MEB	10097	50.205	50.205	0.841	305	67.3	0	3.45	0.0235	3.48	96.8	-1.10
M5-738-SWB (QA)	1405-53		100699MEB	421	50.223	50.223	0.841	305	67.3	0	0.04	0.0235			
M5-738-SWB (QA MS)	1405-53-MS		100699MEB	10582	49.251	49.251	0.841	305	67.3	0	3.69	0.0239	3.54	103.0	
M5-738-SWB (QA MSD)	1405-53-MSD		100699MEB	10461	49.200	49.200	0.841	305	67.3	0	3.65	0.024	3.55	101.6	-1.08
Dorm-2 (SRM)			100699MEB			0.025					3.76		4.47	84.1	
Std 150 (ICV)			100699MEB			0.025					95.40		87.3	109.3	
Std 134 (SVS)		"M"	100699MEB			0.04					2.32		3.12	74.4	
Std 134 (SVS)			100699MEB			0.04					3.70		3.12	118.6	
Std 150 (CCV)			100699MEB			0.025					89.80		87.3	102.9	
Std 150 (CCV)			100699MEB			0.025					85.30		87.3	97.7	
Std 150 (CCV)			100699MEB			0.03					81.70		87.3	93.6	
M5-859-SWB	1405-165		101999MEB	1002	50.384	50.384	0.919	279	71.1	0	0.22	0.0214			
Method Blank	BLK101899-r1		101999MEB	0	50.211	50.211	0.919	279	71.1	0	-0.09	0.0215			
Method Blank	BLK101899-r2		101999MEB	211	49.889	49.889	0.919	352	75.6	0	-0.04	0.0216			
Instrument Blank		The Instrument Blanks were performed but, not reported													
M5-823-SWB (QA)	1405-161		101999MEB	4296	50.069	50.069	0.919	279	71.1	0	1.23	0.0216			
M5-823-SWB (QA-DUP)	1405-161-DUP		101999MEB	4413	50.635	50.635	0.919	279	71.1	0	1.25	0.0213			1.75
QA-636-SWB (QA)	1405-171		101999MEB	3031	50.64	50.64	0.919	352	75.6	0	0.76	0.0213			
QA-636-SWB (QA-DUP)	1405-171-DUP		101999MEB	3069	50.649	50.649	0.919	352	75.6	0	0.77	0.0213			1.39
M5-672-SWB (QA)	1405-157		101999MEB	1587	50.707	50.707	0.919	279	71.1	0	0.39	0.0213			
M5-672-SWB (QA MS)	1405-157-MS		101999MEB	12483	50.311	50.311	0.919	279	71.1	0	3.71	0.0215	3.47	95.6	
M5-672-SWB (QA MSD)	1405-157-MSD		101999MEB	13752	49.238	49.238	0.919	279	71.1	0	4.19	0.0219	3.54	107.1	12.03
QA-630-SWB (QA)	1405-169		101999MEB	522	50.835	50.835	0.919	279	71.1	0	0.07	0.0212			
QA-630-SWB (QA MS)	1405-169-MS		101999MEB	12575	50.42	50.42	0.919	279	71.1	0	3.73	0.0214	3.46	105.8	
QA-630-SWB (QA MSD)	1405-169-MSD		101999MEB	12880	49.737	49.737	0.919	279	71.1	0	3.88	0.0217	3.51	108.4	3.81
QA-651-SWB (QA)	1405-174		101999MEB	1448	50.938	50.938	0.919	352	75.6	0	0.31	0.0212			
QA-651-SWB (QA MS)	1405-174-MS		101999MEB	11139	50.009	50.009	0.919	352	75.6	0	3.10	0.0216	3.49	80.1	
QA-651-SWB (QA MSD)	1405-174-MSD		101999MEB	12623	49.971	49.971	0.919	352	75.6	0	3.53	0.0216	3.49	92.4	12.95
Dorm-2 (SRM)			101999MEB			0.025					4.24		4.47	94.9	
Std 150 (ICV)			101999MEB			0.025					88.6		87.3	101.5	
Std 134 (SVS)			101999MEB			0.040					2.87		3.12	92.0	
Std 134 (SVS)			101999MEB			0.040					2.74		3.12	87.8	
Std 134 (SVS)		"M"	101999MEB			0.040					1.94		3.12	62.2	
Std 150 (CCV)			101999MEB			0.025					103		87	118.0	
Std 135 (CCV)			101999MEB			0.200					343		322	106.5	
Std 150 (CCV)			101999MEB			0.025					88.1		87.3	100.9	
Std 134 (SVS)		"M"	101999MEB			0.040					0.0000		3.12	0.0	
Std 150 (CCV)			101999MEB			0.025					90.2		87.3	103.3	
Std 150 (CCV)			101999MEB			0.025					96.3		87.3	110.3	
Std 150 (CCV)			101999MEB			0.025					87.6		87.3	100.3	
Std 150 (CCV)			101999MEB			0.025					93.0		87.3	106.5	

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

10 % Recalculated Results for Methylmercury in Surface Water Analyzed by Battelle Marine Sciences Laboratory for the September 1999 Wet Season (M5)

Entered by mwb / njs 02/04/00 Checked by: njs 4-13-00

Sampling Station ID	Battelle MSL Code	Data Qualifier Note	QC Batch	Instrument Peak Height	Distilled Sample Volume(ml)	Volume Analyzed (ml)	Distillation Correction Factor	Y intercept	Slope (factor)	Blank Correction Factor	Hg Concentration ng/L	DETECTION LIMIT ng/L	TRUE CONC	%R	Relative Percent Difference
M5-878-SWB	1405-61		100799MEB	855	50.872	50.872	0.777	386	67.4	0	0.18	0.0251			
Method Blank	BLK100699		100799MEB	196	50.234	50.234	0.777	386	67.4	0	-0.07	0.0254			
Instrument Blank		The Instrument Blanks were performed but, not reported													
QA-671-SWB (QA)	1405-68		100799MEB	1329	50.889	50.889	0.777	386	67.4	0	0.35	0.0251			
QA-671-SWB (QA-DUP)	1405-68-DUP		100799MEB	1248	50.196	50.196	0.777	386	67.4	0	0.33	0.0254			-7.61
M5-746-SWB (QA)	1405-59		100799MEB	485	49.59	49.59	0.777	386	67.4	0	0.04	0.0257			
M5-746-SWB (QA MS)	1405-59-MS		100799MEB	10284	50.065	50.065	0.777	386	67.4	0	3.78	0.0255	3.49	107.1	
M5-746-SWB (QA MSD)	1405-59-MSD		100799MEB	10450	50.408	50.408	0.777	386	67.4	0	3.81	0.0253	3.46	109.1	0.98
M5-920-SWB (QA)	1405-64		100799MEB	1053	51.002	51.002	0.777	386	67.4	0	0.25	0.025			
M5-920-SWB (QA MS)	1405-64-MS	"DQO"	100799MEB	10807	49.284	49.284	0.777	386	67.4	0	4.04	0.0259	3.54	107.0	
M5-920-SWB (QA MSD)	1405-64-MSD	"DQO"	100799MEB	8027	50.317	50.317	0.777	386	67.4	0	2.90	0.0254	3.47	76.4	-32.80
Dorm-2 (SRM)			100799MEB			0.025					3.75		4.47	83.9	
Std 150 (ICV)			100799MEB			0.025					99.8		87.3	114.3	
Std 134 (SVS)			100799MEB			0.04					3.00		3.12	96.2	
Std 134 (SVS)		"M"	100799MEB			0.04					1.20		3.12	38.5	
Std 150 (CCV)			100799MEB			0.025					69.3		87.3	79.4	
Std 150 (CCV)			100799MEB			0.025					86.8		87.3	99.4	
Std 150 (CCV)			100799MEB			0.025					91.8		87.3	105.2	
Std 150 (CCV)			100799MEB			0.025					88.2		87.3	101.0	
Std 150 (CCV)			100799MEB			0.03					76.5		87.3	87.6	

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September (M5) 1999 Samples for Critical Parameters Analyzed by Battelle With the 10% Full QA/QC Review

Total Methylmercury in Surface Water

	Sampling Station ID			
Laboratory Records	**M5-622-SWB**	**M5-633-SWB**	**M5-643-SWB**	**M5-656-SWB**
Data Report (Attached)				
Laboratory ID Code	1405-115	1405-124	1405-132	1405-142
Sampling Location ID	M5-622-SWB	M5-633-SWB	M5-643-SWB	M5-656-SWB
Sample Type	surface water = SW	surface water = SW	surface water = SW	surface water = SW
Collection Date	09/30/99	09/29/99	09/29/99	09/28/99
Digestion Date	10/12/99	10/12/99	10/13/99	10/13/99
Analysis Date	10/13/99	10/13/99	10/14/99	10/14/99
QC Batch ID	101399MEB	101399MEB	101499MEB	101499MEB
Digestion Volume	49.402	50.657	50.666	49.418
Total Volume	49.402	50.657	50.666	49.418
Sample Volume Analyzed	aliquot	aliquot	aliquot	aliquot
Dilution	1	1	1	1
Results	4.61	0.18	0.48	0.245
Measuring Unit	ng/L (ppt)	ng/L (ppt)	ng/L (ppt)	ng/L (ppt)
EPA Method	1631/1630	1631/1630	1631/1630	1631/1630
Analyst	Niewolny	Niewolny	Niewolny	Niewolny
Method Detection limit	0.0226	0.022	0.0248	0.0255
Data Report (Verified)				
Data Entry Checked by Another	NR	NR	NR	NR
All Calculation Checked	LIMS	LIMS	LIMS	LIMS
Holding Time Met	Yes	Yes	Yes	Yes
QC Report (Attached)				
Laboratory ID Code	1405-115	1405-124	1405-132	1405-142
QC Batch ID	101399MEB	101399MEB	101499MEB	101499MEB
Method Blanks	Good	Good	Good	Good
Instrument Blanks	NR	NR	NR	NR
Duplicates (RPD)	All Good	All Good	All Good	All Good
Matrix Spike Recoveries (75-125)	All Good	All Good	"M"	"M"
Blank Spike/CCV Recoveries	All Good	All Good	All Good	All Good
Detection Range	0.0226 and >	0.022 and >	0.0248 and >	0.0255 and >
Correlation Coefficient (>0.995)	0.9989	0.9989	0.9991	0.9991
QC Report (Verified)				
Data Entry Checked by Another	NR	NR	NR	NR
All Calculation Checked	LIMS	LIMS	LIMS	LIMS
QC Limits Met			"M"	"M"

Notes

"*" Method blank is above the true MDL, but less than 3 times the MDL.

The instrument blanks were performed but, not reported.

	M5-622-SWB	M5-633-SWB	M5-643-SWB	M5-656-SWB
Narrative Description (Attached)				
Total Samples/Matrix	151/Waters	151/Waters	151/Waters	151/Waters
Methods/Parameters	X	X	X	X
Range of Samples analyzed	X	X	X	X
Holding Time Summary	X	X	X	X
Analytical Problems	X	X	X	X
QA/QC Acceptance Limits	X	X	X	X
Integrity of Data Quality	X	X	X	X
Deviations From SOP	X	X	X	X
Observations	X	X	X	X
Sample Management Records				
Sampling Location ID	X	X	X	X
Matrix	X	X	X	X
Preservative (Acid, Temp..)	NR	NR	NR	NR
Collection Data/Time	X	X	X	X
Laboratory ID Code	X	X	X	X
Sample Handling/Storage	X	X	X	X
Log-in Procedures	NR	NR	NR	NR
Raw Data (Attached)				
Sample Work Sheets	X	X	X	X
Sample Run Logs	X	X	X	X
Instrument Raw Data	X	X	X	X
Bench Sheets	NA	NA	NA	NA
Sample Preparation Logs	X	X	X	X
Raw Data (Verified)				
Sample ID Transferred	X	X	X	X
All Calculation Checked	X	X	X	X
Measuring Unit	ng/L	ng/L	ng/L	ng/L
PE Results (Attached)				
Organization	NR	NR	NR	NR
Performance (Pass/Fail)	NR	NR	NR	NR
Validation Criteria				
Applied Qualifiers			"M"	"M"

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavaliable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September (M5) 1999 Samples for Critical Parameters Analyzed by Battelle With the 10% Full QA/QC Review

Total Methylmercury in Surface Water

Laboratory Records	Sampling Station ID			
	M5-661-SWB	**M5-672-SWB**	**M5-684-SWB**	**M5-693-SWB**
Data Report (Attached)				
Laboratory ID Code	1405-147	1405-157	1405-8	1405-17
Sampling Location ID	M5-661-SWB	M5-672-SWB	M5-684-SWB	M5-693-SWB
Sample Type	surface water = SW	surface water = SW	surface water = SW	surface water = SW
Collection Date	09/29/99	09/27/99	09/26/99	09/25/99
Digestion Date	10/14/99	10/18/99	09/29/99	09/30/99
Analysis Date	10/15/99	10/19/99	09/30/99	10/01/99
QC Batch ID	101599MEB	101999MEB	093099MEB	100199MEB
Digestion Volume	50.489	50.707	50.042	50.083
Total Volume	50.489	50.707	50.042	50.083
Sample Volume Analyzed	aliquot	aliquot	aliquot	aliquot
Dilution	1	1	1	1
Results	0.34	0.39	0.2	0.09
Measuring Unit	ng/L (ppt)	ng/L (ppt)	ng/L (ppt)	ng/L (ppt)
EPA Method	1631/1630	1631/1630	1631/1630	1631/1630
Analyst	Niewolny	Niewolny	Niewolny	Niewolny
Method Detection limit	0.0234	0.0213	0.0214	0.0211
Data Report (Verified)				
Data Entry Checked by Another	NR	NR	NR	NR
All Calculation Checked	LIMS	LIMS	LIMS	LIMS
Holding Time Met	Yes	Yes	Yes	Yes
QC Report (Attached)				
Laboratory ID Code	1405-147	1405-157	1405-8	1405-17
QC Batch ID	101599MEB	101999MEB	093099MEB	100199MEB
Method Blanks	Good	Good	Good	Good
Instrument Blanks	NR	NR	NR	NR
Duplicates (RPD)	All Good	All Good	All Good	All Good
Matrix Spike Recoveries (75-125)	"M"	"M"	"M"	All Good
Blank Spike/CCV Recoveries	All Good	All Good	All Good	All Good
Detection Range	0.0234 and >	0.0213 and >	0.0214 and >	0.0211 and >
Correlation Coefficient (>0.995)	0.9976	0.996	0.99952	0.99768
QC Report (Verified)				
Data Entry Checked by Another	NR	NR	NR	NR
All Calculation Checked	LIMS	LIMS	LIMS	LIMS
QC Limits Met	"M"	"M"	"M"	

Notes

"*" Method blank is above the true MDL, but less than 3 times the MDL.

The instrument blanks were performed but, not reported.

	M5-661-SWB	M5-672-SWB	M5-684-SWB	M5-693-SWB
Narrative Description (Attached)				
Total Samples/Matrix	151/Waters	151/Waters	151/Waters	151/Waters
Methods/Parameters	X	X	X	X
Range of Samples analyzed	X	X	X	X
Holding Time Summary	X	X	X	X
Analytical Problems	X	X	X	X
QA/QC Acceptance Limits	X	X	X	X
Integrity of Data Quality	X	X	X	X
Deviations From SOP	X	X	X	X
Observations	X	X	X	X
Sample Management Records				
Sampling Location ID	X	X	X	X
Matrix	X	X	X	X
Preservative (Acid, Temp..)	NR	NR	NR	NR
Collection Data/Time	X	X	X	X
Laboratory ID Code	X	X	X	X
Sample Handling/Storage	X	X	X	X
Log-in Procedures	NR	NR	NR	NR
Raw Data (Attached)				
Sample Work Sheets	X	X	X	X
Sample Run Logs	X	X	X	X
Instrument Raw Data	X	X	X	X
Bench Sheets	NA	NA	NA	NA
Sample Preparation Logs	X	X	X	X
Raw Data (Verified)				
Sample ID Transferred	X	X	X	X
All Calculation Checked	X	X	X	X
Measuring Unit	ng/L	ng/L	ng/L	ng/L
PE Results (Attached)				
Organization	NR	NR	NR	NR
Performance (Pass/Fail)	NR	NR	NR	NR
Validation Criteria				
Applied Qualifiers	"M"	"M"	"M"	

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavaliable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September (M5) 1999 Samples for Critical Parameters Analyzed by Battelle With the 10% Full QA/QC Review

Total Methylmercury in Surface Water

Laboratory Records	Sampling Station ID			
	M5-703-SWB	**M5-712-SWB**	**M5-726-SWB**	**M5-738-SWB**
Data Report (Attached)				
Laboratory ID Code	1405-25	1405-33	1405-43	1405-142
Sampling Location ID	M5-703-SWB	M5-712-SWB	M5-726-SWB	M5-738-SWB
Sample Type	surface water = SW	surface water = SW	surface water = SW	surface water = SW
Collection Date	09/24/99	09/24/99	09/23/99	09/22/99
Digestion Date	09/30/99	10/18/99	10/05/99	10/05/99
Analysis Date	10/01/99	10/19/99	10/06/99	10/06/99
QC Batch ID	100199MEB	100599MEB	100699MEB	100699MEB
Digestion Volume	49.631	49.446	49.411	50.223
Total Volume	49.631	49.446	49.411	50.223
Sample Volume Analyzed	aliquot	aliquot	aliquot	aliquot
Dilution	1	1	1	1
Results	0.26	0.09	0.1	0.04
Measuring Unit	ng/L (ppt)	ng/L (ppt)	ng/L (ppt)	ng/L (ppt)
EPA Method	1631/1630	1631/1630	1631/1630	1631/1630
Analyst	Niewolny	Niewolny	Niewolny	Niewolny
Method Detection limit	0.0213	0.0238	0.0239	0.0235
Data Report (Verified)				
Data Entry Checked by Another	NR	NR	NR	NR
All Calculation Checked	LIMS	LIMS	LIMS	LIMS
Holding Time Met	Yes	Yes	Yes	Yes
QC Report (Attached)				
Laboratory ID Code	1405-25	1405-33	1405-43	1405-142
QC Batch ID	100199MEB	100599MEB	100699MEB	100699MEB
Method Blanks	Good	Good	Good	Good
Instrument Blanks	NR	NR	NR	NR
Duplicates (RPD)	All Good	All Good	34.62	34.62
Matrix Spike Recoveries (75-125)	All Good	All Good	"M"	"M"
Blank Spike/CCV Recoveries	All Good	All Good	All Good	All Good
Detection Range	0.0213 and >	0.0238 and >	0.0239 and >	0.0235 and >
Correlation Coefficient (>0.995)	0.99768	0.9992	0.9978	1.9978
QC Report (Verified)				
Data Entry Checked by Another	NR	NR	NR	NR
All Calculation Checked	LIMS	LIMS	LIMS	LIMS
QC Limits Met			"M", "DQO"	"M", "DQO"

Notes

"*" Method blank is above the true MDL, but less than 3 times the MDL.

The instrument blanks were performed but, not reported.

	M5-703-SWB	M5-712-SWB	M5-726-SWB	M5-738-SWB
Narrative Description (Attached)				
Total Samples/Matrix	151/Waters	151/Waters	151/Waters	151/Waters
Methods/Parameters	X	X	X	X
Range of Samples analyzed	X	X	X	X
Holding Time Summary	X	X	X	X
Analytical Problems	X	X	X	X
QA/QC Acceptance Limits	X	X	X	X
Integrity of Data Quality	X	X	X	X
Deviations From SOP	X	X	X	X
Observations	X	X	X	X
Sample Management Records				
Sampling Location ID	X	X	X	X
Matrix	X	X	X	X
Preservative (Acid, Temp..)	NR	NR	NR	NR
Collection Data/Time	X	X	X	X
Laboratory ID Code	X	X	X	X
Sample Handling/Storage	X	X	X	X
Log-in Procedures	NR	NR	NR	NR
Raw Data (Attached)				
Sample Work Sheets	X	X	X	X
Sample Run Logs	X	X	X	X
Instrument Raw Data	X	X	X	X
Bench Sheets	NA	NA	NA	NA
Sample Preparation Logs	X	X	X	X
Raw Data (Verified)				
Sample ID Transferred	X	X	X	X
All Calculation Checked	X	X	X	X
Measuring Unit	ng/L	ng/L	ng/L	ng/L
PE Results (Attached)				
Organization	NR	NR	NR	NR
Performance (Pass/Fail)	NR	NR	NR	NR
Validation Criteria				
Applied Qualifiers			"M", "DQO"	"M", "DQO"

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavaliable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September (M5) 1999 Samples for Critical Parameters Analyzed by Battelle With the 10% Full QA/QC Review

Total Methylmercury in Surface Water

Laboratory Records	Sampling Station ID: M5-823-SWB	Sampling Station ID: M5-920-SWB
Data Report (Attached)		
Laboratory ID Code	1405-161	1405-68
Sampling Location ID	M5-823-SWB	M5-920-SWB
Sample Type	surface water = SW	surface water = SW
Collection Date	09/30/99	09/23/99
Digestion Date	10/18/99	10/06/99
Analysis Date	10/19/99	10/07/99
QC Batch ID	101999MEB	100799MEB
Digestion Volume	50.069	51.002
Total Volume	50.069	50.002
Sample Volume Analyzed	aliquot	aliquot
Dilution	1	1
Results	1.25	0.25
Measuring Unit	ng/L (ppt)	ng/L (ppt)
EPA Method	1631/1630	1631/1630
Analyst	Niewolny	Niewolny
Method Detection limit	0.0216	0.025
Data Report (Verified)		
Data Entry Checked by Another	NR	NR
All Calculation Checked	LIMS	LIMS
Holding Time Met	Yes	Yes
QC Report (Attached)		
Laboratory ID Code	1405-161	1405-68
QC Batch ID	101999MEB	100799MEB
Method Blanks	Good	Good
Instrument Blanks	NR	NR
Duplicates (RPD)	All Good	-32.8
Matrix Spike Recoveries (75-125)	"M"	"M"
Blank Spike/CCV Recoveries	All Good	All Good
Detection Range	0.0216 and >	0.025 and >
Correlation Coefficient (>0.995)	0.9976	0.9981
QC Report (Verified)		
Data Entry Checked by Another	NR	NR
All Calculation Checked	LIMS	LIMS
QC Limits Met	"M"	"M", "DQO"

Notes

"*" Method blank is above the true MDL, but less than 3 times the MDL.

The instrument blanks were performed but, not reported.

	M5-823-SWB	M5-920-SWB
Narrative Description (Attached)		
Total Samples/Matrix	151/Waters	151/Waters
Methods/Parameters	X	X
Range of Samples analyzed	X	X
Holding Time Summary	X	X
Analytical Problems	X	X
QA/QC Acceptance Limits	X	X
Integrity of Data Quality	X	X
Deviations From SOP	X	X
Observations	X	X
Sample Management Records		
Sampling Location ID	X	X
Matrix	X	X
Preservative (Acid, Temp..)	NR	NR
Collection Data/Time	X	X
Laboratory ID Code	X	X
Sample Handling/Storage	X	X
Log-in Procedures	NR	NR
Raw Data (Attached)		
Sample Work Sheets	X	X
Sample Run Logs	X	X
Instrument Raw Data	X	X
Bench Sheets	NA	NA
Sample Preparation Logs	X	X
Raw Data (Verified)		
Sample ID Transferred	X	X
All Calculation Checked	X	X
Measuring Unit	ng/L	ng/L
PE Results (Attached)		
Organization	NR	NR
Performance (Pass/Fail)	NR	NR
Validation Criteria		
Applied Qualifiers	"M"	"M", "DQO"

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

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"NR" Data was unavaliable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

Soil - SERC

SERC Lab / EPA REMAP results
Mineral Content and Ash Free Dry Weight

Date: 12/09/99 Data Entered by LCL 03/16/00
Sample Type: Sediment Data Entry Checked by: NJS 3-17-00

												% Inorganic Content		% Organic Content
Sample ID	Collection Date	Collection Time	Digestion Date	Hold Time	Time Since Digestion	Technician	Vial Weight (g)	Sample Weight (g)	Dried Cup + Sed (g)	Ashed Sed. (g)	Mineral Content (Fraction)	% Mineral Content	Ash Free Dry Weight	% Ash Free Dry Weight Content
M5-622-SDF	09/30/99	1125	12/09/99	28	70	CB	1.0034	0.0257	1.0192	0.0158	0.6148	61.5	0.3852	38.5
M5-623-SDF	09/30/99	915	12/09/99	28	70	CB	1	0.0247	1.0013	0.0013	0.0526	5.3	0.9474	94.7
M5-624-SDF	09/30/99	1018	12/09/99	28	70	CB	0.9994	0.025	1.0009	0.0015	0.0600	6.0	0.9400	94.0
M5-625-SDF	09/30/99	1257	12/09/99	28	70	CB	0.9969	0.0251	0.9978	0.0009	0.0359	3.6	0.9641	96.4
M5-626-SDF	09/30/99	908	12/09/99	28	70	CB	0.9896	0.0254	0.9912	0.0016	0.0630	6.3	0.9370	93.7
M5-627-SDF	09/30/99	1050	12/09/99	28	70	CB	0.9871	0.0252	0.988	0.0009	0.0357	3.6	0.9643	96.4
M5-628-SDF	09/29/99	1716	12/09/99	28	71	CB	0.9907	0.0248	0.9922	0.0015	0.0605	6.0	0.9395	94.0
M5-630-SDF	09/28/99	1520	12/09/99	28	72	CB	0.9974	0.0261	1.0003	0.0029	0.1111	11.1	0.8889	88.9
M5-631-SDF	09/29/99	1414	12/09/99	28	71	CB	0.9972	0.0247	0.9976	0.0004	0.0162	1.6	0.9838	98.4
M5-632-SDF	09/29/99	1010	12/09/99	28	71	CB	0.9953	0.0246	1.002	0.0067	0.2724	27.2	0.7276	72.8
M5-633-SDF	09/29/99	1115	12/09/99	28	71	CB	0.9941	0.0247	0.9972	0.0031	0.1255	12.6	0.8745	87.4
M5-634-SDF	09/29/99	1510	12/09/99	28	71	CB	1.0032	0.025	1.0065	0.0033	0.1320	13.2	0.8680	86.8
M5-635-SDF	09/29/99	1216	12/09/99	28	71	CB	1.0006	0.0259	1.0017	0.0011	0.0425	4.2	0.9575	95.8
M5-636-SDF	09/29/99	1405	12/09/99	28	71	CB	0.9976	0.0254	0.9994	0.0018	0.0709	7.1	0.9291	92.9
M5-637-SDF	09/29/99	1210	12/09/99	28	71	CB	0.995	0.0263	1.0009	0.0059	0.2243	22.4	0.7757	77.6
M5-638-SDF	09/29/99	1630	12/09/99	28	71	CB	0.9918	0.0253	0.9978	0.0060	0.2372	23.7	0.7628	76.3
M5-639-SDF	09/28/99	1715	12/09/99	28	72	CB	0.9997	0.0255	1.004	0.0043	0.1686	16.9	0.8314	83.1
M5-640-SDF	09/29/99	1116	12/09/99	28	71	CB	1.0028	0.0245	1.0048	0.0020	0.0816	8.2	0.9184	91.8
M5-641-SDF	09/28/99	1615	12/09/99	28	72	CB	1.0046	0.0248	1.0136	0.0090	0.3629	36.3	0.6371	63.7
M5-642-SDF	09/29/99	1011	12/09/99	28	71	CB	1.006	0.025	1.0085	0.0025	0.1000	10.0	0.9000	90.0
M5-643-SDF	09/29/99	910	12/09/99	28	71	CB	0.9976	0.0248	1.0091	0.0115	0.4637	46.4	0.5363	53.6
M5-644-SDF	09/30/99	1145	12/09/99	28	70	CB	1.0065	0.025	1.0022	-0.0043	-0.1720	-17.2	1.1720	117.2
M5-645-SDF	09/28/99	1447	12/09/99	28	72	CB	0.9986	0.0251	1.0024	0.0038	0.1514	15.1	0.8486	84.9
M5-646-SDF	09/28/99	1515	12/09/99	28	72	CB	0.9981	0.0252	1.0023	0.0042	0.1667	16.7	0.8333	83.3
M5-647-SDF	09/28/99	1102	12/09/99	28	72	CB/SLH/JL	1.0001	0.0254	1.0027	0.0026	0.1024	10.2	0.8976	89.8
M5-648-SDF	09/28/99	1300	12/09/99	28	72	CB/SLH/JL	1.0013	0.0248	1.0041	0.0028	0.1129	11.3	0.8871	88.7
M5-649-SDF	09/28/99	1620	12/09/99	28	72	CB/SLH/JL	1.0003	0.0251	1.0039	0.0036	0.1434	14.3	0.8566	85.7
M5-650-SDF	09/28/99	1158	12/09/99	28	72	CB/SLH/JL	1.003	0.0248	1.0056	0.0026	0.1048	10.5	0.8952	89.5
M5-651-SDF	09/28/99	1410	12/09/99	28	72	CB/SLH/JL	1.0073	0.0258	1.0194	0.0121	0.4690	46.9	0.5310	53.1
M5-652-SDF	09/28/99	959	12/09/99	28	72	CB/SLH/JL	1.0057	0.0256	1.0089	0.0032	0.1250	12.5	0.8750	87.5
M5-653-SDF	09/28/99	1300	12/09/99	28	72	CB/SLH/JL	1.0026	0.0252	1.0238	0.0212	0.8413	84.1	0.1587	15.9
M5-654-SDF	09/28/99	1145	12/09/99	28	72	CB/SLH/JL	1.0011	0.0243	1.0039	0.0028	0.1152	11.5	0.8848	88.5
M5-655-SDF	09/28/99	1028	12/09/99	28	72	CB/SLH/JL	0.9973	0.0259	1.003	0.0057	0.2201	22.0	0.7799	78.0
M5-656-SDF	09/28/99	900	12/09/99	28	72	CB/SLH/JL	0.9973	0.0259	1.0051	0.0078	0.3012	30.1	0.6988	69.9
M5-657-SDF	09/27/99	1751	12/09/99	28	73	CB/SLH/JL	1.0034	0.0254	1.0094	0.0060	0.2362	23.6	0.7638	76.4
M5-658-SDF	09/28/99	1722	12/09/99	28	72	CB/SLH/JL	1.0033	0.0257	1.0052	0.0019	0.0739	7.4	0.9261	92.6
M5-659-SDF	09/27/99	1205	12/09/99	28	73	CB/SLH/JL	1.0063	0.0246	1.0115	0.0052	0.2114	21.1	0.7886	78.9
M5-660-SDF	09/27/99	1450	12/09/99	28	73	CB/SLH/JL	1.0032	0.0245	1.0145	0.0113	0.4612	46.1	0.5388	53.9
M5-661-SDF	09/29/99	857	12/09/99	28	71	CB/SLH/JL	1.0034	0.0251	1.0106	0.0072	0.2869	28.7	0.7131	71.3
M5-662-SDF	09/27/99	1610	12/09/99	28	73	CB/SLH/JL	1.005	0.0248	1.0064	0.0014	0.0565	5.6	0.9435	94.4
M5-663-SDF	09/27/99	1330	12/09/99	28	73	CB/SLH/JL	1.0064	0.0251	1.0141	0.0077	0.3068	30.7	0.6932	69.3
M5-664-SDF	09/27/99	1100	12/09/99	28	73	CB/SLH/JL	1.0033	0.0248	1.0133	0.0100	0.4032	40.3	0.5968	59.7
M5-665-SDF	09/27/99	1710	12/09/99	28	73	CB/SLH/JL	1.0025	0.0252	1.0058	0.0033	0.1310	13.1	0.8690	86.9
M5-666-SDF	09/27/99	1655	12/09/99	28	73	CB/SLH/JL	1.0046	0.0251	1.0073	0.0027	0.1076	10.8	0.8924	89.2
M5-667-SDF	09/27/99	1545	12/09/99	28	73	CB/SLH/JL	1.0034	0.247	1.0066	0.0032	0.0130	1.3	0.9870	98.7
M5-668-SDF	09/27/99	1207	12/09/99	28	73	CB/SLH/JL	1.0006	0.0245	1.004	0.0034	0.1388	13.9	0.8612	86.1
M5-669-SDF	09/27/99	1000	12/09/99	28	73	CB/SLH/JL	0.9997	0.0254	1.0026	0.0029	0.1142	11.4	0.8858	88.6
M5-670-SDF	09/27/99	850	12/09/99	28	73	CB/SLH/JL	1.0051	0.0251	1.0119	0.0068	0.2709	27.1	0.7291	72.9
M5-671-SDF	09/26/99	850	12/13/99	28	78	SLH/JL	0.9971	0.0251	1.0023	0.0052	0.2072	20.7	0.7928	79.3
M5-672-SDF	09/27/99	1100	12/13/99	28	77	SLH/JL	1.0043	0.0245	1.0056	0.0013	0.0531	5.3	0.9469	94.7
M5-673-SDF	09/27/99	1400	12/13/99	28	77	SLH/JL	1.0126	0.0248	1.0293	0.0167	0.6734	67.3	0.3266	32.7
M5-674-SDF	09/26/99	1025	12/13/99	28	78	SLH/JL	1.0143	0.0255	1.0159	0.0016	0.0627	6.3	0.9373	93.7
M5-675-SDF	09/26/99	1130	12/13/99	28	78	SLH/JL	1.0121	0.0251	1.0144	0.0023	0.0916	9.2	0.9084	90.8
M5-676-SDF	09/28/99	925	12/13/99	28	76	SLH/JL	1.011	0.0256	1.0137	0.0027	0.1055	10.5	0.8945	89.5
M5-677-SDF	09/26/99	1310	12/13/99	28	78	SLH/JL	1.0056	0.0255	1.0083	0.0027	0.1059	10.6	0.8941	89.4

Sample ID	Collection Date	Collection Time	Digestion Date	Hold Time	Time Since Digestion	Technician	Vial Weight (g)	Sample Weight (g)	Dried Cup + Sed (g)	Ashed Sed. (g)	Mineral Content (Fraction)	% Mineral Content	Ash Free Dry Weight	% Ash Free Dry Weight Content
M5-678-SDF	09/26/99	1213	12/13/99	28	78	SLH/JL	1.0054	0.0252	1.0073	0.0019	0.0754	7.5	0.9246	92.5
M5-679-SDF	09/26/99	1335	12/13/99	28	78	SLH/JL	1.001	0.0248	1.0045	0.0035	0.1411	14.1	0.8589	85.9
M5-680-SDF	09/27/99	900	12/13/99	28	77	SLH/JL	1.003	0.0249	1.0057	0.0027	0.1084	10.8	0.8916	89.2
M5-681-SDF	09/26/99	1410	12/13/99	28	78	SLH/JL	0.9994	0.0255	1.004	0.0046	0.1804	18.0	0.8196	82.0
M5-682-SDF	09/26/99	1615	12/13/99	28	78	SLH/JL	1.006	0.0255	1.0122	0.0062	0.2431	24.3	0.7569	75.7
M5-683-SDF	09/26/99	850	12/13/99	28	78	SLH/JL	1.0062	0.0248	1.009	0.0028	0.1129	11.3	0.8871	88.7
M5-684-SDF	09/26/99	1530	12/13/99	28	78	SLH/JL	1.0088	0.0258	1.0137	0.0049	0.1899	19.0	0.8101	81.0
M5-685-SDF	09/26/99	1434	12/13/99	28	78	SLH/JL	1.0074	0.0249	1.0092	0.0018	0.0723	7.2	0.9277	92.8
M5-686-SDF	09/25/99	915	12/13/99	28	79	SLH/JL	1.0068	0.0257	1.0095	0.0027	0.1051	10.5	0.8949	89.5
M5-687-SDF	09/25/99	1035	12/13/99	28	79	SLH/JL	1.0087	0.0246	1.0108	0.0021	0.0854	8.5	0.9146	91.5
M5-688-SDF	09/26/99	1527	12/13/99	28	78	SLH/JL	1.0116	0.0254	1.0131	0.0015	0.0591	5.9	0.9409	94.1
M5-689-SDF	09/25/99	1205	12/13/99	28	79	SLH/JL	1.013	0.025	1.0174	0.0044	0.1760	17.6	0.8240	82.4
M5-690-SDF	09/25/99	1350	12/13/99	28	79	SLH/JL	1.0104	0.0257	1.0129	0.0025	0.0973	9.7	0.9027	90.3
M5-691-SDF	09/25/99	1554	12/13/99	28	79	SLH/JL	1.0141	0.0247	1.0156	0.0015	0.0607	6.1	0.9393	93.9
M5-692-SDF	09/25/99	1200	12/13/99	28	79	SLH/JL	1.0156	0.0247	1.0177	0.0021	0.0850	8.5	0.9150	91.5
M5-693-SDF	09/25/99	1139	12/13/99	28	79	SLH/JL	1.0204	0.0259	1.0255	0.0051	0.1969	19.7	0.8031	80.3
M5-694-SDF	09/25/99	1702	12/13/99	28	79	SLH/JL	1.0213	0.0245	1.0233	0.0020	0.0816	8.2	0.9184	91.8
M5-695-SDF	09/25/99	1400	12/13/99	28	79	SLH/JL	0.9956	0.0255	1.005	0.0094	0.3686	36.9	0.6314	63.1
M5-696-SDF	09/25/99	1510	12/13/99	28	79	SLH/JL	0.9917	0.0257	0.9951	0.0034	0.1323	13.2	0.8677	86.8
M5-697-SDF	09/26/99	1637	12/13/99	28	78	SLH/JL	0.9943	0.0245	0.9977	0.0034	0.1388	13.9	0.8612	86.1
M5-698-SDF	09/25/99	1030	12/13/99	28	79	SLH/JL	0.9993	0.0251	1.0016	0.0023	0.0916	9.2	0.9084	90.8
M5-699-SDF	09/25/99	1635	12/13/99	28	79	SLH/JL	0.9962	0.0251	0.9991	0.0029	0.1155	11.6	0.8845	88.4
M5-700-SDF	09/25/99	1750	12/13/99	28	79	SLH/JL	0.9998	0.0251	1.0044	0.0046	0.1833	18.3	0.8167	81.7
M5-701-SDF	09/26/99	1740	12/13/99	28	78	SLH/JL	1.0216	0.0254	1.0251	0.0035	0.1378	13.8	0.8622	86.2
M5-702-SDF	09/25/99	918	12/13/99	28	79	SLH/JL	1.0195	0.0254	1.0338	0.0143	0.5630	56.3	0.4370	43.7
M5-703-SDF	09/24/99	1655	12/13/99	28	80	SLH/JL	1.0196	0.0251	1.0372	0.0176	0.7012	70.1	0.2988	29.9
M5-704-SDF	09/24/99	1615	12/13/99	28	80	SLH/JL	1.0208	0.0246	1.032	0.0112	0.4553	45.5	0.5447	54.5
M5-705-SDF	09/24/99	1725	12/13/99	28	80	SLH/JL	1.0217	0.0257	1.0244	0.0027	0.1051	10.5	0.8949	89.5
M5-706-SDF	09/24/99	1555	12/13/99	28	80	SLH/JL	1.0217	0.0253	1.0386	0.0169	0.6680	66.8	0.3320	33.2
M5-707-SDF	09/24/99	1500	12/13/99	28	80	SLH/JL	1.0193	0.0246	1.0324	0.0131	0.5325	53.3	0.4675	46.7
M5-708-SDF	09/24/99	900	12/13/99	28	80	SLH/JL	1.0147	0.0253	1.0202	0.0055	0.2174	21.7	0.7826	78.3
M5-709-SDF	09/24/99	1330	12/13/99	28	80	SLH/JL	1.0139	0.0256	1.0327	0.0188	0.7344	73.4	0.2656	26.6
M5-708D-SDF	09/24/99	900	12/13/99	28	80	SLH/JL	1.0145	0.0247	1.0212	0.0067	0.2713	27.1	0.7287	72.9
M5-709D-SDF	09/24/99	1330	12/13/99	28	80	SLH/JL	1.0179	0.0254	1.0365	0.0186	0.7323	73.2	0.2677	26.8
M5-710-SDF	09/24/99	1430	12/13/99	28	80	SLH/JL	1.0077	0.0253	1.0244	0.0167	0.6601	66.0	0.3399	34.0
M5-711-SDF	09/24/99	1115	12/13/99	28	80	SLH/JL	1.0086	0.0246	1.014	0.0054	0.2195	22.0	0.7805	78.0
M5-712-SDF	09/24/99	905	12/13/99	28	80	SLH/JL	1.0158	0.025	1.0321	0.0163	0.6520	65.2	0.3480	34.8
M5-714-SDF	09/23/99	1715	12/13/99	28	81	SLH/JL	1.0136	0.0253	1.0275	0.0139	0.5494	54.9	0.4506	45.1
M5-715-SDF	09/24/99	1145	12/13/99	28	80	SLH/JL	1.0098	0.0248	1.0265	0.0167	0.6734	67.3	0.3266	32.7
M5-716-SDF	09/24/99	1310	12/13/99	28	80	SLH/JL	1.0087	0.0246	1.0167	0.0080	0.3252	32.5	0.6748	67.5
M5-718-SDF	09/24/99	1030	12/14/99	28	81	JL	1.0095	0.0248	1.0263	0.0168	0.6774	67.7	0.3226	32.3
M5-719-SDF	09/23/99	1715	12/14/99	28	82	JL	1.0134	0.0255	1.0284	0.0150	0.5882	58.8	0.4118	41.2
M5-720-SDF	09/23/99	0	12/14/99	28	82	JL	1.0152	0.0246	1.0297	0.0145	0.5894	58.9	0.4106	41.1
M5-722-SDF	09/23/99	1600	12/14/99	28	82	JL	1.0181	0.025	1.0335	0.0154	0.6160	61.6	0.3840	38.4
M5-723-SDF	09/23/99	1500	12/14/99	28	82	JL	1.0206	0.0255	1.0394	0.0188	0.7373	73.7	0.2627	26.3
M5-724-SDF	09/23/99	1442	12/14/99	28	82	JL	1.0269	0.0247	1.0323	0.0054	0.2186	21.9	0.7814	78.1
M5-725-SDF	09/23/99	1323	12/14/99	28	82	JL	1.0302	0.0251	1.0444	0.0142	0.5657	56.6	0.4343	43.4
M5-726-SDF	09/23/99	1230	12/14/99	28	82	JL	1.0304	0.0254	1.0343	0.0039	0.1535	15.4	0.8465	84.6
M5-727-SDF	09/23/99	1216	12/14/99	28	82	JL	1.0312	0.0246	1.0476	0.0164	0.6667	66.7	0.3333	33.3
M5-728-SDF	09/23/99	1350	12/14/99	28	82	JL	1.0279	0.0253	1.0439	0.0160	0.6324	63.2	0.3676	36.8
M5-729-SDF	09/23/99	1027	12/14/99	28	82	JL	1.0239	0.0247	1.0388	0.0149	0.6032	60.3	0.3968	39.7
M5-730-SDF	09/23/99	1120	12/14/99	28	82	JL	1.0229	0.0255	1.0255	0.0026	0.1020	10.2	0.8980	89.8
M5-731-SDF	09/22/99	1725	12/14/99	28	83	JL	0.9909	0.0256	1.0071	0.0162	0.6328	63.3	0.3672	36.7
M5-732-SDF	09/23/99	917	12/14/99	28	82	JL	0.9889	0.0253	0.9968	0.0079	0.3123	31.2	0.6877	68.8
M5-733-SDF	09/23/99	910	12/15/99	28	83	JL	0.9923	0.0245	1.0059	0.0136	0.5551	55.5	0.4449	44.5
M5-734-SDF	09/22/99	1540	12/14/99	28	83	JL	0.9872	0.0257	0.9953	0.0081	0.3152	31.5	0.6848	68.5
M5-735-SDF	09/22/99	1700	12/14/99	28	83	JL	0.988	0.0252	1.0057	0.0177	0.7024	70.2	0.2976	29.8
M5-738-SDF	09/22/99	1410	12/14/99	28	83	JL	1.0042	0.025	1.0171	0.0129	0.5160	51.6	0.4840	48.4
M5-740-SDF	09/22/99	1245	12/14/99	28	83	JL	1.0026	0.0251	1.019	0.0164	0.6534	65.3	0.3466	34.7
M5-741-SDF	09/22/99	1534	12/15/99	28	84	JL	0.9904	0.0248	1.0074	0.0170	0.6855	68.5	0.3145	31.5
M5-742-SDF	09/22/99	1418	12/14/99	28	83	JL	0.9919	0.0254	1.0116	0.0197	0.7756	77.6	0.2244	22.4
M5-743-SDF	09/22/99	1130	12/14/99	28	83	JL	0.9957	0.025	1.0124	0.0167	0.6680	66.8	0.3320	33.2
M5-744-SDF	09/22/99	1224	12/15/99	28	84	JL	0.987	0.0252	0.9941	0.0071	0.2817	28.2	0.7183	71.8

Sample ID	Collection Date	Collection Time	Digestion Date	Hold Time	Time Since Digestion	Technician	Vial Weight (g)	Sample Weight (g)	Dried Cup + Sed (g)	Ashed Sed. (g)	Mineral Content (Fraction)	% Mineral Content	Ash Free Dry Weight	% Ash Free Dry Weight Content
M5-745-SDF	09/22/99	1120	12/15/99	28	84	JL	0.9821	0.025	0.9981	0.0160	0.6400	64.0	0.3600	36.0
M5-746-SDF	09/22/99	948	12/14/99	28	83	JL	1.0016	0.0253	1.0163	0.0147	0.5810	58.1	0.4190	41.9
M5-747-SDF	09/22/99	942	12/14/99	28	83	JL	1.0022	0.0256	1.0226	0.0204	0.7969	79.7	0.2031	20.3
M5-823-SDF	09/30/99	0	12/14/99	28	75	JL	1.0002	0.0252	1.0023	0.0021	0.0833	8.3	0.9167	91.7
M5-828-SDF	09/29/99	0	12/15/99	28	77	JL	0.9842	0.0254	0.9856	0.0014	0.0551	5.5	0.9449	94.5
M5-838-SDF	09/29/99	0	12/14/99	28	76	JL	1.0004	0.0257	1.005	0.0046	0.1790	17.9	0.8210	82.1
M5-848-SDF	09/28/99	0	12/14/99	28	77	JL	0.9988	0.025	1.0016	0.0028	0.1120	11.2	0.8880	88.8
M5-859-SDF	09/28/99	0	12/14/99	28	77	JL	0.9994	0.0256	1.0044	0.0050	0.1953	19.5	0.8047	80.5
M5-868-SDF	09/27/99	0	12/14/99	28	78	JL	1.0013	0.0254	1.0038	0.0025	0.0984	9.8	0.9016	90.2
M5-878-SDF	09/26/99	0	12/14/99	28	79	JL	0.9835	0.0253	0.9851	0.0016	0.0632	6.3	0.9368	93.7
M5-890-SDF	09/25/99	0	12/15/99	28	81	JL	0.9859	0.0253	0.9882	0.0023	0.0909	9.1	0.9091	90.9
M5-908-SDF	09/24/99	0	12/14/99	28	81	JL	0.9869	0.0257	0.9933	0.0064	0.2490	24.9	0.7510	75.1
M5-920-SDF	09/23/99	0	12/14/99	28	82	JL	0.9891	0.0254	1.002	0.0129	0.5079	50.8	0.4921	49.2
M5-932-SDF	09/23/99	0	12/15/99	28	83	JL	0.9881	0.0255	0.9955	0.0074	0.2902	29.0	0.7098	71.0
M5-944-SDF	09/22/99	1224	12/14/99	28	83	JL	1.0031	0.0249	1.0085	0.0054	0.2169	21.7	0.7831	78.3

10 % Recalculated Results for Methylmercury in Soil Samples Analyzed by Florida International University Laboratory for the September 1999 Wet Season (M5)

Entered by mwb 4-11-00

Sampling Station ID	Data Qualifier Note	QC Batch	Wet Sample Weight (g)	Dry/Wet Weight Ratio	First Extraction Volume(ml)	Back Extraction Volume(ml)	Final Extraction Volume(ul)	Spiked Concentration (ng/g)	MeHg Peak Area	Y intercept	Slope	MeHg Concentration (ng/g)	AVE MeHg Conc. (ng/g)	%RPD	%R	AVE %R	Final Result ng/g
M5-622-SDF-A		022100a	4.495	0.021	4.2	0.6	200		7.70	0.62	2.36	13.72					
M5-622-SDF-B		022100a	4.606	0.021	4.2	0.6	200		9.92	0.62	2.36	17.24	15.48	-22.8			
M5-622-SDF-C		022100a	4.538	0.021	4.2	0.6	200	7.87	11.05	0.62	2.36	19.50			73.45		
M5-622-SDF-D		022100a	4.317	0.021	4.2	0.6	200	8.27	14.34	0.62	2.36	26.60	23.05	-30.8	113.09	93.27	16.60
Blank-1		022100a			3.8	0.8	200		0.00	0.62	2.36	0.00					
Blank-2		022100a			4.0	0.8	200		0.00	0.62	2.36	0.00					
ccv		022100a						2.50	6.880	0.62	2.36	2.92			116.61		
M5-633-SDF-A		032000b	4.537	0.123	3.4	0.6	200			0.50	2.44	0.00					
M5-633-SDF-B		032000b	4.922	0.123	3.6	0.6	200		12.76	0.50	2.44	4.00	4.00	NA			
M5-633-SDF-C	"M"	032000b	4.963	0.123	3.6	0.6	200	1.23	15.07	0.50	2.44	4.68			NA		
M5-633-SDF-D	M'	032000b	4.842	0.123	4.0	0.6	200	1.26	15.76	0.50	2.44	4.52	4.60	3.6	48.38	48.38	8.27
Blank-1		032000b			4.0	0.6	200		0.00	0.50	2.44	0.00					
Blank-2		032000b			4.0	0.6	200		0.00	0.50	2.44	0.00					
ccv		032000b						3.75	6.590	0.50	2.44	2.70			72.02		
ccv		032000b						3.75	7.740	0.50	2.44	3.17			84.59		
M5-643-SDF-A		022100b	5.477	0.250	3.2	0.6	200		14.80	0.61	3.20	1.76					
M5-643-SDF-B		022100b	4.590	0.250	3.0	0.6	200		12.96	0.61	3.20	1.96	1.86	-10.8			
M5-643-SDF-C	"M"	022100b	4.550	0.250	3.0	0.6	200	0.66	16.67	0.61	3.20	2.54			118.94		
M5-643-SDF-D	M'	022100b	4.760	0.250	3.0	0.6	200	0.63	19.58	0.61	3.20	2.86	2.70	-11.6	142.19	130.56	1.42
Blank-1		022100b			3.0	0.6	200		0.00	0.61	3.20						
Blank-2		022100b			4.0	0.6	200		0.00	0.61	3.20						
ccv		022100b						2.50	10.04	0.61	3.20	3.14			125.50		

Data Qualifiers

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

10 % Recalculated Results for Methylmercury in Soil Samples Analyzed by Florida International University Laboratory for the September 1999 Wet Season (M5)

Entered by mwb 02/02/00

Sampling Station ID	Data Qualifier Note	QC Batch	Wet Sample Weight (g)	Dry/Wet Weight Ratio	First Extraction Volume(ml)	Back Extraction Volume(ml)	Final Extraction Volume(ul)	Spiked Concentration (ng/g)	MeHg Peak Area	Y intercept	Slope	MeHg Concentration (ng/g)		%RPD	%R	AVE %R	Final Result ng/g
M5-653-SDF-A		22800	6.060	0.447	2.6	0.6	200			-0.02	5.90	0.00					
M5-653-SDF-B		22800	5.583	0.447	2.6	0.6	200		2.17	-0.02	5.90	0.09	0.09	NA			
M5-653-SDF-C		22800	6.381	0.447	2.8	0.6	200	0.26	9.87	-0.02	5.90	0.35			NA		
M5-653-SDF-D		22800	5.437	0.447	2.2	0.6	200	0.31	6.94	-0.02	5.90	0.37	0.36	-4.9	87.80	87.80	0.11
Blank-1		22800			4.0	0.6	200		0.00	-0.02	5.90	0.00					
Blank-2		22800			4.0	0.6	200		0.00	-0.02	5.90	0.00					
ccv	"DQO (NR)"	22800															
M5-663-SDF-A		022500b	5.209	0.125	3.8	0.6	200		6.77	0.13	3.02	1.51					
M5-663-SDF-B		022500b	4.609	0.125	4.0	0.6	200		5.78	0.13	3.02	1.38	1.45	8.7			
M5-663-SDF-C	"M"	022500b	5.109	0.125	4.0	0.6	200	1.17	8.33	0.13	3.02	1.80			24.75		
M5-663-SDF-D	M'	022500b	4.830	0.125	3.2	0.6	200	1.24	6.97	0.13	3.02	1.99	1.90	-10.1	48.94	36.84	3.93
Blank-1		022500b			3.8	0.6	200		0.00	0.13	3.02	0.00					
Blank-2		022500b			4.0	0.6	200		0.00	0.13	3.02	0.00					
ccv		022500b						2.50	8.480	0.13	3.02	2.81			112.32		
ccv		022500b						2.50	7.050	0.13	3.02	2.33			93.38		
M5-673-SDF-A		022100a	5.076	0.202	4.0	0.6	200		6.97	0.52	1.99	1.42					
M5-673-SDF-B		022100a	4.834	0.202	4.2	0.6	200		6.52	0.52	1.99	1.33	1.38	6.7			
M5-673-SDF-C	"M"	022100a	4.997	0.202	4.0	0.6	200	0.74	8.13	0.52	1.99	1.69			35.56		
M5-673-SDF-D	M'	022100a	5.058	0.202	3.6	0.6	200	0.73	7.41	0.52	1.99	1.69	1.69	0.0	48.74	42.15	3.27
Blank-1		022100a			4.4	0.6	200		0.00	0.52	1.99	0.00					
Blank-2		022100a			4.0	0.6	200		0.00	0.52	1.99	0.00					
ccv		022100a						2.50	6.060	0.52	1.99	3.05			121.81		
ccv	"DQO"	022100a						2.50	6.880	0.52	1.99	3.46			138.29		
M5-683-SDF-A		30300a	5.186	0.0868	4.0	0.6	200		1.54	0.45	2.66	0.54					
M5-683-SDF-B		30300a	4.518	0.0868	3.2	0.6	200		0.84	0.45	2.66	0.42	0.48	24.4			
M5-683-SDF-C	"M"	30300a	4.524	0.0868	4.0	0.6	200	1.91	7.60	0.45	2.66	3.03			130.67		
M5-683-SDF-D	M'	30300a	5.182	0.0868	4.0	0.6	200	1.67	7.58	0.45	2.66	2.64	2.84	13.8	132.95	131.81	0.36
Blank-1		30300a			3.2	0.6	200		0.00	0.45	2.66	0.00					
Blank-2		30300a			4.0	0.6	200		0.00	0.45	2.66	0.00					
ccv	"DQO"	30300a						2.50	9.310	0.45	2.66	3.50			140.00		

Data Qualifiers

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

10 % Recalculated Results for Methylmercury in Soil Samples Analyzed by Florida International University Laboratory for the September 1999 Wet Season (M5)

Entered by mwb 4-11-00

Sampling Station ID	Data Qualifier Note	QC Batch	Wet Sample Weight (g)	Dry/Wet Weight Ratio	First Extraction Volume(ml)	Back Extraction Volume(ml)	Final Extraction Volume(ul)	Spiked Concentration (ng/g)	MeHg Peak Area	Y intercept	Slope	MeHg Concentration (ng/g)	AVE MeHg Conc. (ng/g)	%RPD	%R	AVE %R	Final Result ng/g
M5-693-SDF-A		30300b	5.279	0.0876	3.2	0.6	200		0.52	1.39	2.59	0.23					
M5-693-SDF-B	"DQO"	30300b	4.996	0.0876	3.0	0.6	200		0.74	1.39	2.59	0.36	0.29	-46.4			
M5-693-SDF-C	"M"	30300b	5.201	0.0876	3.2	0.6	200	1.65	8.71	1.39	2.59	3.84			219.29		
M5-693-SDF-D	"M"	30300b	5.347	0.0876	3.2	0.6	200	1.60	8.39	1.39	2.59	3.60	3.72	6.5	202.46	210.87	0.14
Blank-1		30300b			3.2	0.6	200		0.00	1.39	2.59	0.00					
Blank-2		30300b			4.0	0.6	200		0.00	1.39	2.59	0.00					
ccv	"DQO"	30300b						2.50	9.400	1.39	2.59	3.63			145.17		
M5-703-SDF-A		030700a	5.368	0.213	2.0	0.6	200		1.50	0.21	2.66	0.41					
M5-703-SDF-B	"DQO"	030700a	5.250	0.213	2.0	0.6	200		1.02	0.21	2.66	0.29	0.35	35.9			
M5-703-SDF-C		030700a	5.548	0.213	2.0	0.6	200	0.63	4.40	0.21	2.66	1.17			119.92		
M5-703-SDF-D	"M"	030700a	5.521	0.213	1.6	0.6	200	0.64	4.05	0.21	2.66	1.35	1.26	-14.5	166.08	143.00	0.24
Blank-1		030700a			4.0	0.6	200		0.00	0.50	2.44	0.00					
Blank-2		030700a			4.0	0.6	200		0.00	0.50	2.44	0.00					
ccv	"DQO (NR)"	030700a															
ccv	"DQO (NR)"	030700a															
M5-714-SDF-A		032300a	4.908	0.186	3.4	0.6	200		1.36	-0.07	1.71	0.43					
M5-714-SDF-B		032300a	4.629	0.186	3.4	0.6	200		1.47	-0.07	1.71	0.49	0.46	-13.6			
M5-714-SDF-C	"M"	032300a	4.865	0.186	3.4	0.6	200	0.83	5.17	-0.07	1.71	1.64			145.88		
M5-714-SDF-D	"M"	032300a	5.835	0.186	3.4	0.6	200	0.69	5.70	-0.07	1.71	1.51	1.57	8.4	147.26	146.57	0.31
Blank-1		032300a			4.0	0.8	200		0.00	-0.07	1.71						
Blank-2		032300a			4.0	0.8	200		0.00	-0.07	1.71						
ccv	"DQO (NR)"	032300a															

Data Qualifiers

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

10 % Recalculated Results for Methylmercury in Soil Samples Analyzed by Florida International University Laboratory for the September 1999 Wet Season (M5)

Entered by mwb 02/02/00

Sampling Station ID	Data Qualifier Note	QC Batch	Wet Sample Weight (g)	Dry/Wet Weight Ratio	First Extraction Volume(ml)	Back Extraction Volume(ml)	Final Extraction Volume(ul)	Spiked Concentration (ng/g)	MeHg Peak Area	Y intercept	Slope	MeHg Concentration (ng/g)		%RPD	%R	AVE %R	Final Result ng/g
M5-726-SDF-A		30800	5.183	0.0819	4.2	0.6	200		2.36	0.11	2.24	0.98					
M5-726-SDF-B		30800	4.935	0.0819	4.0	0.6	200		1.93	0.11	2.24	0.89	0.94	10.3			
M5-726-SDF-C	"M"	30800	5.010	0.0819	4.2	0.6	200	1.83	7.88	0.11	2.24	3.40			132.09		
M5-726-SDF-D	"M"	30800	4.804	0.0819	4.2	0.6	200	1.91	7.45	0.11	2.24	3.35	3.38	1.4	129.12	130.61	0.72
Blank-1		30800			4.0	0.6	200		0.00	0.11	2.24	0.00					
Blank-2		30800			4.0	0.6	200		0.00	0.11	2.24	0.00					
ccv		30800						2.50	6.210	0.13	3.02	2.06			82.25		
ccv		30800						2.50	6.240	0.13	3.02	2.07			82.65		
M5-738-SDF-A		022500b	5.101	0.2806	3.6	0.6	200			0.27	2.04	0.00					
M5-738-SDF-B		022500b	5.139	0.2806	4.2	0.6	200		2.08	0.27	2.04	0.28	0.28	"NA"			
M5-738-SDF-C		022500b	5.038	0.2806	4.2	0.6	200	0.53	6.01	0.27	2.04	0.83			"NA"		
M5-738-SDF-D		022500b	5.370	0.2806	4.2	0.6	200	0.50	6.59	0.27	2.04	0.85	0.84	-2.8	114.03	114.03	0.25
Blank-1		022500b			3.8	0.6	200		0.00	0.27	2.04	0.00					
Blank-2		022500b			4.0	0.6	200		0.00	0.27	2.04	0.00					
ccv	"DQO"	022500b						2.50	8.480	0.27	2.04	4.16			166.27		
ccv	"DQO"	022500b						2.50	7.050	0.27	2.04	3.46			138.24		
M5-828-SDF-A		040400b	5.022	0.0453	4.0	0.6	200		5.66	0.39	2.21	4.69					
M5-828-SDF-B		040400b															
M5-828-SDF-C	"M"	040400b	5.030	0.0453	4.0	0.6	200	3.29	8.36	0.39	2.21	6.92			67.68		6.93
M5-828-SDF-D	"M"	040400b															
Blank-1		040400b			4.0	0.6	200		0.00	0.52	1.99	0.00					
Blank-2		040400b			4.0	0.6	200		0.00	0.52	1.99	0.00					
ccv		040400b						2.50	6.310	0.52	1.99	3.17			126.83		
ccv		040400b						2.50	5.700	0.52	1.99	2.86			114.57		
M5-944-SDF-A		31000	4.386	0.0785	4.0	0.6	200		1.88	0.61	1.94	1.17					
M5-944-SDF-B	"DQO"	31000	4.482	0.0785	4.0	0.6	200		0.40	0.61	1.94	0.24	0.71	131.1			
M5-944-SDF-C	"M"	31000	5.594	0.0785	4.0	0.6	200	1.71	5.69	0.61	1.94	2.78			94.16		
M5-944-SDF-D	"M"	31000	5.626	0.0785	4.0	0.6	200	1.70	6.85	0.61	1.94	3.33	3.06	-17.9	181.59	137.88	0.51
Blank-1		31000			3.2	0.6	200		0.00	0.61	1.94	0.00					
Blank-2		31000			4.0	0.6	200		0.00	0.61	1.94	0.00					
ccv	"DQO"	31000						2.50	9.310	0.61	1.94	4.80			191.96		

Data Qualifiers

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

SERC Mercury Lab / EPA REMAP results
Methyl Mercury Analysis

: Analysis not performed
: Analysis not required
: Averaged Results

Data Entered by: MWB 3-30-00
Data Entry Checked by: NJS 4-7-00

Sampling Station ID	Matrix	Analysis Method	Collection Date	Collection Time	Digestion Date	Run Date	Holding Time (Days)	Time Elapsed From Dig	MeHg Units (ng/g)	QA/QC Batch ID	QA Data % R	QA Data %RPD	QA Data Matrix %R	Notes
M5-622-SDF	SD		09/30/99	1125	02/21/00	02/21/00	28	144	16.51	022100a		22.8	93.6	
M5-623-SDF	SD		09/30/99	915	02/21/00	02/21/00	28	144	1.46	022100a		51.7	157.6	
M5-624-SDF	SD		09/30/99	1018	02/21/00	02/21/00	28	144	7.11	022100a		6.4	115.1	
M5-625-SDF	SD		09/30/99	1257	02/21/00	02/21/00	28	144	0.33	022100a		40.0	167.7	
M5-626-SDF	SD		09/30/99	908	02/21/00	02/21/00	28	144	12.80	022100a		21.0	102.7	
M5-627-SDF	SD		09/30/99	1050	02/21/00	02/21/00	28	144	2.46	022100a		27.2	132.5	
M5-628-SDF	SD		09/29/99	1716	02/21/00	02/21/00	28	145	10.73	022100a		10.6	76.9	
M5-628-SDF	SD		09/29/99	1716	04/04/00	04/04/00	28	188	8.81	040400b		NA	68.36	
QA-630-SDF	SD		09/29/99				28		0.39	31500		2.4	107.6	
M5-631-SDF	SD		09/29/99	1414	02/21/00	02/21/00	28	145	0.73	022100a		38.5	131.6	
M5-632-SDF	SD		09/29/99	1010	02/21/00	02/21/00	28	145	41.75	022100a		4.4	67.87	
M5-633-SDF	SD		09/29/99	1115	03/20/00	03/20/00	28	173	8.20	032000b		NA	48.7	
QA-634-SDF	SD		09/29/99				28		1.36	31500		29.1	116.4	
M5-635-SDF	SD		09/29/99	1216	02/21/00	02/21/00	28	145	8.57	022100b		5.1	79.7	
QA-636-SDF	SD		09/29/99				28		0.00	31500		0.0	115.2	
M5-637-SDF	SD		09/29/99	1210	02/21/00	02/21/00	28	145	1.71	022100b		1.7	102.1	
M5-638-SDF	SD		09/29/99	1630	04/04/00	04/04/00	28	188	19.72	040400b		NA	71.61	
M5-639-SDF	SD		09/28/99	1715	02/21/00	02/21/00	28	146	4.30	022100b		2.5	84.1	
M5-640-SDF	SD		09/29/99	1116	02/21/00	02/21/00	28	145	7.17	022100b		12.6	115.2	
M5-641-SDF	SD		09/28/99	1615	02/21/00	02/21/00	28	146	6.45	022100b		7.5	47.31	
M5-642-SDF	SD		09/29/99	1011	02/21/00	02/21/00	28	145	0.23	022100b		NA	140.8	
M5-643-SDF	SD		09/29/99	910	02/21/00	02/21/00	28	145	1.43	022100b		10.8	130.2	
M5-644-SDF	SD		09/30/99	1145	02/21/00	02/21/00	28	144	24.05	022100b		0.22	38.01	
QA-645-SDF	SD		09/28/99				28		0.00	31500		0.0	105.2	
M5-646-SDF	SD		09/28/99	1515	02/25/00	02/25/00	28	150	4.10	022500b		9.6	144.3	
M5-647-SDF	SD		09/28/99	1102	03/20/00	03/20/00	28	174	3.73	032000b		36.0	86.45	
M5-648-SDF	SD		09/28/99	1300	02/28/00	02/28/00	28	153	3.1	22800		31	122.2	
M5-649-SDF	SD		09/28/99	1620	03/20/00	03/20/00	28	174	6.9	032000b		15.5	108.89	
M5-650-SDF	SD		09/28/99	1158	02/28/00	02/28/00	28	153	0.86	22800		NA	204.5	
QA-651-SDF	SD		09/28/99				28		1.16	31500		28.0	86.2	
QA-652-SDF	SD		09/28/99				28		2.67	31500		0.9	85	
M5-653-SDF	SD		09/28/99	1300	02/28/00	02/28/00	28	153	0.10	22800		NA	93.65	
M5-654-SDF	SD		09/28/99	1145	02/28/00	02/28/00	28	153	4.21	22800		7.9	117.9	
M5-655-SDF	SD		09/28/99	1028	02/29/00	02/29/00	28	154	1.58	22900		20.7	57.8	
M5-656-SDF	SD		09/28/99	900	02/29/00	02/29/00	28	154	2.73	22900		17.5	97.9	
M5-657-SDF	SD		09/27/99	1751	03/20/00	03/20/00	28	175	3.63	032000b		5.2	84.64	
M5-658-SDF	SD		09/28/99	1722	02/29/00	02/29/00	28	154	0.00	22900		0.0	126.19	
M5-659-SDF	SD		09/27/99	1205	02/29/00	02/29/00	28	155	4.15	22900		6.1	51.67	
M5-660-SDF	SD		09/27/99	1450	02/29/00	02/29/00	28	155	1.67	22900		0.5	121.3	
M5-661-SDF	SD		09/29/99	857	02/29/00	02/29/00	28	153	0.23	22900		24.1	127.4	
M5-662-SDF	SD		09/27/99	1610	02/29/00	02/29/00	28	155	0.15	22900		5.0	134.1	
M5-663-SDF	SD		09/27/99	1330	02/29/00	02/29/00	28	155	3.90	22900		9.0	37.1	
M5-664-SDF	SD		09/27/99	1100	02/29/00	02/29/00	28	155	5.52	22900		20.0	30.9	
M5-665-SDF	SD		09/27/99	1710	03/02/00	03/02/00	28	157	0.27	30200		38.6	160.5	
M5-666-SDF	SD		09/27/99	1655	03/02/00	03/02/00	28	157	3.77	30200		16.0	74.7	
M5-667-SDF	SD		09/27/99	1545	03/02/00	03/02/00	28	157	4.37	30200		20.8	59.6	
M5-668-SDF	SD		09/27/99	1207	03/02/00	03/02/00	28	157	0.57	30200		35.6	127.8	
M5-669-SDF	SD		09/27/99	1000	03/02/00	03/02/00	28	157	0.39	30200		7.6	135.8	
M5-670-SDF	SD		09/27/99	850	04/04/00	04/04/00	28	190	0.00	040400b		NA	87.71	
QA-671-SDF	SD		09/26/99				28		2.42	31500		51.6	101.5	
M5-672-SDF	SD		09/27/99	1100	03/02/00	03/02/00	28	157	2.55	30200		14.8	90.4	
M5-673-SDF	SD		09/27/99	1400	03/02/00	03/02/00	28	157	3.29	30200		6.5	41.9	
M5-674-SDF	SD		09/26/99	1025	03/02/00	03/02/00	28	158	0.00	30200		0.0	136.01	
M5-675-SDF	SD		09/26/99	1130	03/02/00	03/02/00	28	158	5.50	30200		17.1	79.7	
M5-676-SDF	SD		09/28/99	925	03/02/00	03/02/00	28	156	0.34	30200		34.6	155.5	
M5-677-SDF	SD		09/26/99	1310	03/20/00	03/20/00	28	176	4.16	032000b		35.7	110.95	
M5-678-SDF	SD		09/26/99	1213	03/03/00	03/03/00	28	159	0.40	30300a		48.9	116.8	
M5-679-SDF	SD		09/26/99	1335	03/03/00	03/03/00	28	159	0.16	30300a		NA	176.7	
M5-680-SDF	SD		09/27/99	900	03/03/00	03/03/00	28	158	0.81	30300a		44.6	125.2	
M5-681-SDF	SD		09/26/99	1410	03/03/00	03/03/00	28	159	0.59	30300a		NA	54.6	
QA-682-SDF	SD				03/24/00	03/24/00	28		0.23	032400a		NA	78.1	
M5-683-SDF	SD		09/26/99	850	03/03/00	03/03/00	28	159	0.36	30300a		25.0	132.1	
M5-684-SDF	SD		09/26/99	1530	03/03/00	03/03/00	28	159	0.32	30300a		0.0	126.1	
M5-685-SDF	SD		09/26/99	1434	03/03/00	03/03/00	28	159	0.25	30300a		17.4	91.3	
M5-686-SDF	SD		09/25/99	915	03/03/00	03/03/00	28	160	0.00	30300a		0.0	141	
M5-687-SDF	SD		09/25/99	1035	03/03/00	03/03/00	28	160	0.67	30300a		4.7	129.4	
M5-688-SDF	SD		09/26/99	1527	03/03/00	03/03/00	28	159	0.94	30300b		NA	129.2	
M5-689-SDF	SD		09/25/99	1205	03/03/00	03/03/00	28	160	0.33	30300b		0.0	170.7	
M5-690-SDF	SD		09/25/99	1350	03/03/00	03/03/00	28	160	0.11	30300b		NA	141	
M5-691-SDF	SD		09/25/99	1554	03/03/00	03/03/00	28	160	0.52	30300b		NA	167.7	

M5-692-SDF	SD		09/25/99	1200	03/03/00	03/03/00	28	160	1.03	30300b		7.6	139.96	
M5-693-SDF	SD		09/25/99	1139	03/03/00	03/03/00	28	160	0.14	30300b		44.8	211.01	
M5-694-SDF	SD		09/25/99	1702	03/03/00	03/03/00	28	160	0.57	30300b		20.0	130.5	
M5-695-SDF	SD		09/25/99	1400	03/03/00	03/03/00	28	160	0.26	30300b		23.1	152.6	
QA-696-SDF	SD		09/25/99				28		0.44	31500		64.1	88.2	
M5-697-SDF	SD		09/26/99	1637	03/03/00	03/03/00	28	159	0.27	30300b		51.3	145.04	
QA-698-SDF	SD		09/25/99				28		1.13	31500		11.9	112	
M5-699-SDF	SD		09/25/99	1635	03/03/00	03/03/00	28	160	0.55	30300b		3.6	150.1	
M5-700-SDF	SD		09/25/99	1750	03/07/00	03/07/00	28	164	0.36	030700a		3.8	145.3	
M5-701-SDF	SD		09/26/99	1740	03/07/00	03/07/00	28	163	0.08	030700a		NA	154.4	
M5-702-SDF	SD		09/25/99	918	03/07/00	03/07/00	28	164	0.09	030700a		25.0	168.5	
M5-703-SDF	SD		09/24/99	1655	03/07/00	03/07/00	28	165	0.24	030700a		34.3	145.4	
M5-704-SDF	SD		09/24/99	1615	03/07/00	03/07/00	28	165	0.21	030700a		19.4	142.8	
M5-705-SDF	SD		09/24/99	1725	03/07/00	03/07/00	28	165	0.84	030700a		7.1	151.97	
M5-706-SDF	SD		09/24/99	1555	03/07/00	03/07/00	28	165	0.06	030700a		NA	164.5	
M5-707-SDF	SD		09/24/99	1500	03/20/00	03/20/00	28	178	0.67	032000b		11.9	98.98	
M5-708-SDF	SD		09/24/99	900	03/07/00	03/07/00	28	165	0.20	030700a		33.3	165.4	
M5-709-SDF	SD		09/24/99	1330	03/07/00	03/07/00	28	165	0.08	030700a		66.7	147.3	
QA-710-SDF	SD				03/24/00	03/24/00	28		0.15	032400a		13.3	101.5	
M5-711-SDF	SD		09/24/99	1115	03/07/00	03/07/00	28	165	0.00	030700a		0.0	47	
M5-712-SDF	SD		09/24/99	905	03/23/00	03/23/00	28	181	0.00	032300a		0.0	162.2	
M5-714-SDF	SD		09/23/99	1715	03/23/00	03/23/00	28	182	0.31	032300a		13.0	146.7	
M5-715-SDF	SD		09/24/99	1145	03/07/00	03/07/00	28	165	0.13	030700b		NA	91.95	
M5-716-SDF	SD		09/24/99	1310	03/23/00	03/23/00	28	181	0.41	032300a		17.2	17.2	
M5-718-SDF	SD		09/24/99	1030	03/07/00	03/07/00	28	165	0.12	030700b		41.7	93.7	
QA-719-SDF	SD				03/24/00	03/24/00	28		0.38	032400a		13.5	99.15	
M5-720-SDF	SD		09/23/99	0	03/07/00	03/07/00	28	166	0.00	030700b		0.0	95.02	
M5-722-SDF	SD		09/23/99	1600	03/07/00	03/07/00	28	166	0.00	030700b		0.0	100.1	
M5-723-SDF	SD		09/23/99	1500	03/07/00	03/07/00	28	166	0.00	030700b		0.0	103.4	
M5-724-SDF	SD		09/23/99	1442	03/07/00	03/07/00	28	166	0.57	030700b		15.4	114.3	
M5-725-SDF	SD		09/23/99	1323	03/08/00	03/08/00	28	167	0.06	30800		11.8	139.4	
M5-726-SDF	SD		09/23/99	1230	03/08/00	03/08/00	28	167	0.72	30800		10.6	130.9	
M5-727-SDF	SD		09/23/99	1216	03/08/00	03/08/00	28	167	0.09	30800		60.0	114.4	
M5-728-SDF	SD		09/23/99	1350	03/08/00	03/08/00	28	167	0.16	30800		33.3	116.2	
M5-729-SDF	SD		09/23/99	1027	03/08/00	03/08/00	28	167	0.19	30800		NA	102.3	
M5-730-SDF	SD		09/23/99	1120	03/08/00	03/08/00	28	167	2.31	30800		3.9	122.5	
M5-731-SDF	SD		09/22/99	1725	03/23/00	03/23/00	28	183	0.00	032300a		0.0	109.8	
M5-732-SDF	SD		09/23/99	917	03/08/00	03/08/00	28	167	0.58	30800		10.5	130.2	
M5-733-SDF	SD		09/23/99	910	03/08/00	03/08/00	28	167	0.17	30800		11.1	105.1	
M5-734-SDF	SD		09/22/99	1540	03/08/00	03/08/00	28	168	0.70	30800		NA	130.2	
M5-735-SDF	SD		09/22/99	1700	03/09/00	03/09/00	28	169	0.13	30900		0.0	80.3	
M5-738-SDF	SD		09/22/99	1410	03/09/00	03/09/00	28	169	0.26	30900		NA	108.8	
M5-740-SDF	SD		09/22/99	1245	03/09/00	03/09/00	28	169	0.10	30900		0.0	70.5	
M5-741-SDF	SD		09/22/99	1534	03/09/00	03/09/00	28	169	0.00	30900		0.0	10705	
M5-742-SDF	SD		09/22/99	1418	03/09/00	03/09/00	28	169	0.12	30900		18.2	89.7	
M5-743-SDF	SD		09/22/99	1130	03/09/00	03/09/00	28	169	0.11	30900		13.3	64.7	
M5-744-SDF	SD		09/22/99	1224	03/09/00	03/09/00	28	169	0.66	30900		8.2	111.1	
M5-745-SDF	SD		09/22/99	1120	03/09/00	03/09/00	28	169	0.28	30900		4.9	73.2	
M5-746-SDF	SD		09/22/99	948	03/09/00	03/09/00	28	169	0.12	30900		13.3	63.2	
M5-747-SDF	SD		09/22/99	942	03/09/00	03/09/00	28	169	0.00	30900		0.0	71.77	
M5-823-SDF	SD		09/30/99	0	02/21/00	02/21/00	28	144	0.88	31000		26.2	164	
M5-828-SDF	SD		09/28/99		04/04/00	04/04/00	28	189	6.95	040400b		NA	67.45	
M5-838-SDF	SD		09/29/99	0	02/21/00	02/21/00	28	145	4.65	31000		16.1	182.8	
M5-848-SDF	SD		09/28/99	0	02/21/00	02/21/00	28	146	7.34	31000		3.4	92.4	
M5-859-SDF	SD		09/27/99	0	02/21/00	02/21/00	28	147	7.40	31000		9.8	42.6	
M5-868-SDF	SD		09/27/99	0	03/10/00	03/10/00	28	165	0.18	31000		25.0	131.5	
M5-878-SDF	SD		09/26/99	0	03/10/00	03/10/00	28	166	0.71	31000		26.5	117.3	
M5-890-SDF	SD		09/25/99	0	03/10/00	03/10/00	28	167	0.23	31000		34.4	136.6	
M5-908-SDF	SD		09/24/99	0	03/10/00	03/10/00	28	168	0.27	31000		50.0	109.4	
M5-920-SDF	SD		09/23/99	0	03/10/00	03/10/00	28	169	0.00	31000		0.0	92.8	
M5-932-SDF	SD		09/23/99	0	03/10/00	03/10/00	28	169	0.49	31000		9.4	131.3	
M5-944-SDF	SD		09/22/99	1224	03/10/00	03/10/00	28	170	0.51	31000		132.4	138.3	
M5-639-FCF	FC		09/28/99		03/23/00	03/23/00	28	177	38.16	32300b		5.2	91.8	
M5-640-FCF	FC		09/29/99		03/23/00	03/23/00	28	176	0.00	32300b		0.0	156.2	
M5-656-FCF	FC		09/28/99		03/23/00	03/23/00	28	177	0.00	32300b		0.0	157.39	
M5-666-FCF	FC		09/27/99		03/23/00	03/23/00	28	178	9.53	32300b		NA	171.72	
M5-681-FCF	FC		09/26/99		03/23/00	03/23/00	28	179	5.87	32300b		43.9	95.59	
M5-683-FCF	FC		09/26/99	850	02/25/00	02/25/00	28	152	3.38	022500a		14.3	95	
M5-698-FCF	FC				03/23/00	03/23/00	28	36608	0.00	32300b		0.0	106.84	
M5-699-FCF	FC		09/25/99		03/23/00	03/23/00	28	180	2.34	32300b		24.1	113.87	
M5-700-FCF	FC		09/25/99		03/23/00	03/23/00	28	180	4.35	32300b		NA	79.79	
M5-712-FCF	FC		09/24/99	905	02/25/00	02/25/00	28	154	0.58	022500a		37.5	96.9	
M5-726-FCF	FC		09/23/99	1230	02/25/00	02/25/00	28	155	0.79	022500a		24.3	131.53	
M5-729-PSF	PS		09/23/99		03/30/00	03/30/00	28	189	0.00	33000		NA	93.5	
M5-731-PSF	PS		09/22/99		03/30/00	03/30/00	28	190	0.00	33000		NA	108.8	

M5-735-PSF	PS		09/22/99		03/30/00	03/30/00	28	190	0.00	33000		NA	68.65	
M5-740-PSF	PS		09/22/99		03/30/00	03/30/00	28	190	0.00	33000		NA	84.17	
M5-741-PSF	PS		09/22/99		03/30/00	03/30/00	28	190	0.00	33000		NA	72.03	
M5-742-PSF	PS		09/22/99		03/30/00	03/30/00	28	190	0.00	33000		NA	85.07	
M5-743-PSF	PS		09/22/99		03/30/00	03/30/00	28	190	0.07	33000		NA	92.15	
M5-744-PSF	PS		09/22/99		04/04/00	04/04/00	28	195	0.70	040400a		NA	170.5	
M5-745-PSF	PS		09/22/99		04/04/00	04/04/00	28	195	0.38	040400a		NA	123	
M5-746-PSF	PS		09/22/99		03/30/00	03/30/00	28	190	0.00	33000		NA	72.86	
M5-747-PSF	PS		09/22/99	942	02/21/00	02/21/00	28	152	0.15	022100a		33.3	155.9	
M5-747-PSF	PS		09/22/99		04/04/00	04/04/00	28	195	0.85	040400a		NA	64.41	

10 % Recalculated Results for Total Phosphorus in Soil/Sediment
Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by MWB 4-7-00 Checked by NJS 4-14-00

Sample	QA/QC Batch	Qualifier Note	Dilution Factor	Weight grams	Peak Height um	Slope um	Sample Results ug/g (ppm)	Detection Limit ug/g (ppm)
M5-622-SDF	EPA1124C		1	0.0251	5174	2664.3945	77.4	0.06
M5-643-SDF	"		1	0.0250	19003	2664.3945	285.3	0.06
M5-663-SDF	"		1	0.0249	9844	2664.3945	148.4	0.06

Sample	QA/QC Batch	Qualifier Note	Dilution Factor	Weight grams	Peak Height um	Slope um	Sample Results ug/g (ppm)	Detection Limit*3 ug/g (ppm)	SPK CONC ug/g (ppm)	R%	RPD
Citrus Leaves	EPA1124C		1	0.0255	74272	2664.3945	1093.2	0.18	1300	84.09	
Citrus Leaves	"		1	0.0254	77618	2664.3945	1146.9	0.18	1300	88.22	-4.799
CCV	"		1		58953	2664.3945	22.1	0.18	23.23	95.25	
CCV	"		1		60170	2664.3945	22.6	0.18	23.23	97.21	
CCV	"		1		61820	2664.3945	23.2	0.18	23.23	99.88	
CCV	"		1		62375	2664.3945	23.4	0.18	23.23	100.78	
CCV	"		1		63064	2664.3945	23.7	0.18	23.23	101.89	
M5-636-SDF	"		1	0.0248	14348	2664.3945	217.1	0.18			
M5-636-SDF-D	"		1	0.0248	13873	2664.3945	210.0	0.18			3.366
M5-658-SDF	"		1	0.0256	18825	2664.3945	276.0	0.18			
M5-658-SDF-D	"		1	0.0250	17280	2664.3945	259.4	0.18			6.190
Blanks	"	"B (NR)"									

Sample	QA/QC Batch	Qualifier Note	Dilution Factor	Weight grams	Peak Height um	Slope um	Sample Results ug/g (ppm)	Detection Limit ug/g (ppm)
M5-633-SDF	EPAM5-C		1	0.0248	28422	2775.5208	412.9	0.06
M5-653-SDF	"		1	0.0248	8898	2775.5208	129.3	0.06
M5-738-SDF	"		1	0.0249	24785	2775.5208	358.6	0.06

Sample	QA/QC Batch	Qualifier Note	Dilution Factor	Weight grams	Peak Height um	Slope um	Sample Results ug/g (ppm)	Detection Limit*3 ug/g (ppm)	SPK CONC ug/g (ppm)	R%	RPD
Citrus Leaves	EPAM5-C		1	0.0246	85425	2775.5208	1251.1	0.18	1300	96.24	
Citrus Leaves			1	0.0245	78812	2775.5208	1159.0	0.18	1300	89.15	7.646
CCV	"		1		57788	2775.5208	20.8	0.18	23.23	89.63	
CCV	"		1		63323	2775.5208	22.8	0.18	23.23	98.21	-9.140
CCV	"		1		64574	2775.5208	23.3	0.18	23.23	100.15	
CCV	"		1		65826	2775.5208	23.7	0.18	23.23	102.09	-1.920
M5-647-SDF	"		1	0.0248	26200	2775.5208	380.6	0.18			
M5-647-SDF-D	"		1	0.0248	25934	2775.5208	376.8	0.18			1.020
M5-735-SDF	"		1	0.0252	5318	2775.5208	76.0	0.18			
M5-735-SDF-D	"		1	0.0252	4525	2775.5208	64.7	0.18			16.113
Blanks	"	"B (NR)"									

"B" Analyte concentration in the associated blank was >3 times the MDL.

"NR" Data was unavailable for review.

10 % Recalculated Results for Total Phosphorus in Soil/Sediment
Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by MWB 4-7-00 Checked by NJS 4-14-00

Sample	QA/QC Batch	Qualifier Note	Dilution Factor	Weight grams	Peak Height um	Slope um	Sample Results ug/g (ppm)	Detection Limit ug/g (ppm)
M5-673-SDF	EPAM5-D		1	0.0253	8544	2845.8136	118.7	0.06
M5-683-SDF	"		1	0.0254	20297	2845.8136	280.8	0.06

Sample	QA/QC Batch	Qualifier Note	Dilution Factor	Weight grams	Peak Height um	Slope um	Sample Results ug/g (ppm)	Detection Limit*3 ug/g (ppm)	SPK CONC ug/g (ppm)	R%	RPD
Citrus Leaves	EPAM5-D		1	0.0246	88147	2845.8136	1259.1	0.18	1300	96.86	
Citrus Leaves	"		1	0.0245	82242	2845.8136	1179.6	0.18	1300	90.74	6.5
CCV	"		1		62262	2845.8136	21.9	0.18	23.23	94.18	
CCV	"		1		65420	2845.8136	23.0	0.18	23.23	98.96	
CCV	"		1		66646	2845.8136	23.4	0.18	23.23	100.81	
CCV	"		1		64271	2845.8136	22.6	0.18	23.23	97.22	
M5-661-SDF	"		1	0.0245	10723	2845.8136	153.8	0.18			
M5-661-SDF-D	"		1	0.0245	10589	2845.8136	151.9	0.18			1.3
M5-676-SDF	"		1	0.0248	17309	2845.8136	245.3	0.18			
M5-676-SDF-D	"		1	0.0248	21182	2845.8136	300.1	0.18			-20.1
M5-685-SDF	"		1	0.0245	25317	2845.8136	363.1	0.18			
M5-685-SDF-D	"		1	0.0247	23903	2845.8136	340.1	0.18			6.6
Blanks	"	"B (NR)"									

"B" Analyte concentration in the associated blank was >3 times the MDL.

"NR" Data was unavailable for review.

10 % Recalculated Results for Total Phosphorus in Soil/Sediment
Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by MWB 4-10-00 Checked by NJS 4-14-00

Sample	QA/QC Batch	Qualifier Note	Dilution Factor	Weight grams	Peak Height um	Slope um	Sample Results ug/g (ppm)	Detection Limit ug/g (ppm)
M5-693-SDF	EPAM5-A		1	0.0249	12431	2680.2162	186.3	0.06
M5-703-SDF	"		1	0.0245	7407	2680.2162	112.8	0.06
M5-714-SDF	"		1	0.0251	28159	2680.2162	408.6	0.06
M5-726-SDF	"		1	0.0245	15762	2680.2162	240.0	0.06
M5-828-SDF	"		1	0.0252	12391	2680.2162	183.5	0.06
M5-944-SDF	"		1	0.0254	18729	2680.2162	275.1	0.06

Sample	QA/QC Batch	Qualifier Note	Dilution Factor	Weight grams	Peak Height um	Slope um	Sample Results ug/g (ppm)	Detection Limit*3 ug/g (ppm)	SPK CONC ug/g (ppm)	R%	RPD
Citrus Leaves	EPAM5-A		1	0.0246	73703	2680.2162	1117.8	0.18	1300	85.99	
Citrus Leaves	"		1	0.0245	67110	2680.2162	1022.0	0.18	1300	78.62	8.958
CCV	"		1		54639	2680.2162	20.4	0.18	23.23	87.76	
CCV	"		1		58541	2680.2162	21.8	0.18	23.23	94.02	
CCV	"		1		59855	2680.2162	22.3	0.18	23.23	96.13	
CCV	"		1		60497	2680.2162	22.6	0.18	23.23	97.17	
CCV	"		1		60628	2680.2162	22.6	0.18	23.23	97.38	
CCV	"		1		57545	2680.2162	21.5	0.18	23.23	92.42	
CCV	"		1		65184	2680.2162	24.3	0.18	23.23	104.69	
CCV	"		1		64668	2680.2162	24.1	0.18	23.23	103.87	
M5-868-SDF	"		1	0.0246	36375	2680.2162	551.7	0.18			
M5-868-SDF-D	"		1	0.0246	37471	2680.2162	568.3	0.18			-2.968
M5-702-SDF	"		1	0.025	10068	2680.2162	150.3	0.18			
M5-702-SDF-D	"		1	0.025	10923	2680.2162	163.0	0.18			-8.146
M5-714-SDF	"		1	0.0251	28159	2680.2162	418.6	0.18			
M5-714-SDF-D	"		1	0.0251	26819	2680.2162	398.7	0.18			4.875
M5-725-SDF	"		1	0.0248	19440	2680.2162	292.5	0.18			
M5-725-SDF-D	"		1	0.0248	18209	2680.2162	273.9	0.18			6.539
Blanks	"	"B (NR)"									

"B" Analyte concentration in the associated blank was >3 times the MDL.

"NR" Data was unavailable for review.

SERC Lab / EPA REMAP results

Total Phosphorus (TP) in Soil by EPA Method 365.1 (modified)

: Analysis not performed

: Analysis not required

: Average of Two

Data Entered by: njs 03-22-00 mwb 03-27-00

Data Entry Checked by: njs 03-28-00

Sampling Station ID	Matrix	Collection Date	Collection Time	Digestion Date	Run Date	Time Elapsed From Dig	Holding Time (Days)	Phosphorus Units (ppm, ug/g)	Detection Limit (ppm)	QA/QC Batch ID	QA Data % R	QA Data %RPD	QA Data Matrix %R	QA Data Sample RPD	Notes
M5-623-SDF	SD	09/30/99	915	11/12/99	11/19/99	43	28	173	0.06	EPAM5-C	115	7.3	92.7		
M5-630-SDF	SD	09/28/99	1520	11/12/99	11/19/99	45	28	176	0.06	EPAM5-C	115	7.3	92.7		
M5-633-SDF	SD	09/29/99	1115	11/12/99	11/19/99	44	28	413	0.06	EPAM5-C	115	7.3	92.7		
M5-634-SDF	SD	09/29/99	1510	11/12/99	11/19/99	44	28	533	0.06	EPAM5-C	115	7.3	92.7		
M5-637-SDF	SD	09/29/99	1210	11/12/99	11/19/99	44	28	401	0.06	EPAM5-C	115	7.3	92.7		
M5-640-SDF	SD	09/29/99	1116	11/12/99	11/19/99	44	28	596	0.06	EPAM5-C	115	7.3	92.7		
M5-641-SDF	SD	09/28/99	1615	11/12/99	11/19/99	45	28	368	0.06	EPAM5-C	115	7.3	92.7		
M5-644-SDF	SD	09/30/99	1145	11/12/99	11/19/99	43	28	477	0.06	EPAM5-C	115	7.3	92.7		
M5-646-SDF	SD	09/28/99	1515	11/12/99	11/19/99	45	28	421	0.06	EPAM5-C	115	7.3	92.7		
M5-647-SDF	SD	09/28/99	1102	11/12/99	11/19/99	45	28	379	0.06	EPAM5-C	115	7.3	92.7	1.02	Averaged Result
M5-648-SDF	SD	09/28/99	1300	11/12/99	11/19/99	45	28	236	0.06	EPAM5-C	115	7.3	92.7		
M5-649-SDF	SD	09/28/99	1620	11/12/99	11/19/99	45	28	298	0.06	EPAM5-C	115	7.3	92.7		
M5-650-SDF	SD	09/28/99	1158	11/12/99	11/19/99	45	28	355	0.06	EPAM5-C	115	7.3	92.7		
M5-651-SDF	SD	09/28/99	1410	11/12/99	11/19/99	45	28	202	0.06	EPAM5-C	115	7.3	92.7		
M5-653-SDF	SD	09/28/99	1300	11/12/99	11/19/99	45	28	129	0.06	EPAM5-C	115	7.3	92.7		
M5-655-SDF	SD	09/28/99	1028	11/12/99	11/19/99	45	28	638	0.06	EPAM5-C	115	7.3	92.7		
M5-657-SDF	SD	09/27/99	1751	11/12/99	11/19/99	46	28	368	0.06	EPAM5-C	115	7.3	92.7		
M5-659-SDF	SD	09/27/99	1205	11/12/99	11/19/99	46	28	362	0.06	EPAM5-C	115	7.3	92.7		
M5-660-SDF	SD	09/27/99	1450	11/12/99	11/19/99	46	28	580	0.06	EPAM5-C	115	7.3	92.7		
M5-661-SDF	SD	09/29/99	857	11/12/99	11/19/99	44	28	153	0.06	EPAM5-D	115	6.2	93.8	1.26	Averaged Result
M5-664-SDF	SD	09/27/99	1100	11/12/99	11/19/99	46	28	209	0.06	EPAM5-D	115	6.2	93.8		
M5-666-SDF	SD	09/27/99	1655	11/12/99	11/19/99	46	28	288	0.06	EPAM5-D	115	6.2	93.8		
M5-667-SDF	SD	09/27/99	1545	11/12/99	11/19/99	46	28	386	0.06	EPAM5-D	115	6.2	93.8		
M5-668-SDF	SD	09/27/99	1207	11/12/99	11/19/99	46	28	234	0.06	EPAM5-D	115	6.2	93.8		
M5-671-SDF	SD	09/26/99	850	11/12/99	11/19/99	47	28	205	0.06	EPAM5-D	115	6.2	93.8		
M5-672-SDF	SD	09/27/99	1100	11/12/99	11/19/99	46	28	288	0.06	EPAM5-D	115	6.2	93.8		
M5-673-SDF	SD	09/27/99	1400	11/12/99	11/19/99	46	28	119	0.06	EPAM5-D	115	6.2	93.8		
M5-674-SDF	SD	09/26/99	1025	11/12/99	11/19/99	47	28	499	0.06	EPAM5-D	115	6.2	93.8		
M5-676-SDF	SD	09/28/99	925	11/12/99	11/19/99	45	28	273	0.06	EPAM5-D	115	6.2	93.8	20.1	Averaged Result
M5-677-SDF	SD	09/26/99	1310	11/12/99	11/19/99	47	28	288	0.06	EPAM5-D	115	6.2	93.8		
M5-678-SDF	SD	09/26/99	1213	11/12/99	11/19/99	47	28	305	0.06	EPAM5-D	115	6.2	93.8		
M5-679-SDF	SD	09/26/99	1335	11/12/99	11/19/99	47	28	265	0.06	EPAM5-D	115	6.2	93.8		
M5-680-SDF	SD	09/27/99	900	11/12/99	11/19/99	46	28	330	0.06	EPAM5-D	115	6.2	93.8		
M5-681-SDF	SD	09/26/99	1410	11/12/99	11/19/99	47	28	135	0.06	EPAM5-D	115	6.2	93.8		
M5-682-SDF	SD	09/26/99	1615	11/12/99	11/19/99	47	28	215	0.06	EPAM5-D	115	6.2	93.8		
M5-683-SDF	SD	09/26/99	850	11/12/99	11/19/99	47	28	281	0.06	EPAM5-D	115	6.2	93.8		
M5-684-SDF	SD	09/26/99	1530	11/12/99	11/19/99	47	28	137	0.06	EPAM5-D	115	6.2	93.8		

Sampling Station ID	Matrix	Collection		Digestion Date	Run Date	Time Elapsed From Dig	Holding Time (Days)	Phosphorus Units (ppm, ug/g)	Detection Limit (ppm)	QA/QC Batch ID	QA Data				Notes
		Date	Time								% R	%RPD	Matrix %R	Sample RPD	
M5-685-SDF	SD	09/26/99	1434	11/12/99	11/19/99	47	28	352	0.06	EPAM5-D	115	6.2	93.8	6.56	Averaged Result
M5-686-SDF	SD	09/25/99	915	11/12/99	11/19/99	48	28	436	0.06	EPAM5-D	115	6.2	93.8		
M5-687-SDF	SD	09/25/99	1035	11/12/99	11/19/99	48	28	402	0.06	EPAM5-D	115	6.2	93.8		
M5-688-SDF	SD	09/26/99	1527	11/12/99	11/19/99	47	28	268	0.06	EPAM5-D	115	6.2	93.8		
M5-689-SDF	SD	09/25/99	1205	11/12/99	11/19/99	48	28	192	0.06	EPAM5-D	115	6.2	93.8		
M5-690-SDF	SD	09/25/99	1350	11/12/99	11/19/99	48	28	177	0.06	EPAM5-D	115	6.2	93.8		
M5-691-SDF	SD	09/25/99	1554	11/12/99	11/19/99	48	28	386	0.06	EPAM5-D	115	6.2	93.8		
M5-733-SDF	SD	09/23/99	910	11/15/99	11/19/99	53	28	162	0.06	EPAM5-C	115	7.3	92.7		
M5-734-SDF	SD	09/22/99	1540	11/15/99	11/19/99	54	28	334	0.06	EPAM5-C	115	7.3	92.7		
M5-735-SDF	SD	09/22/99	1700	11/15/99	11/19/99	54	28	70	0.06	EPAM5-C	115	7.3	92.7	16.11	Averaged Result
M5-738-SDF	SD	09/22/99	1410	11/15/99	11/19/99	54	28	359	0.06	EPAM5-C	115	7.3	92.7		
M5-740-SDF	SD	09/22/99	1245	11/15/99	11/19/99	54	28	120	0.06	EPAM5-C	115	7.3	92.7		
M5-741-SDF	SD	09/22/99	1534	11/15/99	11/19/99	54	28	119	0.06	EPAM5-C	115	7.3	92.7		
M5-742-SDF	SD	09/22/99	1418	11/15/99	11/19/99	54	28	78	0.06	EPAM5-C	115	7.3	92.7		
M5-743-SDF	SD	09/22/99	1130	11/15/99	11/19/99	54	28	42	0.06	EPAM5-C	115	7.3	92.7		
M5-622-SDF	SD	09/30/99	1125	11/18/99	11/24/99	49	28	77	0.06	EPA1124C	115	4.6	86.2		
M5-624-SDF	SD	09/30/99	1018	11/18/99	11/24/99	49	28	267	0.06	EPA1124C	115	4.6	86.2		
M5-625-SDF	SD	09/30/99	1257	11/18/99	11/24/99	49	28	179	0.06	EPA1124C	115	4.6	86.2		
M5-626-SDF	SD	09/30/99	908	11/18/99	11/24/99	49	28	243	0.06	EPA1124C	115	4.6	86.2		
M5-627-SDF	SD	09/30/99	1050	11/18/99	11/24/99	49	28	417	0.06	EPA1124C	115	4.6	86.2		
M5-628-SDF	SD	09/29/99	1716	11/18/99	11/24/99	50	28	285	0.06	EPA1124C	115	4.6	86.2		
M5-631-SDF	SD	09/29/99	1414	11/18/99	11/24/99	50	28	226	0.06	EPA1124C	115	4.6	86.2		
M5-632-SDF	SD	09/29/99	1010	11/18/99	11/24/99	50	28	552	0.06	EPA1124C	115	4.6	86.2		
M5-635-SDF	SD	09/29/99	1216	11/18/99	11/24/99	50	28	271	0.06	EPA1124C	115	4.6	86.2		
M5-636-SDF	SD	09/29/99	1405	11/18/99	11/24/99	50	28	214	0.06	EPA1124C	115	4.6	86.2	3.37	Averaged Result
M5-638-SDF	SD	09/29/99	1630	11/18/99	11/24/99	50	28	741	0.06	EPA1124C	115	4.6	86.2		
M5-639-SDF	SD	09/28/99	1715	11/18/99	11/24/99	51	28	453	0.06	EPA1124C	115	4.6	86.2		
M5-642-SDF	SD	09/29/99	1011	11/18/99	11/24/99	50	28	372	0.06	EPA1124C	115	4.6	86.2		
M5-643-SDF	SD	09/29/99	910	11/18/99	11/24/99	50	28	285	0.06	EPA1124C	115	4.6	86.2		
M5-645-SDF	SD	09/28/99	1447	11/18/99	11/24/99	51	28	409	0.06	EPA1124C	115	4.6	86.2		
M5-647-SDF	SD	09/28/99	1102	11/18/99	11/24/99	51	28	396	0.06	EPA1124C	115	4.6	86.2		
M5-652-SDF	SD	09/28/99	959	11/18/99	11/24/99	51	28	430	0.06	EPA1124C	115	4.6	86.2		
M5-654-SDF	SD	09/28/99	1145	11/18/99	11/24/99	51	28	384	0.06	EPA1124C	115	4.6	86.2		
M5-656-SDF	SD	09/28/99	900	11/18/99	11/24/99	51	28	298	0.06	EPA1124C	115	4.6	86.2		
M5-658-SDF	SD	09/28/99	1722	11/18/99	11/24/99	51	28	268	0.06	EPA1124C	115	4.6	86.2	6.19	Averaged Result
M5-662-SDF	SD	09/27/99	1610	11/18/99	11/24/99	52	28	291	0.06	EPA1124C	115	4.6	86.2		
M5-663-SDF	SD	09/27/99	1330	11/18/99	11/24/99	52	28	148	0.06	EPA1124C	115	4.6	86.2		
M5-665-SDF	SD	09/27/99	1710	11/18/99	11/24/99	52	28	263	0.06	EPA1124C	115	4.6	86.2		
M5-669-SDF	SD	09/27/99	1000	11/18/99	11/24/99	52	28	236	0.06	EPA1124C	115	4.6	86.2		
M5-670-SDF	SD	09/27/99	850	11/18/99	11/24/99	52	28	150	0.06	EPA1124C	115	4.6	86.2		
M5-708-SDF	SD	09/24/99	900	11/18/99	11/24/99	55	28	162	0.06	EPA1124C	115	4.6	86.2		
M5-744-SDF	SD	09/22/99	1224	11/15/99	11/19/99	54	28	162	0.06	EPAM5-A	105	8.4	82.3		

Sampling Station ID	Matrix	Collection		Digestion Date	Run Date	Time Elapsed From Dig	Holding Time (Days)	Phosphorus Units (ppm, ug/g)	Detection Limit (ppm)	QA/QC Batch ID	QA Data				Notes
		Date	Time								% R	%RPD	Matrix %R	Sample RPD	
M5-745-SDF	SD	09/22/99	1120	11/15/99	11/19/99	54	28	26	0.06	EPAM5-A	105	8.4	82.3		
M5-746-SDF	SD	09/22/99	948	11/15/99	11/19/99	54	28	72	0.06	EPAM5-A	105	8.4	82.3		
M5-747-SDF	SD	09/22/99	942	11/15/99	11/19/99	54	28	96	0.06	EPAM5-A	105	8.4	82.3		
M5-823-SDF	SD	09/30/99	0	11/15/99	11/19/99	46	28	132	0.06	EPAM5-A	105	8.4	82.3		
M5-828-SDF	SD	09/29/99	0	11/15/99	11/19/99	47	28	183	0.06	EPAM5-A	105	8.4	82.3		
M5-838-SDF	SD	09/29/99	0	11/15/99	11/19/99	47	28	124	0.06	EPAM5-A	105	8.4	82.3		
M5-848-SDF	SD	09/28/99	0	11/15/99	11/19/99	48	28	177	0.06	EPAM5-A	105	8.4	82.3		
M5-859-SDF	SD	09/27/99	0	11/15/99	11/19/99	49	28	169	0.06	EPAM5-A	105	8.4	82.3		
M5-868-SDF	SD	09/27/99	0	11/15/99	11/19/99	49	28	560	0.06	EPAM5-A	105	8.4	82.3	-2.97	Averaged Result
M5-878-SDF	SD	09/26/99	0	11/15/99	11/19/99	50	28	224	0.06	EPAM5-A	105	8.4	82.3		
M5-890-SDF	SD	09/25/99	0	11/15/99	11/19/99	51	28	179	0.06	EPAM5-A	105	8.4	82.3		
M5-908-SDF	SD	09/24/99	0	11/15/99	11/19/99	52	28	175	0.06	EPAM5-A	105	8.4	82.3		
M5-920-SDF	SD	09/23/99	0	11/15/99	11/19/99	53	28	157	0.06	EPAM5-A	105	8.4	82.3		
M5-932-SDF	SD	09/23/99	0	11/15/99	11/19/99	53	28	378	0.06	EPAM5-A	105	8.4	82.3		
M5-944-SDF	SD	09/22/99	1224	11/15/99	11/19/99	54	28	275	0.06	EPAM5-A	105	8.4	82.3		
M5-693-SDF	SD	09/25/99	1139	11/15/99	11/19/99	51	28	186	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-694-SDF	SD	09/25/99	1702	11/15/99	11/19/99	51	28	191	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-695-SDF	SD	09/25/99	1400	11/15/99	11/19/99	51	28	82	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-696-SDF	SD	09/25/99	1510	11/15/99	11/19/99	51	28	141	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-697-SDF	SD	09/26/99	1637	11/15/99	11/19/99	50	28	95	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-698-SDF	SD	09/25/99	1030	11/15/99	11/19/99	51	28	279	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-699-SDF	SD	09/25/99	1635	11/15/99	11/19/99	51	28	225	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-700-SDF	SD	09/25/99	1750	11/15/99	11/19/99	51	28	195	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-701-SDF	SD	09/26/99	1740	11/15/99	11/19/99	50	28	158	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-702-SDF	SD	09/25/99	918	11/15/99	11/19/99	51	28	157	0.06	EPAM5-A	105	8.4	82.3	-8.15	Sample was label as SPF and Reported as SDF
M5-703-SDF	SD	09/24/99	1655	11/15/99	11/19/99	52	28	113	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-704-SDF	SD	09/24/99	1615	11/15/99	11/19/99	52	28	142	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-705-SDF	SD	09/24/99	1725	11/15/99	11/19/99	52	28	231	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-706-SDF	SD	09/24/99	1555	11/15/99	11/19/99	52	28	192	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-707-SDF	SD	09/24/99	1500	11/15/99	11/19/99	52	28	691	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-709-SDF	SD	09/24/99	1330	11/15/99	11/19/99	52	28	133	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-710-SDF	SD	09/24/99	1430	11/15/99	11/19/99	52	28	166	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-711-SDF	SD	09/24/99	1115	11/15/99	11/19/99	52	28	178	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-712-SDF	SD	09/24/99	905	11/15/99	11/19/99	52	28	232	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-714-SDF	SD	09/23/99	1715	11/15/99	11/19/99	53	28	409	0.06	EPAM5-A	105	8.4	82.3	4.87	Sample was label as SPF and Reported as SDF
M5-715-SDF	SD	09/24/99	1145	11/15/99	11/19/99	52	28	109	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-716-SDF	SD	09/24/99	1310	11/15/99	11/19/99	52	28	246	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-718-SDF	SD	09/24/99	1030	11/15/99	11/19/99	52	28	115	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-719-SDF	SD	09/23/99	1715	11/15/99	11/19/99	53	28	172	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-720-SDF	SD	09/23/99	0	11/15/99	11/19/99	53	28	122	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-722-SDF	SD	09/23/99	1600	11/15/99	11/19/99	53	28	77	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-723-SDF	SD	09/23/99	1500	11/15/99	11/19/99	53	28	85	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF

Sampling Station ID	Matrix	Collection		Digestion Date	Run Date	Time Elapsed From Dig	Holding Time (Days)	Phosphorus Units (ppm, ug/g)	Detection Limit (ppm)	QA/QC Batch ID	QA Data				Notes
		Date	Time								% R	%RPD	Matrix %R	Sample RPD	
M5-724-SDF	SD	09/23/99	1442	11/15/99	11/19/99	53	28	158	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-725-SDF	SD	09/23/99	1323	11/15/99	11/19/99	53	28	283	0.06	EPAM5-A	105	8.4	82.3	6.54	Sample was label as SPF and Reported as SDF
M5-726-SDF	SD	09/23/99	1230	11/15/99	11/19/99	53	28	240	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-727-SDF	SD	09/23/99	1216	11/15/99	11/19/99	53	28	119	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-728-SDF	SD	09/23/99	1350	11/15/99	11/19/99	53	28	132	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-729-SDF	SD	09/23/99	1027	11/15/99	11/19/99	53	28	240	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-730-SDF	SD	09/23/99	1120	11/15/99	11/19/99	53	28	494	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-731-SDF	SD	09/22/99	1725	11/15/99	11/19/99	54	28	91	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF
M5-732-SDF	SD	09/23/99	917	11/15/99	11/19/99	53	28	419	0.06	EPAM5-A	105	8.4	82.3		Sample was label as SPF and Reported as SDF

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-622-SDF Soil/Sediment

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	09/30/99	09/30/99	09/30/99	09/30/99	09/30/99
Digestion Date	Not Analyzed	02/21/00	11/18/99	12/09/99	
Analysis Date	Battelle Lab	02/21/00	11/24/99	12/09/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume		4.495,4.606	0.025 g	0.025 g	80
Total Volume		200ul	0.025 g	0.025 g	80
Sample Volume Analyzed		aliquot	Aliquot	0.025 g	80
Dilution		See Worksheets	1	NA	No Data Reviewed
Results		16.6	77	38.5	
Measuring Unit	ppm	ng/g	ug/g	%	g/mL
EPA Method	CVAF	CVAF	EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst		JL	Angel	SG/JL	SG/JL
Method Detection limit		0.2 ng/g	0.06	0	0.001 g/mL
Data Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked		Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	28 days	No (Goal Only)	No (Goal Only)	NA	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID		022100a	EPA1124C	12/09/99	
Method Blanks		NA	NA in Soils	NA	
Instrument Blanks		All Good	"NR"	NA	
Duplicates (RPD)		Good	<20 RPD	None Reported	
Matrix Spike Recoveries (75-125)		All Good	All Good	NA	
Calibration % R (high and low range)		Good	Good	NA	
Detection Range		0.2 ng/g and >	60 ppm and >	NA	
Correlation Coefficient (>0.995)		0.9815	0.9964	NA	
QC Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	
All Calculation Checked		Yes	X	X	
QC Limits Met	Battelle Lab	"R2"	"B (NR)"	"DQO (NR)"	No Data Reviewed

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time goals. Holding time is a goal only.

Bulk Density data was not provided for review or reporting.

Station ID M5-622-SDF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage		Hg lab	Not Noted	Not Noted	
Log-in Procedures	Internal COC	Internal COC	Internal COC	Internal COC	
Raw Data (Attached)					
Sample Work Sheets		X	X	X	
Sample Run Logs		X	X	X	
Instrument Raw Data		X	X	NA	
Bench Sheets		X	X	X	
Sample Preparation Logs		X	X	X	
Raw Data (Verified)					
Sample ID Transferred		X	X	X	
All Calculation Checked		X	X	X	
Measuring Unit		ng/g	ug/g	%	g/mL
PE Results (Attached)					
Organization	NA	NA	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA	NA	NA
Validation Criteria					
Applied Qualifiers		"R2"	"B (NR)"	"DQO (NR)"	

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated
"R2" Correlation Coefficient is out of QAPP limits.
"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.
"B" Analyte concentration in the associated blank was >3 times the MDL.
"H" Analysis digestion performed after holding times have expired.
"NR" Data was unavailable for review.
"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-633-SDF Soil/Sediment

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	09/29/99	09/29/99	09/29/99	09/29/99	09/29/99
Digestion Date	Not Analyzed	03/20/00	11/12/99	12/09/99	
Analysis Date	Battelle Lab	03/20/00	11/19/99	12/09/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume		4.537, 4.922	0.025 g	0.025 g	80
Total Volume		200ul	0.025 g	0.025 g	80
Sample Volume Analyzed		aliquot	Aliquot	0.025 g	80
Dilution		See Worksheets	1	NA	No Data Reviewed
Results		8.2	413	87.4	
Measuring Unit	ppm	ng/g	ug/g	%	g/mL
EPA Method	CVAF	CVAF	EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst		JL	Angel	SG/JL	SG/JL
Method Detection limit		0.2 ng/g	0.06	0	0.001 g/mL
Data Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked		Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	28 days	No (Goal Only)	No (Goal Only)	NA	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID		032000b	EPAM5-C	12/09/99	
Method Blanks		NA	NA in Soils	NA	
Instrument Blanks		All Good	"NR"	NA	
Duplicates (RPD)		Good	<20 RPD	None Reported	
Matrix Spike Recoveries (75-125)		48.70%	All Good	NA	
Calibration % R (high and low range)		Good	Good	NA	
Detection Range		0.2 ng/g and >	60 ppm and >	NA	
Correlation Coefficient (>0.995)		0.9808	0.9965	NA	
QC Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	
All Calculation Checked		Yes	X	X	
QC Limits Met	Battelle Lab	"R2", "M"	"B (NR)"	"DQO (NR)"	No Data Reviewed

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time goals. Holding time is a goal only.

Bulk Density data was not provided for review or reporting.

Station ID M5-633-SDF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage		Hg lab	Not Noted	Not Noted	
Log-in Procedures	Internal COC	Internal COC	Internal COC	Internal COC	
Raw Data (Attached)					
Sample Work Sheets		X	X	X	
Sample Run Logs		X	X	X	
Instrument Raw Data		X	X	NA	
Bench Sheets		X	X	X	
Sample Preparation Logs		X	X	X	
Raw Data (Verified)					
Sample ID Transferred		X	X	X	
All Calculation Checked		X	X	X	
Measuring Unit		ng/g	ug/g	%	g/mL
PE Results (Attached)					
Organization	NA	NA	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA	NA	NA
Validation Criteria					
Applied Qualifiers		"R2", "M"	"B (NR)"	"DQO (NR)"	

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated
"R2" Correlation Coefficient is out of QAPP limits.
"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.
"B" Analyte concentration in the associated blank was >3 times the MDL.
"H" Analysis digestion performed after holding times have expired.
"NR" Data was unavailable for review.
"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-643-SDF Soil/Sediment

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	09/29/99	09/29/99	09/29/99	09/29/99	09/29/99
Digestion Date	Not Analyzed	02/21/00	11/18/99	12/09/99	
Analysis Date	Battelle Lab	02/21/00	11/24/99	12/09/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume		5.477, 4.590	0.025 g	0.025 g	80
Total Volume		200ul	0.025 g	0.025 g	80
Sample Volume Analyzed		aliquot	Aliquot	0.025 g	80
Dilution		See Worksheets	1	NA	No Data Reviewed
Results		1.4	285	53.6	
Measuring Unit	ppm	ng/g	ug/g	%	g/mL
EPA Method	CVAF	CVAF	EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst		JL	Angel	SG/JL	SG/JL
Method Detection limit		0.2 ng/g	0.06	0	0.001 g/mL
Data Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked		Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	28 days	No (Goal Only)	No (Goal Only)	NA	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID		022100b	EPA1124C	12/09/99	
Method Blanks		NA	NA in Soils	NA	
Instrument Blanks		All Good	"NR"	NA	
Duplicates (RPD)		Good	<20 RPD	None Reported	
Matrix Spike Recoveries (75-125)		130.56	All Good	NA	
Calibration % R (high and low range)		Good	Good	NA	
Detection Range		0.2 ng/g and >	60 ppm and >	NA	
Correlation Coefficient (>0.995)		0.9914	0.9964	NA	
QC Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	
All Calculation Checked		Yes	X	X	
QC Limits Met	Battelle Lab	"R2", "M"	"B (NR)"	"DQO (NR)"	No Data Reviewed

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time goals. Holding time is a goal only.

Bulk Density data was not provided for review or reporting.

Station ID M5-643-SDF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage		Hg lab	Not Noted	Not Noted	
Log-in Procedures	Internal COC	Internal COC	Internal COC	Internal COC	
Raw Data (Attached)					
Sample Work Sheets		X	X	X	
Sample Run Logs		X	X	X	
Instrument Raw Data		X	X	NA	
Bench Sheets		X	X	X	
Sample Preparation Logs		X	X	X	
Raw Data (Verified)					
Sample ID Transferred		X	X	X	
All Calculation Checked		X	X	X	
Measuring Unit		ng/g	ug/g	%	g/mL
PE Results (Attached)					
Organization	NA	NA	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA	NA	NA
Validation Criteria					
Applied Qualifiers		"R2", "M"	"B (NR)"	"DQO (NR)"	

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated
"R2" Correlation Coefficient is out of QAPP limits.
"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.
"B" Analyte concentration in the associated blank was >3 times the MDL.
"H" Analysis digestion performed after holding times have expired.
"NR" Data was unavailable for review.
"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-653-SDF Soil/Sediment

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	09/28/99	09/28/99	09/28/99	09/28/99	09/28/99
Digestion Date	Not Analyzed	02/28/99	11/12/99	12/09/99	
Analysis Date	Battelle Lab	02/28/99	11/19/99	12/09/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume		6.060, 5.583	0.025 g	0.025 g	80
Total Volume		200ul	0.025 g	0.025 g	80
Sample Volume Analyzed		aliquot	Aliquot	0.025 g	80
Dilution		See Worksheets	1	NA	No Data Reviewed
Results		0.1	129	15.9	
Measuring Unit	ppm	ng/g	ug/g	%	g/mL
EPA Method	CVAF	CVAF	EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst		JL	Angel	SG/JL	SG/JL
Method Detection limit		0.2 ng/g	0.06	0	0.001 g/mL
Data Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked		Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	28 days	No (Goal Only)	No (Goal Only)	NA	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID		22800	EPAM5-C	12/09/99	
Method Blanks		NA	NA in Soils	NA	
Instrument Blanks		All Good	"NR"	NA	
Duplicates (RPD)		Good	<20 RPD	None Reported	
Matrix Spike Recoveries (75-125)		All Good	All Good	NA	
Calibration % R (high and low range)		Not Reviewed	Good	NA	
Detection Range		0.2 ng/g and >	60 ppm and >	NA	
Correlation Coefficient (>0.995)		0.9985	0.9965	NA	
QC Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	
All Calculation Checked		Yes	X	X	
QC Limits Met	Battelle Lab	"DQO (NR)"	"B (NR)"	"DQO (NR)"	No Data Reviewed

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time goals. Holding time is a goal only.

Bulk Density data was not provided for review or reporting.

Station ID M5-653-SDF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage		Hg lab	Not Noted	Not Noted	
Log-in Procedures	Internal COC	Internal COC	Internal COC	Internal COC	
Raw Data (Attached)					
Sample Work Sheets		X	X	X	
Sample Run Logs		X	X	X	
Instrument Raw Data		X	X	NA	
Bench Sheets		X	X	X	
Sample Preparation Logs		X	X	X	
Raw Data (Verified)					
Sample ID Transferred		X	X	X	
All Calculation Checked		X	X	X	
Measuring Unit		ng/g	ug/g	%	g/mL
PE Results (Attached)					
Organization	NA	NA	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA	NA	NA
Validation Criteria					
Applied Qualifiers		"DQO (NR)"	"B (NR)"	"DQO (NR)"	

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated
"R2" Correlation Coefficient is out of QAPP limits.
"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.
"B" Analyte concentration in the associated blank was >3 times the MDL.
"H" Analysis digestion performed after holding times have expired.
"NR" Data was unavailable for review.
"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-663-SDF Soil/Sediment

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	09/27/99	09/27/99	09/27/99	09/27/99	09/27/99
Digestion Date	Not Analyzed	02/29/00	11/18/99	12/09/99	
Analysis Date	Battelle Lab	02/29/00	11/24/99	12/09/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume		5.209, 4.609	0.025 g	0.025 g	80
Total Volume		200ul	0.025 g	0.025 g	80
Sample Volume Analyzed		aliquot	Aliquot	0.025 g	80
Dilution		See Worksheets	1	NA	No Data Reviewed
Results		3.9	148	69.3	
Measuring Unit	ppm	ng/g	ug/g	%	g/mL
EPA Method	CVAF	CVAF	EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst		JL	Angel	SG/JL	SG/JL
Method Detection limit		0.2 ng/g	0.06	0	0.001 g/mL
Data Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked		Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	28 days	No (Goal Only)	No (Goal Only)	NA	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID		022500b	EPA1124C	12/09/99	
Method Blanks		NA	NA in Soils	NA	
Instrument Blanks		All Good	"NR"	NA	
Duplicates (RPD)		Good	<20 RPD	None Reported	
Matrix Spike Recoveries (75-125)		36.84%	All Good	NA	
Calibration % R (high and low range)		Good	Good	NA	
Detection Range		0.2 ng/g and >	60 ppm and >	NA	
Correlation Coefficient (>0.995)		0.9871	0.9964	NA	
QC Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	
All Calculation Checked		Yes	X	X	
QC Limits Met	Battelle Lab	"R2", "M"	"B (NR)"	"DQO (NR)"	No Data Reviewed

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time goals. Holding time is a goal only.

Bulk Density data was not provided for review or reporting.

Station ID M5-663-SDF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage		Hg lab	Not Noted	Not Noted	
Log-in Procedures	Internal COC	Internal COC	Internal COC	Internal COC	
Raw Data (Attached)					
Sample Work Sheets		X	X	X	
Sample Run Logs		X	X	X	
Instrument Raw Data		X	X	NA	
Bench Sheets		X	X	X	
Sample Preparation Logs		X	X	X	
Raw Data (Verified)					
Sample ID Transferred		X	X	X	
All Calculation Checked		X	X	X	
Measuring Unit		ng/g	ug/g	%	g/mL
PE Results (Attached)					
Organization	NA	NA	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA	NA	NA
Validation Criteria					
Applied Qualifiers		"R2", "M"	"B (NR)"	"DQO (NR)"	

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated
"R2" Correlation Coefficient is out of QAPP limits.
"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.
"B" Analyte concentration in the associated blank was >3 times the MDL.
"H" Analysis digestion performed after holding times have expired.
"NR" Data was unavailable for review.
"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-673-SDF Soil/Sediment

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	09/27/99	09/27/99	09/27/99	09/27/99	09/27/99
Digestion Date	Not Analyzed	03/02/00	11/12/99	12/09/99	
Analysis Date	Battelle Lab	03/02/00	11/19/99	12/09/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume		5.076, 4.834	0.025 g	0.025 g	80
Total Volume		200ul	0.025 g	0.025 g	80
Sample Volume Analyzed		aliquot	Aliquot	0.025 g	80
Dilution		See Worksheets	1	NA	No Data Reviewed
Results		3.3	119	32.7	
Measuring Unit	ppm	ng/g	ug/g	%	g/mL
EPA Method	CVAF	CVAF	EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst		JL	Angel	SG/JL	SG/JL
Method Detection limit		0.2 ng/g	0.06	0	0.001 g/mL
Data Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked		Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	28 days	No (Goal Only)	No (Goal Only)	NA	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID		022100a	EPAM5-D	12/09/99	
Method Blanks		NA	NA in Soils	NA	
Instrument Blanks		All Good	"NR"	NA	
Duplicates (RPD)		Good	<20 RPD	None Reported	
Matrix Spike Recoveries (75-125)		All Good	All Good	NA	
Calibration % R (high and low range)		Good	Good	NA	
Detection Range		0.2 ng/g and >	60 ppm and >	NA	
Correlation Coefficient (>0.995)		0.9815	0.9973	NA	
QC Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	
All Calculation Checked		Yes	X	X	
QC Limits Met	Battelle Lab	"R2"	"B (NR)"	"DQO (NR)"	No Data Reviewed

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time goals. Holding time is a goal only.

Bulk Density data was not provided for review or reporting.

Station ID M5-673-SDF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage		Hg lab	Not Noted	Not Noted	
Log-in Procedures	Internal COC	Internal COC	Internal COC	Internal COC	
Raw Data (Attached)					
Sample Work Sheets		X	X	X	
Sample Run Logs		X	X	X	
Instrument Raw Data		X	X	NA	
Bench Sheets		X	X	X	
Sample Preparation Logs		X	X	X	
Raw Data (Verified)					
Sample ID Transferred		X	X	X	
All Calculation Checked		X	X	X	
Measuring Unit		ng/g	ug/g	%	g/mL
PE Results (Attached)					
Organization	NA	NA	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA	NA	NA
Validation Criteria					
Applied Qualifiers		"R2"	"B (NR)"	"DQO (NR)"	

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated
"R2" Correlation Coefficient is out of QAPP limits.
"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.
"B" Analyte concentration in the associated blank was >3 times the MDL.
"H" Analysis digestion performed after holding times have expired.
"NR" Data was unavailable for review.
"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-683-SDF Soil/Sediment

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	09/26/99	09/26/99	09/26/99	09/26/99	09/26/99
Digestion Date	Not Analyzed	03/03/00	11/12/99	12/13/99	
Analysis Date	Battelle Lab	03/03/00	11/19/99	12/13/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume		5.186, 4.518	0.025 g	0.025 g	80
Total Volume		200ul	0.025 g	0.025 g	80
Sample Volume Analyzed		aliquot	Aliquot	0.025 g	80
Dilution		See Worksheets	1	NA	No Data Reviewed
Results		0.36	77	88.7	
Measuring Unit	ppm	ng/g	ug/g	%	g/mL
EPA Method	CVAF	CVAF	EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst		JL	Angel	SG/JL	SG/JL
Method Detection limit		0.2 ng/g	0.06	0	0.001 g/mL
Data Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked		Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	28 days	No (Goal Only)	No (Goal Only)	NA	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID		30300a	EPAM5-D	12/13/99	
Method Blanks		NA	NA in Soils	NA	
Instrument Blanks		All Good	"NR"	NA	
Duplicates (RPD)		Good	<20 RPD	None Reported	
Matrix Spike Recoveries (75-125)		131.81%	All Good	NA	
Calibration % R (high and low range)		140%	Good	NA	
Detection Range		0.2 ng/g and >	60 ppm and >	NA	
Correlation Coefficient (>0.995)		0.9823	0.9973	NA	
QC Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	
All Calculation Checked		Yes	X	X	
QC Limits Met	Battelle Lab	"R2", "M", "DQO"	"B (NR)"	"DQO (NR)"	No Data Reviewed

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time goals. Holding time is a goal only.

Bulk Density data was not provided for review or reporting.

Station ID M5-683-SDF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage		Hg lab	Not Noted	Not Noted	
Log-in Procedures	Internal COC	Internal COC	Internal COC	Internal COC	
Raw Data (Attached)					
Sample Work Sheets		X	X	X	
Sample Run Logs		X	X	X	
Instrument Raw Data		X	X	NA	
Bench Sheets		X	X	X	
Sample Preparation Logs		X	X	X	
Raw Data (Verified)					
Sample ID Transferred		X	X	X	
All Calculation Checked		X	X	X	
Measuring Unit		ng/g	ug/g	%	g/mL
PE Results (Attached)					
Organization	NA	NA	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA	NA	NA
Validation Criteria					
Applied Qualifiers		"R2", "M", "DQO"	"B (NR)"	"DQO (NR)"	

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated
"R2" Correlation Coefficient is out of QAPP limits.
"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.
"B" Analyte concentration in the associated blank was >3 times the MDL.
"H" Analysis digestion performed after holding times have expired.
"NR" Data was unavailable for review.
"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-693-SDF Soil/Sediment

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	09/25/99	09/25/99	09/25/99	09/25/99	09/25/99
Digestion Date	Not Analyzed	03/03/00	11/15/99	12/13/99	
Analysis Date	Battelle Lab	03/03/00	11/19/99	12/13/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume		5.279, 4.996	0.025 g	0.025 g	80
Total Volume		200ul	0.025 g	0.025 g	80
Sample Volume Analyzed		aliquot	Aliquot	0.025 g	80
Dilution		See Worksheets	1	NA	No Data Reviewed
Results		0.14	186	80.3	
Measuring Unit	ppm	ng/g	ug/g	%	g/mL
EPA Method	CVAF	CVAF	EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst		JL	Angel	SG/JL	SG/JL
Method Detection limit		0.2 ng/g	0.06	0	0.001 g/mL
Data Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked		Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	28 days	No (Goal Only)	No (Goal Only)	NA	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID		30300b	EPAM5-A	12/13/99	
Method Blanks		NA	NA in Soils	NA	
Instrument Blanks		All Good	"NR"	NA	
Duplicates (RPD)		-46.4	<20 RPD	None Reported	
Matrix Spike Recoveries (75-125)		210.87%	All Good	NA	
Calibration % R (high and low range)		145.17%	Good	NA	
Detection Range		0.2 ng/g and >	60 ppm and >	NA	
Correlation Coefficient (>0.995)		0.9397	0.995	NA	
QC Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	
All Calculation Checked		Yes	X	X	
QC Limits Met	Battelle Lab	"R2", "DQO", "M"	"B (NR)"	"DQO (NR)"	No Data Reviewed

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time goals. Holding time is a goal only.

Bulk Density data was not provided for review or reporting.

Station ID M5-693-SDF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage		Hg lab	Not Noted	Not Noted	
Log-in Procedures	Internal COC	Internal COC	Internal COC	Internal COC	
Raw Data (Attached)					
Sample Work Sheets		X	X	X	
Sample Run Logs		X	X	X	
Instrument Raw Data		X	X	NA	
Bench Sheets		X	X	X	
Sample Preparation Logs		X	X	X	
Raw Data (Verified)					
Sample ID Transferred		X	X	X	
All Calculation Checked		X	X	X	
Measuring Unit		ng/g	ug/g	%	g/mL
PE Results (Attached)					
Organization	NA	NA	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA	NA	NA
Validation Criteria					
Applied Qualifiers		"R2", "DQO", "M"	"B (NR)"	"DQO (NR)"	

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated
"R2" Correlation Coefficient is out of QAPP limits.
"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.
"B" Analyte concentration in the associated blank was >3 times the MDL.
"H" Analysis digestion performed after holding times have expired.
"NR" Data was unavailable for review.
"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-703-SDF Soil/Sediment

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	09/24/99	09/24/99	09/24/99	09/24/99	09/24/99
Digestion Date	Not Analyzed	03/07/00	11/15/99	12/13/99	
Analysis Date	Battelle Lab	03/07/00	11/19/99	12/13/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume		5.368, 5.250	0.025 g	0.025 g	80
Total Volume		200ul	0.025 g	0.025 g	80
Sample Volume Analyzed		aliquot	Aliquot	0.025 g	80
Dilution		See Worksheets	1	NA	No Data Reviewed
Results		0.24	113	29.9	
Measuring Unit	ppm	ng/g	0	%	g/mL
EPA Method	CVAF	CVAF	EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst		JL	Angel	SG/JL	SG/JL
Method Detection limit		0.2 ng/g	0.06	0	0.001 g/mL
Data Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked		Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	28 days	No (Goal Only)	No (Goal Only)	NA	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID		030700a	EPAM5-A	12/13/99	
Method Blanks		NA	NA in Soils	NA	
Instrument Blanks		All Good	"NR"	NA	
Duplicates (RPD)		35.9	<20 RPD	None Reported	
Matrix Spike Recoveries (75-125)		143	All Good	NA	
Calibration % R (high and low range)		Not Reported	Good	NA	
Detection Range		0.2 ng/g and >	60 ppm and >	NA	
Correlation Coefficient (>0.995)		0.9925	0.995	NA	
QC Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	
All Calculation Checked		Yes	X	X	
QC Limits Met	Battelle Lab	"R2", "M", "DQO"	"B (NR)"	"DQO (NR)"	No Data Reviewed

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time goals. Holding time is a goal only.

Bulk Density data was not provided for review or reporting.

Station ID M5-703-SDF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage		Hg lab	Not Noted	Not Noted	
Log-in Procedures	Internal COC	Internal COC	Internal COC	Internal COC	
Raw Data (Attached)					
Sample Work Sheets		X	X	X	
Sample Run Logs		X	X	X	
Instrument Raw Data		X	X	NA	
Bench Sheets		X	X	X	
Sample Preparation Logs		X	X	X	
Raw Data (Verified)					
Sample ID Transferred		X	X	X	
All Calculation Checked		X	X	X	
Measuring Unit		ng/g	ug/g	%	g/mL
PE Results (Attached)					
Organization	NA	NA	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA	NA	NA
Validation Criteria					
Applied Qualifiers		"R2", "M", "DQO"	"B (NR)"	"DQO (NR)"	

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated
"R2" Correlation Coefficient is out of QAPP limits.
"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.
"B" Analyte concentration in the associated blank was >3 times the MDL.
"H" Analysis digestion performed after holding times have expired.
"NR" Data was unavailable for review.
"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-714-SDF Soil/Sediment

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	09/23/99	09/23/99	09/23/99	09/23/99	09/23/99
Digestion Date	Not Analyzed	03/23/00	11/15/99	12/13/99	
Analysis Date	Battelle Lab	03/23/00	11/19/99	12/13/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume		4.908, 4.629	0.025 g	0.025 g	80
Total Volume		200ul	0.025 g	0.025 g	80
Sample Volume Analyzed		aliquot	Aliquot	0.025 g	80
Dilution		See Worksheets	1	NA	No Data Reviewed
Results		0.31	409	45.1	
Measuring Unit	ppm	ng/g	ug/g	%	g/mL
EPA Method	CVAF	CVAF	EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst		JL	Angel	SG/JL	SG/JL
Method Detection limit		0.2 ng/g	0.06	0	0.001 g/mL
Data Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked		Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	28 days	No (Goal Only)	No (Goal Only)	NA	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID		032300a	EPAM5-A	12/13/99	
Method Blanks		NA	NA in Soils	NA	
Instrument Blanks		All Good	"NR"	NA	
Duplicates (RPD)		Good	<20 RPD	None Reported	
Matrix Spike Recoveries (75-125)		146.26%	All Good	NA	
Calibration % R (high and low range)		Not Reported	Good	NA	
Detection Range		0.2 ng/g and >	60 ppm and >	NA	
Correlation Coefficient (>0.995)		0.9935	0.995	NA	
QC Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	
All Calculation Checked		Yes	X	X	
QC Limits Met	Battelle Lab	"R2", "M", "DQO"	"B (NR)"	"DQO (NR)"	No Data Reviewed

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time goals. Holding time is a goal only.

Bulk Density data was not provided for review or reporting.

Station ID M5-714-SDF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage		Hg lab	Not Noted	Not Noted	
Log-in Procedures	Internal COC	Internal COC	Internal COC	Internal COC	
Raw Data (Attached)					
Sample Work Sheets		X	X	X	
Sample Run Logs		X	X	X	
Instrument Raw Data		X	X	NA	
Bench Sheets		X	X	X	
Sample Preparation Logs		X	X	X	
Raw Data (Verified)					
Sample ID Transferred		X	X	X	
All Calculation Checked		X	X	X	
Measuring Unit		ng/g	ug/g	%	g/mL
PE Results (Attached)					
Organization	NA	NA	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA	NA	NA
Validation Criteria					
Applied Qualifiers		"R2", "M", "DQO"	"B (NR)"	"DQO (NR)"	

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated
"R2" Correlation Coefficient is out of QAPP limits.
"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.
"B" Analyte concentration in the associated blank was >3 times the MDL.
"H" Analysis digestion performed after holding times have expired.
"NR" Data was unavailable for review.
"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-726-SDF Soil/Sediment

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	09/23/99	09/23/99	09/23/99	09/23/99	09/23/99
Digestion Date	Not Analyzed	03/08/00	11/15/99	12/14/99	
Analysis Date	Battelle Lab	03/08/00	11/19/99	12/14/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume		5.183, 4.935	0.025 g	0.025 g	80
Total Volume		200ul	0.025 g	0.025 g	80
Sample Volume Analyzed		aliquot	Aliquot	0.025 g	80
Dilution		See Worksheets	1	NA	No Data Reviewed
Results		0.72	240	84.6	
Measuring Unit	ppm	ng/g	ug/g	%	g/mL
EPA Method	CVAF	CVAF	EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst		JL	Angel	SG/JL	SG/JL
Method Detection limit		0.2 ng/g	0.06	0	0.001 g/mL
Data Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked		Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	28 days	No (Goal Only)	No (Goal Only)	NA	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID		30800	EPAM5-A	12/14/99	
Method Blanks		NA	NA in Soils	NA	
Instrument Blanks		All Good	"NR"	NA	
Duplicates (RPD)		Good	<20 RPD	None Reported	
Matrix Spike Recoveries (75-125)		130.61%	All Good	NA	
Calibration % R (high and low range)		Good	Good	NA	
Detection Range		0.2 ng/g and >	60 ppm and >	NA	
Correlation Coefficient (>0.995)		0.9995	0.995	NA	
QC Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	
All Calculation Checked		Yes	X	X	
QC Limits Met	Battelle Lab	"M"	"B (NR)"	"DQO (NR)"	No Data Reviewed

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time goals. Holding time is a goal only.

Bulk Density data was not provided for review or reporting.

Station ID M5-726-SDF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage		Hg lab	Not Noted	Not Noted	
Log-in Procedures	Internal COC	Internal COC	Internal COC	Internal COC	
Raw Data (Attached)					
Sample Work Sheets		X	X	X	
Sample Run Logs		X	X	X	
Instrument Raw Data		X	X	NA	
Bench Sheets		X	X	X	
Sample Preparation Logs		X	X	X	
Raw Data (Verified)					
Sample ID Transferred		X	X	X	
All Calculation Checked		X	X	X	
Measuring Unit		ng/g	ug/g	%	g/mL
PE Results (Attached)					
Organization	NA	NA	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA	NA	NA
Validation Criteria					
Applied Qualifiers		"M"	"B (NR)"	"DQO (NR)"	

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated
"R2" Correlation Coefficient is out of QAPP limits.
"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.
"B" Analyte concentration in the associated blank was >3 times the MDL.
"H" Analysis digestion performed after holding times have expired.
"NR" Data was unavailable for review.
"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-738-SDF Soil/Sediment

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	09/22/99	09/22/99	09/22/99	09/22/99	09/22/99
Digestion Date	Not Analyzed	03/09/00	11/12/99	12/14/99	
Analysis Date	Battelle Lab	03/09/00	11/19/99	12/14/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume		5.101, 5.139	0.025 g	0.025 g	80
Total Volume		200ul	0.025 g	0.025 g	80
Sample Volume Analyzed		aliquot	Aliquot	0.025 g	80
Dilution		See Worksheets	1	NA	No Data Reviewed
Results		0.26	359	48.4	
Measuring Unit	ppm	ng/g	ug/g	%	g/mL
EPA Method	CVAF	CVAF	EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst		JL	Angel	SG/JL	SG/JL
Method Detection limit		0.2 ng/g	0.06	0	0.001 g/mL
Data Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked		Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	28 days	No (Goal Only)	No (Goal Only)	NA	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID		022500b	EPAM5-C	12/14/99	
Method Blanks		NA	NA in Soils	NA	
Instrument Blanks		All Good	"NR"	NA	
Duplicates (RPD)		Good	<20 RPD	None Reported	
Matrix Spike Recoveries (75-125)		All Good	All Good	NA	
Calibration % R (high and low range)		166, 114	Good	NA	
Detection Range		0.2 ng/g and >	60 ppm and >	NA	
Correlation Coefficient (>0.995)		0.9871	0.9965	NA	
QC Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	
All Calculation Checked		Yes	X	X	
QC Limits Met	Battelle Lab	"R2", "DQO"	"B (NR)"	"DQO (NR)"	No Data Reviewed

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time goals. Holding time is a goal only.

Bulk Density data was not provided for review or reporting.

Station ID M5-738-SDF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage		Hg lab	Not Noted	Not Noted	
Log-in Procedures	Internal COC	Internal COC	Internal COC	Internal COC	
Raw Data (Attached)					
Sample Work Sheets		X	X	X	
Sample Run Logs		X	X	X	
Instrument Raw Data		X	X	NA	
Bench Sheets		X	X	X	
Sample Preparation Logs		X	X	X	
Raw Data (Verified)					
Sample ID Transferred		X	X	X	
All Calculation Checked		X	X	X	
Measuring Unit		ng/g	ug/g	%	g/mL
PE Results (Attached)					
Organization	NA	NA	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA	NA	NA
Validation Criteria					
Applied Qualifiers		"R2", "DQO"	"B (NR)"	"DQO (NR)"	

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated
"R2" Correlation Coefficient is out of QAPP limits.
"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.
"B" Analyte concentration in the associated blank was >3 times the MDL.
"H" Analysis digestion performed after holding times have expired.
"NR" Data was unavailable for review.
"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-828-SDF Soil/Sediment

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	09/28/99	09/28/99	09/28/99	09/28/99	09/28/99
Digestion Date	Not Analyzed	04/04/00	11/15/99	12/15/99	
Analysis Date	Battelle Lab	04/04/00	11/19/99	12/15/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume		5.022	0.025 g	0.025 g	80
Total Volume		200ul	0.025 g	0.025 g	80
Sample Volume Analyzed		aliquot	Aliquot	0.025 g	80
Dilution		See Worksheets	1	NA	No Data Reviewed
Results		6.9	184	94.5	
Measuring Unit	ppm	ng/g	ug/g	%	g/mL
EPA Method	CVAF	CVAF	EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst		JL	Angel	SG/JL	SG/JL
Method Detection limit		0.2 ng/g	0.06	0	0.001 g/mL
Data Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked		Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	28 days	No (Goal Only)	No (Goal Only)	NA	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID		040400b	EPAM5-A	12/15/99	
Method Blanks		NA	NA in Soils	NA	
Instrument Blanks		All Good	"NR"	NA	
Duplicates (RPD)		Not Reported	<20 RPD	None Reported	
Matrix Spike Recoveries (75-125)		67.68%	All Good	NA	
Calibration % R (high and low range)		Good	Good	NA	
Detection Range		0.2 ng/g and >	60 ppm and >	NA	
Correlation Coefficient (>0.995)		0.9942	0.995	NA	
QC Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	
All Calculation Checked		Yes	X	X	
QC Limits Met	Battelle Lab	"R2", "M", "DQO (NR)"	"B (NR)"	"DQO (NR)"	No Data Reviewed

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time goals. Holding time is a goal only.

Bulk Density data was not provided for review or reporting.

Station ID M5-828-SDF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage		Hg lab	Not Noted	Not Noted	
Log-in Procedures	Internal COC	Internal COC	Internal COC	Internal COC	
Raw Data (Attached)					
Sample Work Sheets		X	X	X	
Sample Run Logs		X	X	X	
Instrument Raw Data		X	X	NA	
Bench Sheets		X	X	X	
Sample Preparation Logs		X	X	X	
Raw Data (Verified)					
Sample ID Transferred		X	X	X	
All Calculation Checked		X	X	X	
Measuring Unit		ng/g	ug/g	%	g/mL
PE Results (Attached)					
Organization	NA	NA	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA	NA	NA
Validation Criteria					
Applied Qualifiers		"R2", "M", "DQO (NR)"	"B (NR)"	"DQO (NR)"	

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated
"R2" Correlation Coefficient is out of QAPP limits.
"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.
"B" Analyte concentration in the associated blank was >3 times the MDL.
"H" Analysis digestion performed after holding times have expired.
"NR" Data was unavailable for review.
"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

May 1999 Samples for Critical Parameters Analyzed by SERC
With the 10% Full QA/QC Review

Station ID M5-944-SDF Soil/Sediment

Laboratory Records	**Total Hg**	**MeHg**	**Total Phosphorus**	**AFDW**	**Bulk Density**
Data Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sampling Location ID	X	X	X	X	X
Sample Type	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil	SD = SF = S = Soil
Collection Date	09/22/99	09/22/99	09/22/99	09/22/99	09/22/99
Digestion Date	Not Analyzed	03/10/00	11/15/99	12/14/99	
Analysis Date	Battelle Lab	03/10/00	11/19/99	12/14/99	
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.				
Digestion Volume		4.386, 4.482	0.025 g	0.025 g	80
Total Volume		200ul	0.025 g	0.025 g	80
Sample Volume Analyzed		aliquot	Aliquot	0.025 g	80
Dilution		See Worksheets	1	NA	No Data Reviewed
Results		0.51	275	78.3	
Measuring Unit	ppm	ng/g	ug/g	%	g/mL
EPA Method	CVAF	CVAF	EPA 365.1	ASTM D2974-87	ASTM D4531-86
Analyst		JL	Angel	SG/JL	SG/JL
Method Detection limit		0.2 ng/g	0.06	0	0.001 g/mL
Data Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes (FTN Associates)	Yes (FTN Associates)	
All Calculation Checked		Yes (FTN Associates)	Yes (FTN Associates)	Yes (FTN Associates)	
Holding Time Met	28 days	No (Goal Only)	No (Goal Only)	NA	
QC Report (Attached)					
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
QC Batch ID		31000	EPAM5-A	12/14/99	
Method Blanks		NA	NA in Soils	NA	
Instrument Blanks		All Good	"NR"	NA	
Duplicates (RPD)		131.1	<20 RPD	None Reported	
Matrix Spike Recoveries (75-125)		137.88%	All Good	NA	
Calibration % R (high and low range)		191.96%	Good	NA	
Detection Range		0.2 ng/g and >	60 ppm and >	NA	
Correlation Coefficient (>0.995)		0.9831	0.995	NA	
QC Report (Verified)					
Lab Data Entry Checked by Analyst		SOP	Yes	Yes	
All Calculation Checked		Yes	X	X	
QC Limits Met	Battelle Lab	"R2", "M", "DQO"	"B (NR)"	"DQO (NR)"	No Data Reviewed

Notes

No descriptive narratives were provided by FIU.

Battelle Laboratory has been assigned to be the main laboratory for Total Hg in soils

Total Phosphorus and MEHG were digested past the holding time goals. Holding time is a goal only.

Bulk Density data was not provided for review or reporting.

Station ID M5-944-SDF

	Total Hg	MeHg	Total Phosphorus	AFDW	Bulk Density
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed				
Total Samples/Matrix					
Methods/Parameters					
Range of Samples analyzed					
Holding Time Summary					
Analytical Problems					
QA/QC Acceptance Limits					
Integrity of Data Quality					
Deviations From SOP					
Observations					
Sample Management Records					
Sampling Location ID	X	X	X	X	
Matrix	X	X	X	X	
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted	Not Noted	
Collection Data/Time	X	X	X	X	
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID				
Sample Handling/Storage		Hg lab	Not Noted	Not Noted	
Log-in Procedures	Internal COC	Internal COC	Internal COC	Internal COC	
Raw Data (Attached)					
Sample Work Sheets		X	X	X	
Sample Run Logs		X	X	X	
Instrument Raw Data		X	X	NA	
Bench Sheets		X	X	X	
Sample Preparation Logs		X	X	X	
Raw Data (Verified)					
Sample ID Transferred		X	X	X	
All Calculation Checked		X	X	X	
Measuring Unit		ng/g	ug/g	%	g/mL
PE Results (Attached)					
Organization	NA	NA	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA	NA	NA
Validation Criteria					
Applied Qualifiers		"R2", "M", "DQO"	"B (NR)"	"DQO (NR)"	

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated
"R2" Correlation Coefficient is out of QAPP limits.
"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.
"B" Analyte concentration in the associated blank was >3 times the MDL.
"H" Analysis digestion performed after holding times have expired.
"NR" Data was unavailable for review.
"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

10 % Recalculated Results for Total Mercury in Fish Tissue

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-13-00 Checked by NJS 4-14-00

Sample	QC Batch	Qualifier Note	Instrument Reading Peak Height	Slope	Hg Concentration (ppt)	Vol. added (mL)	Average Reagent Blank (ng)	Corrected Hg Concentration (ng)	Weight of fish (g)	Hg Concentration ppb	Averaged Result ppb	Averaged Reported Result (ppb)	Detection Limit/*3 ppb	SPK CONC (ppb)	R%	Standard Deviation	Relative Standard Deviation
M5-622FSF-1	HG21JF1	r^2=0.9989	126.6	0.3961	319.6	1	0.1925	19.94	0.2070	96.34			3.2/9.6				
	"		125.9	0.3961	317.8	1	0.1925	19.83	0.2070	95.81	96.08		3.2/9.6				
M5-622FSF-2	"		122.6	0.3961	309.5	0.5	0.1925	37.88	0.2109	179.60			3.2/9.6				
	"		122	0.3961	308.0	0.5	0.1925	37.69	0.2109	178.72	179.16		3.2/9.6				
M5-622FSF-3	"		117.6	0.3961	296.9	0.5	0.1925	36.33	0.2712	133.94			3.2/9.6				
	"		119	0.3961	300.4	0.5	0.1925	36.76	0.2712	135.55	134.75		3.2/9.6				
M5-622FSF-4	"		106.4	0.3961	268.6	1	0.1925	16.73	0.1219	137.25			3.2/9.6				
	"		103.3	0.3961	260.8	1	0.1925	16.24	0.1219	133.20	135.23		3.2/9.6				
M5-622FSF-5	"		124.2	0.3961	313.6	0.5	0.1925	38.38	0.1490	257.55			3.2/9.6				
	"		123.9	0.3961	312.8	0.5	0.1925	38.28	0.1490	256.93	257.24		3.2/9.6				
M5-622FSF-6	"		110.3	0.3961	278.5	0.5	0.1925	34.06	0.1634	208.44			3.2/9.6				
	"		110.1	0.3961	278.0	0.5	0.1925	34.00	0.1634	208.06	208.25		3.2/9.6				
M5-622FSF-7	"		128.4	0.3961	324.2	1	0.1925	20.23	0.1009	200.49			3.2/9.6				
	"		127.5	0.3961	321.9	1	0.1925	20.09	0.1009	199.07	199.78	172.93	3.2/9.6				
METHOD BLK-1	"		1.4	0.3961	3.5	1		0.22					3.2/9.6				
	"		1.5	0.3961	3.8	1		0.24			0.23		3.2/9.6				
METHOD BLK-0.5	"		0.6	0.3961	1.5	0.5		0.19					3.2/9.6				
	"		0.4	0.3961	1.0	0.5		0.12			0.16	0.193	3.2/9.6				
Instrument Blank	"		0.7	0.3961	1.8	1		0.11		0.0018	0.0018	0.0018	3.2/9.6				
DORM 1	"		39.8	0.3961	100.5	0.1	0.1925	60.40	0.0126	4793.39			3.2/9.6	4600	104.20		
DORM 1	"		40.3	0.3961	101.7	0.1	0.1925	61.16	0.0126	4853.80	4823.60	4823.60	3.2/9.6	4600	105.52	42.72	0.9
CCV-1	"		79.5	0.3961	200.7					0.2007			3.2/9.6	0.2	100.35		
CCV-2	"		79.4	0.3961	200.5					0.2005			3.2/9.6	0.2	100.23		
CCV-3	"		77.4	0.3961	195.4					0.1954			3.2/9.6	0.2	97.70		
CCV-4	"		75.8	0.3961	191.4					0.1914			3.2/9.6	0.2	95.68		
CCV-5	"		76.7	0.3961	193.6					0.1936			3.2/9.6	0.2	96.82		
CCV-6	"		77.2	0.3961	194.9					0.1949			3.2/9.6	0.2	97.45		
CCV-7	"		75.7	0.3961	191.1					0.1911			3.2/9.6	0.2	95.56		
CCV-8	"		75.8	0.3961	191.4					0.1914			3.2/9.6	0.2	95.68		
CCV-9	"		75.5	0.3961	190.6					0.1906			3.2/9.6	0.2	95.30		
CCV10-	"		75.8	0.3961	191.4					0.1914			3.2/9.6	0.2	95.68		
CCV-11	"		81.9	0.3961	206.8					0.2068			3.2/9.6	0.2	103.38		
CCV-12	"		82.1	0.3961	207.3					0.2073			3.2/9.6	0.2	103.64		
CCV-13	"		82.2	0.3961	207.5					0.2075			3.2/9.6	0.2	103.76		
CCV-14	"		82.0	0.3961	207.0					0.2070		0.196	3.2/9.6	0.2	103.51	0.0069	3.51

10 % Recalculated Results for Total Mercury in Fish Tissue

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-13-00 Checked by

Sample	QC Batch	Qualifier Note	Instrument Reading Peak Height	Slope	Hg Concentration (ppt)	Vol. added (mL)	Average Reagent Blank (ng)	Corrected Hg Concentration (ng)	Weight of fish (g)	Hg Concentration ppb	Averaged Result ppb	Averaged Reported Result (ppb)	Detection Limit/*3 ppb	SPK CONC (ppb)	R%	Standard Deviation	Relative Standard Deviation
M5-633FSF-1	HG22JF1	r^2=1.0000	25.9	0.4090	63.3	1	0.0925	3.90	0.1699	22.9			3.2/9.6				
	"		26.1	0.4090	63.8	1	0.0925	3.93	0.1699	23.1	23.03		3.2/9.6				
M5-633FSF-2	"		15.6	0.4090	38.1	0.2	0.0925	11.46	0.2727	42.0			3.2/9.6				
	"		15.9	0.4090	38.9	0.2	0.0925	11.69	0.2727	42.9	42.45		3.2/9.6				
M5-633FSF-3	"		47.1	0.4090	115.2	0.5	0.0925	14.07	0.1349	104.3			3.2/9.6				
	"		46.8	0.4090	114.4	0.5	0.0925	13.98	0.1349	103.6	103.98		3.2/9.6				
M5-633FSF-4	"		17.8	0.4090	43.5	0.5	0.0925	5.26	0.1287	40.9			3.2/9.6				
	"		17.1	0.4090	41.8	0.5	0.0925	5.05	0.1287	39.2	40.06		3.2/9.6				
M5-633FSF-5	"		11.2	0.4090	27.4	0.5	0.0925	3.28	0.1183	27.7			3.2/9.6				
	"		10.8	0.4090	26.4	0.5	0.0925	3.16	0.1183	26.7	27.18		3.2/9.6				
M5-633FSF-6	"		13.7	0.4090	33.5	1	0.0925	2.02	0.0728	27.7			3.2/9.6				
	"		13.2	0.4090	32.3	1	0.0925	1.94	0.0728	26.7	27.19		3.2/9.6				
M5-633FSF-7	"		11.3	0.4090	27.6	1	0.0925	1.65	0.0774	21.3			3.2/9.6				
	"		11.3	0.4090	27.6	1	0.0925	1.65	0.0774	21.3	21.29	40.74	3.2/9.6				
Method Blank-1	"		0.5	0.4090	1.2	1		0.08					3.2/9.6				
	"		0.3	0.4090	0.7	1		0.05			0.06		3.2/9.6				
Method Blank-0.5	"		0.6	0.4090	1.5	0.5		0.18					3.2/9.6				
	"		0.2	0.4090	0.5	0.5		0.06			0.12	0.091	3.2/9.6				
Instrument Blank	"		0.5	0.4090	1.2	1		0.08		0.0012	0.0012	0.0012	3.2/9.6				
DORM 3		"M"	71.5	0.4090	174.8	0.1	0.0925	105.32	0.0125	8425.8							
DORM 3	"	"M"	25.6	0.4090	62.6	0.1	0.0925	37.65	0.0125	3012.0	5718.89	5718.889	3.2/9.6	4600	65.48	3828.09	66.9
CCV-1	"		82.1	0.4090	200.7					0.2007			3.2/9.6	0.2	100.37		
CCV-2	"		81.4	0.4090	199.0					0.1990			3.2/9.6	0.2	99.51		
CCV-3	"		82	0.4090	200.5					0.2005			3.2/9.6	0.2	100.24		
CCV-4	"		81.7	0.4090	199.8					0.1998			3.2/9.6	0.2	99.88		
CCV-5	"		79.7	0.4090	194.9					0.1949			3.2/9.6	0.2	97.43		
CCV-6	"		79.9	0.4090	195.4					0.1954			3.2/9.6	0.2	97.68		
CCV-7	"		80.3	0.4090	196.3					0.1963			3.2/9.6	0.2	98.17		
CCV-8	"		81.1	0.4090	198.3					0.1983			3.2/9.6	0.2	99.14		
CCV-9	"		81.2	0.4090	198.5					0.1985			3.2/9.6	0.2	99.27		
CCV-10	"		81.4	0.4090	199.0					0.1990			3.2/9.6	0.2	99.51		
CCV-11	"		81.7	0.4090	199.8					0.1998			3.2/9.6	0.2	99.88		
CCV-12	"		81.9	0.4090	200.2					0.2002			3.2/9.6	0.2	100.12		
CCV-13	"		79.3	0.4090	193.9					0.1939			3.2/9.6	0.2	96.94		
CCV-14	"		79.4	0.4090	194.1					0.1941			3.2/9.6	0.2	97.07		
CCV-15	"		76.3	0.4090	186.6					0.1866			3.2/9.6	0.2	93.28		
CCV-16	"		77.8	0.4090	190.2					0.1902			3.2/9.6	0.2	95.11		
CCV-17	"		78.7	0.4090	192.4					0.1924			3.2/9.6	0.2	96.21		
CCV-18	"		80.1	0.4090	195.8					0.1958			3.2/9.6	0.2	97.92		
CCV-19	"		80.1	0.4090	195.8					0.1958			3.2/9.6	0.2	97.92		
CCV-20	"		81.5	0.4090	199.3					0.1993			3.2/9.6	0.2	99.63		
CCV-21	"		79.4	0.4090	194.1					0.1941			3.2/9.6	0.2	97.07		
CCV-22	"		79.7	0.4090	194.9					0.1949		0.196	3.2/9.6	0.2	97.43	0.0036	1.85

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

10 % Recalculated Results for Total Mercury in Fish Tissue

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-13-00 Checked by NJS 4-14-00

Sample	QC Batch	Qualifier Note	Instrument Reading Peak Height	Slope	Hg Concentration (ppt)	Vol. added (mL)	Average Reagent Blank (ng)	Corrected Hg Concentration (ng)	Weight of fish (g)	Hg Concentration ppb	Averaged Result ppb	Averaged Reported Result (ppb)	Detection Limit/*3 ppb	SPK CONC (ppb)	R%	Standard Deviation	Relative Standard Deviation
M5-643FSF-1	HG26JF1	r^2=0.9998	3.1	0.3912	7.9	0.2	0.0425	2.36	0.567	4.2			3.2/9.6				
	"		3.1	0.3912	7.9	0.2	0.0425	2.36	0.567	4.2	4.16		3.2/9.6				
M5-643FSF-2	"		31.5	0.3912	80.5	0.2	0.0425	24.36	0.2718	89.6			3.2/9.6				
	"		31.1	0.3912	79.5	0.2	0.0425	24.05	0.2718	88.5	89.04		3.2/9.6				
M5-643FSF-3	"		25.2	0.3912	64.4	0.5	0.0425	7.88	0.1505	52.4			3.2/9.6				
	"		25	0.3912	63.9	0.5	0.0425	7.82	0.1505	51.9	52.16		3.2/9.6				
M5-643FSF-4	"		15.1	0.3912	38.6	1	0.0425	2.39	0.087	27.5			3.2/9.6				
	"		15.3	0.3912	39.1	1	0.0425	2.42	0.087	27.8	27.65		3.2/9.6				
M5-643FSF-5	"		71.2	0.3912	182.0	1	0.0425	11.42	0.1084	105.4			3.2/9.6				
	"		70.8	0.3912	181.0	1	0.0425	11.36	0.1084	104.8	105.09		3.2/9.6				
M5-643FSF-6	"		5.6	0.3912	14.3	1	0.0425	0.86	0.0681	12.6			3.2/9.6				
	"		5.8	0.3912	14.8	1	0.0425	0.89	0.0681	13.1	12.86		3.2/9.6				
M5-643FSF-7	"		4.3	0.3912	11.0	0.5	0.0425	1.31	0.0574	22.8			3.2/9.6				
	"		4.5	0.3912	11.5	0.5	0.0425	1.37	0.0574	23.9	23.36	44.90	3.2/9.6				
METHOD BLK-1	"		0.50	0.3912	1.3	1		0.08					3.2/9.6				
	"		0.40	0.3912	1.0	1		0.06			0.07		3.2/9.6				
METHOD BLK-0.5	"		0.10	0.3912	0.3	0.5		0.03					3.2/9.6				
	"		0.00	0.3912	0.0	0.5		0.00			0.02	0.044	3.2/9.6				
Instrument Blank	"		0.2	0.3912	0.5	1		0.03		0.00051	0.0005	0.0005	3.2/9.6				
DORM 7			38.8	0.3912	99.2	0.1	0.0425	59.76	0.0127	4705.85							
DORM 7	"		39.7	0.3912	101.5	0.1	0.0425	61.15	0.0127	4815.08	4760.46	4760.46	3.2/9.6	4600	104.68	77.24	1.6
CCV-1	"		77.4	0.3912	197.9					0.1979			3.2/9.6	0.2	98.93		
CCV-2	"		77.5	0.3912	198.1					0.1981			3.2/9.6	0.2	99.05		
CCV-3	"		75.8	0.3912	193.8					0.1938			3.2/9.6	0.2	96.88		
CCV-4	"		77.4	0.3912	197.9					0.1979			3.2/9.6	0.2	98.93		
CCV-5	"		73.3	0.3912	187.4					0.1874			3.2/9.6	0.2	93.69		
CCV-6	"		75.1	0.3912	192.0					0.1920			3.2/9.6	0.2	95.99		
CCV-7	"		76.5	0.3912	195.6					0.1956			3.2/9.6	0.2	97.78		
CCV-8	"		74.7	0.3912	191.0					0.1910			3.2/9.6	0.2	95.48		
CCV-9	"		74.5	0.3912	190.4					0.1904			3.2/9.6	0.2	95.22		
CCV10-	"		73.7	0.3912	188.4					0.1884			3.2/9.6	0.2	94.20		
CCV-11	"		75.9	0.3912	194.0					0.1940			3.2/9.6	0.2	97.01		
CCV-12	"		75.9	0.3912	194.0					0.1940			3.2/9.6	0.2	97.01		
CCV-13	"		82.2	0.3912	210.1					0.2101			3.2/9.6	0.2	105.06		
CCV-14	"		82.3	0.3912	210.4					0.2104			3.2/9.6	0.2	105.19		
CCV-15	"		84.1	0.3912	215.0					0.2150			3.2/9.6	0.2	107.49		
CCV-16	"		84.2	0.3912	215.2					0.2152			3.2/9.6	0.2	107.62		
CCV-17	"		83.9	0.3912	214.5					0.2145			3.2/9.6	0.2	107.23		
CCV-18	"		82.7	0.3912	211.4					0.2114			3.2/9.6	0.2	105.70		
CCV-19	"		79.5	0.3912	203.2					0.2032			3.2/9.6	0.2	101.61		
CCV-20	"		80.4	0.3912	205.5					0.2055			3.2/9.6	0.2	102.76		
CCV-21	"		78.1	0.3912	199.6					0.1996			3.2/9.6	0.2	99.82		
CCV-22	"		79.9	0.3912	204.2					0.2042			3.2/9.6	0.2	102.12		
CCV-23	"		78.5	0.3912	200.7					0.2007			3.2/9.6	0.2	100.33		
CCV-24	"		76.9	0.3912	196.6					0.1966		0.200	3.2/9.6	0.2	98.29	0.0087	4.36

RR = Rerun

10 % Recalculated Results for Total Mercury in Fish Tissue

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-14-00 Checked by

Sample	QC Batch	Qualifier Note	Instrument Reading Peak Height	Y Intercept Slope	Hg Concentration (ppt)	Vol. added (mL)	Average Reagent Blank (ng)	Corrected Hg Concentration (ng)	Weight of fish (g)	Hg Concentration ppb	Averaged Result ppb	Averaged Reported Result (ppb)	Detection Limit/*3 ppb	SPK CONC (ppb)	R%	Standard Deviation	Relative Standard Deviation
M5-653FSF-1	HG28JF1	r^2=0.9982	3.3	0.4039	8.2	1	0.1225	0.39	0.0926	4.2			3.2/9.6				
	"		2.9	0.4039	7.2	1	0.1225	0.33	0.0926	3.6	3.90		3.2/9.6				
M5-653FSF-2	"		5.5	0.4039	13.6	1	0.1225	0.74	0.1051	7.0			3.2/9.6				
	"		5	0.4039	12.4	1	0.1225	0.66	0.1051	6.3	6.63		3.2/9.6				
M5-653FSF-3	"		4.1	0.4039	10.2	1	0.1225	0.52	0.0923	5.6			3.2/9.6				
	"		3.9	0.4039	9.7	1	0.1225	0.49	0.0923	5.3	5.43		3.2/9.6				
M5-653FSF-4	"		8.9	0.4039	22.0	1	0.1225	1.27	0.1377	9.2			3.2/9.6				
	"		7.6	0.4039	18.8	1	0.1225	1.06	0.1377	7.7	8.46		3.2/9.6				
M5-653FSF-5	"		3.4	0.4039	8.4	1	0.1225	0.41	0.0722	5.6			3.2/9.6				
	"		3.2	0.4039	7.9	1	0.1225	0.38	0.0722	5.2	5.43		3.2/9.6				
M5-653FSF-6	"		4.6	0.4039	11.4	1	0.1225	0.60	0.0728	8.2			3.2/9.6				
	"		4.4	0.4039	10.9	1	0.1225	0.56	0.0728	7.7	7.96		3.2/9.6				
M5-653FSF-7	"		4.4	0.4039	10.9	1	0.1225	0.56	0.0533	10.6			3.2/9.6				
	"		3.7	0.4039	9.2	1	0.1225	0.45	0.0533	8.5	9.55	6.77	3.2/9.6				
METHOD BLK-1	"		0.3	0.4039	0.7	1		0.05					3.2/9.6				
	"		0.3	0.4039	0.7	1		0.05			0.05		3.2/9.6				
METHOD BLK-0.5	"		0.7	0.4039	1.7	0.5		0.21					3.2/9.6				
	"		0.6	0.4039	1.5	0.5		0.18			0.20	0.122	3.2/9.6				
Instrument Blank	"		0.7	0.4039	1.7	1		0.11		0.0017	0.0017	0.0017	3.2/9.6				
DORM 14	"		39	0.4039	96.6	0.1	0.1225	58.10	0.011	5282.03			3.2/9.6	4600	114.83		
DORM 14	"		38.5	0.4039	95.3	0.1	0.1225	57.36	0.011	5214.17	5248.10	5248.10	3.2/9.6	4600	113.35	47.99	0.9
CCV-1	"		74.5	0.4039	184.5					0.1845			3.2/9.6	0.2	92.23		
CCV-2	"		75.7	0.4039	187.4					0.1874			3.2/9.6	0.2	93.71		
CCV-3	"		73.1	0.4039	181.0					0.1810			3.2/9.6	0.2	90.49		
CCV-4	"		73.2	0.4039	181.2					0.1812			3.2/9.6	0.2	90.62		
CCV-5	"		76.2	0.4039	188.7					0.1887			3.2/9.6	0.2	94.33		
CCV-6	"		75.4	0.4039	186.7					0.1867			3.2/9.6	0.2	93.34		
CCV-7	"		75	0.4039	185.7					0.1857			3.2/9.6	0.2	92.84		
CCV-8	"		74.9	0.4039	185.4					0.1854			3.2/9.6	0.2	92.72		
CCV-9	"		70.9	0.4039	175.5					0.1755			3.2/9.6	0.2	87.77		
CCV10-	"		71.2	0.4039	176.3					0.1763			3.2/9.6	0.2	88.14		
CCV-11	"		74.4	0.4039	184.2					0.1842			3.2/9.6	0.2	92.10		
CCV-12	"		74.2	0.4039	183.7					0.1837			3.2/9.6	0.2	91.85		
CCV-13	"		74.4	0.4039	184.2					0.1842			3.2/9.6	0.2	92.10		
CCV-14	"		74.4	0.4039	184.2					0.1842			3.2/9.6	0.2	92.10		
CCV-15	"		74.1	0.4039	183.5					0.1835			3.2/9.6	0.2	91.73		
CCV-16	"		73.7	0.4039	182.5					0.1825			3.2/9.6	0.2	91.24		
CCV-17	"		74.1	0.4039	183.5					0.1835			3.2/9.6	0.2	91.73		
CCV-18	"		74.1	0.4039	183.5					0.1835			3.2/9.6	0.2	91.73		
CCV-19	"		73.8	0.4039	182.7					0.1827			3.2/9.6	0.2	91.36		
CCV-20	"		74.6	0.4039	184.7					0.1847			3.2/9.6	0.2	92.35		
CCV-21	"		73.4	0.4039	181.7					0.1817			3.2/9.6	0.2	90.86		
CCV-22	"		74.1	0.4039	183.5					0.1835		0.183	3.2/9.6	0.2	91.73	0.0031	1.67

RR = Rerun

10 % Recalculated Results for Total Mercury in Fish Tissue

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-14-00 Checked by NJS 4-14-00

Sample	QC Batch	Qualifier Note	Instrument Reading Peak Height	Slope	Hg Concentration (ppt)	Vol. added (mL)	Average Reagent Blank (ng)	Corrected Hg Concentration (ng)	Weight of fish (g)	Hg Concentration ppb	Averaged Result ppb	Averaged Reported Result (ppb)	Detection Limit/*3 ppb	SPK CONC (ppb)	R%	Standard Deviation	Relative Standard Deviation
M5-663FSF-1	HG28JF2	r^2=0.9990	171.8	0.4316	398.1	1	0.3325	24.74	0.1356	182.48			3.2/9.6				
	"		172.4	0.4316	399.4	1	0.3325	24.83	0.1356	183.13	182.81		3.2/9.6				
M5-663FSF-2	"		127.1	0.4316	294.5	1	0.3325	18.22	0.112	162.68			3.2/9.6				
	"		133.7	0.4316	309.8	1	0.3325	19.18	0.112	171.28	166.98		3.2/9.6				
M5-663FSF-3	"		88.5	0.4316	205.1	1	0.3325	12.59	0.1073	117.29			3.2/9.6				
	"		89.5	0.4316	207.4	1	0.3325	12.73	0.1073	118.65	117.97		3.2/9.6				
M5-663FSF-4	"		86	0.4316	199.3	1	0.3325	12.22	0.1079	113.26			3.2/9.6				
	"		87.7	0.4316	203.2	1	0.3325	12.47	0.1079	115.56	114.41		3.2/9.6				
M5-663FSF-5	"		67.4	0.4316	156.2	1	0.3325	9.51	0.0964	98.61			3.2/9.6				
	"		67.2	0.4316	155.7	1	0.3325	9.48	0.0964	98.30	98.46		3.2/9.6				
M5-663FSF-6	"		69.8	0.4316	161.7	0.5	0.3325	19.56	0.1626	120.29			3.2/9.6				
	"		62.5	0.4316	144.8	0.5	0.3325	17.48	0.1626	107.50	113.90	132.42	3.2/9.6				
METHOD BLK-1	"		1.2	0.4316	2.8	1		0.18					3.2/9.6				
	"		1	0.4316	2.3	1		0.15			0.16		3.2/9.6				
METHOD BLK-0.5	"		2.1	0.4316	4.9	0.5		0.60					3.2/9.6				
	"		1.4	0.4316	3.2	0.5		0.40			0.50	0.330	3.2/9.6				
Instrument Blank	"		0.65	0.4316	1.5	1		0.09		0.0015	0.0015	0.0015	3.2/9.6				
DORM 15	"		38.6	0.4316	89.4	0.1	0.3325	53.60	0.0109	4917.1			3.2/9.6	4600	106.89		
DORM 15	"		37.8	0.4316	87.6	0.1	0.3325	52.48	0.0114	4603.4	4760.27	4760.27	3.2/9.6	4600	100.07	221.82	4.7
CCV-1	"		89.7	0.4316	207.8					0.2078			3.2/9.6	0.2	103.92		
CCV-2	"		91.8	0.4316	212.7					0.2127			3.2/9.6	0.2	106.35		
CCV-3	"		82.3	0.4316	190.7					0.1907			3.2/9.6	0.2	95.34		
CCV-4	"		88.6	0.4316	205.3					0.2053			3.2/9.6	0.2	102.64		
CCV-5	"		88.3	0.4316	204.6					0.2046			3.2/9.6	0.2	102.29		
CCV-6	"		89.3	0.4316	206.9					0.2069			3.2/9.6	0.2	103.45		
CCV-7	"		80.1	0.4316	185.6					0.1856			3.2/9.6	0.2	92.79		
CCV-8	"		88.8	0.4316	205.7					0.2057			3.2/9.6	0.2	102.87		
CCV-9	"		90.4	0.4316	209.5					0.2095			3.2/9.6	0.2	104.73		
CCV-10	"		89.7	0.4316	207.8					0.2078			3.2/9.6	0.2	103.92		
CCV-11	"		77.5	0.4316	179.6					0.1796			3.2/9.6	0.2	89.78		
CCV-12	"		86.2	0.4316	199.7					0.1997			3.2/9.6	0.2	99.86		
CCV-13	"		87.3	0.4316	202.3					0.2023			3.2/9.6	0.2	101.14		
CCV-14	"		86.4	0.4316	200.2					0.2002			3.2/9.6	0.2	100.09		
CCV-15	"		77.5	0.4316	179.6					0.1796			3.2/9.6	0.2	89.78		
CCV-16	"		86	0.4316	199.3					0.1993			3.2/9.6	0.2	99.63		
CCV-17	"		84.7	0.4316	196.2					0.1962			3.2/9.6	0.2	98.12		
CCV-18	"		85.6	0.4316	198.3					0.1983			3.2/9.6	0.2	99.17		
CCV-19	"		77.7	0.4316	180.0					0.1800			3.2/9.6	0.2	90.01		
CCV-20	"		84.2	0.4316	195.1					0.1951			3.2/9.6	0.2	97.54		
CCV-21	"		85.6	0.4316	198.3					0.1983			3.2/9.6	0.2	99.17		
CCV-22	"		85.5	0.4316	198.1					0.1981		0.198	3.2/9.6	0.2	99.05	0.0098	4.95

10 % Recalculated Results for Total Mercury in Fish Tissue

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-14-00 Checked by NJS 4-14-00

Sample	QC Batch	Qualifier Note	Instrument Reading Peak Height	Slope	Hg Concentration (ppt)	Vol. added (mL)	Average Reagent Blank (ng)	Corrected Hg Concentration (ng)	Weight of fish (g)	Hg Concentration ppb	Averaged Result ppb	Averaged Reported Result (ppb)	Detection Limit/*3 ppb	SPK CONC (ppb)	R%	Standard Deviation	Relative Standard Deviation
M5-673FSF-1	HG29JF1	r^2=0.9993	139.5	0.4124	338.3	0.5	0.3325	41.27	0.1786	231.10			3.2/9.6				
	"		141	0.4124	341.9	0.5	0.3325	41.72	0.1786	233.60	232.35		3.2/9.6				
M5-673FSF-2	"		95.7	0.4124	232.1	0.5	0.3325	28.21	0.1504	187.57			3.2/9.6				
	"		105.5	0.4124	255.8	0.5	0.3325	31.13	0.1504	207.00	197.29		3.2/9.6				
M5-673FSF-3	"		90.7	0.4124	219.9	1	0.3325	13.52	0.0999	135.37			3.2/9.6				
	"		90.8	0.4124	220.2	1	0.3325	13.54	0.0999	135.52	135.44		3.2/9.6				
M5-673FSF-4	"		44.5	0.4124	107.9	0.2	0.3325	32.36	0.1894	170.87			3.2/9.6				
	"		47.3	0.4124	114.7	0.2	0.3325	34.42	0.1894	181.73	176.30		3.2/9.6				
M5-673FSF-5	"		137.5	0.4124	333.4	1	0.3325	20.67	0.1001	206.52			3.2/9.6				
	"		137.4	0.4124	333.2	1	0.3325	20.66	0.1001	206.37	206.44		3.2/9.6				
M5-673FSF-6	"		61.7	0.4124	149.6	0.5	0.3325	18.07	0.1476	122.42			3.2/9.6				
	"		67.7	0.4124	164.2	0.5	0.3325	19.86	0.1476	134.55	128.49		3.2/9.6				
M5-673FSF-7	"		59.2	0.4124	143.5	1	0.3325	8.71	0.0764	114.02			3.2/9.6				
	"		59.5	0.4124	144.3	1	0.3325	8.76	0.0764	114.62	114.32	170.09	3.2/9.6				
METHOD BLK-1	"		0.9	0.4124	2.2	1		0.14					3.2/9.6				
	"		0.7	0.4124	1.7	1		0.11			0.12		3.2/9.6				
METHOD BLK-0.5	"		0.6	0.4124	1.5	0.5		0.18					3.2/9.6				
	"		0.6	0.4124	1.5	0.5		0.18			0.18	0.151	3.2/9.6				
Instrument Blank	"		0.15	0.4124	0.4	1		0.02		0.0004	0.0004	0.0004	3.2/9.6				
DORM 22	"		32.7	0.4124	79.3	0.1	0.3325	47.48	0.0126	3768.3			3.2/9.6	4600	81.92		
DORM 22	"		34.9	0.4124	84.6	0.1	0.3325	50.70	0.0126	4023.6	3895.95	3895.95	3.2/9.6	4600	87.47	180.52	4.6
CCV-1	"		83.4	0.4124	202.2					0.2022			3.2/9.6	0.2	101.12		
CCV-2	"		83.7	0.4124	203.0					0.2030			3.2/9.6	0.2	101.48		
CCV-3	"		74.9	0.4124	181.6					0.1816			3.2/9.6	0.2	90.81		
CCV-4	"		82.8	0.4124	200.8					0.2008			3.2/9.6	0.2	100.39		
CCV-5	"		80.8	0.4124	195.9					0.1959			3.2/9.6	0.2	97.96		
CCV-6	"		80.4	0.4124	195.0					0.1950			3.2/9.6	0.2	97.48		
CCV-7	"		74.2	0.4124	179.9					0.1799			3.2/9.6	0.2	89.96		
CCV-8	"		80.7	0.4124	195.7					0.1957			3.2/9.6	0.2	97.84		
CCV-9	"		83.2	0.4124	201.7					0.2017			3.2/9.6	0.2	100.87		
CCV-10	"		82.4	0.4124	199.8					0.1998			3.2/9.6	0.2	99.90		
CCV-11	"		77.8	0.4124	188.7					0.1887			3.2/9.6	0.2	94.33		
CCV-12	"		83	0.4124	201.3					0.2013			3.2/9.6	0.2	100.63		
CCV-13	"		82.2	0.4124	199.3					0.1993			3.2/9.6	0.2	99.66		
CCV-14	"		82.7	0.4124	200.5					0.2005			3.2/9.6	0.2	100.27		
CCV-15	"		77.3	0.4124	187.4					0.1874			3.2/9.6	0.2	93.72		
CCV-16	"		83	0.4124	201.3					0.2013		0.196	3.2/9.6	0.2	100.63	0.0075	3.82

RR = Rerun

10 % Recalculated Results for Total Mercury in Fish Tissue

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-14-00 Checked by NJS 4-14-00

Sample	QC Batch	Qualifier Note	Instrument Reading Peak Height	Slope	Hg Concentration (ppt)	Vol. added (mL)	Average Reagent Blank (ng)	Corrected Hg Concentration (ng)	Weight of fish (g)	Hg Concentration ppb	Averaged Result ppb	Averaged Reported Result (ppb)	Detection Limit/*3 ppb	SPK CONC (ppb)	R%	Standard Deviation	Relative Standard Deviation
M5-683FSF-1	HG01KF1	r^2=0.9998	118.1	0.4151	284.5	0.5	0.3325	34.66	0.1167	297.02			3.2/9.6				
	"		117	0.4151	281.9	0.5	0.3325	34.34	0.1167	294.23	295.62		3.2/9.6				
M5-683FSF-2	"		96.6	0.4151	232.7	0.5	0.3325	28.29	0.1212	233.43			3.2/9.6				
	"		96.3	0.4151	232.0	0.5	0.3325	28.20	0.1212	232.69	233.06		3.2/9.6				
M5-683FSF-3	"		86.8	0.4151	209.1	0.2	0.3325	63.03	0.1936	325.55			3.2/9.6				
	"		87.3	0.4151	210.3	0.2	0.3325	63.39	0.1936	327.44	326.49		3.2/9.6				
M5-683FSF-4	"		110.4	0.4151	266.0	0.5	0.3325	32.38	0.1369	236.53			3.2/9.6				
	"		109.5	0.4151	263.8	0.5	0.3325	32.11	0.1369	234.58	235.55		3.2/9.6				
M5-683FSF-5	"		42.8	0.4151	103.1	0.5	0.3325	12.35	0.0867	142.44			3.2/9.6				
	"		41.9	0.4151	100.9	0.5	0.3325	12.08	0.0867	139.37	140.90		3.2/9.6				
M5-683FSF-6	"		65.5	0.4151	157.8	1	0.3325	9.61	0.0917	104.78			3.2/9.6				
	"		66.4	0.4151	160.0	1	0.3325	9.75	0.0917	106.27	105.53		3.2/9.6				
M5-683FSF-7	"		105.2	0.4151	253.4	1	0.3325	15.63	0.0804	194.45			3.2/9.6				
	"		105.2	0.4151	253.4	1	0.3325	15.63	0.0804	194.45	194.45	218.80	3.2/9.6				
METHOD BLK-1	"		0.5	0.4151	1.20	1		0.08					3.2/9.6				
	"		0.4	0.4151	0.96	1		0.06			0.07		3.2/9.6				
METHOD BLK-0.5	"		0.3	0.4151	0.72	0.5		0.09					3.2/9.6				
	"		0.2	0.4151	0.48	0.5		0.06			0.07	0.071	3.2/9.6				
Instrument Blank	"		0.2	0.4151	0.48	1		0.03		0.0005	0.0005	0.0005	3.2/9.6				
DORM 26	"		37.4	0.4151	90.1	0.1	0.3325	54.00	0.0109	4953.9			3.2/9.6	4600	107.69		
DORM 26	"		37.7	0.4151	90.8	0.1	0.3325	54.43	0.0109	4993.8	4973.85	4973.85	3.2/9.6	4600	108.56	28.27	0.6
CCV-1	"		89.2	0.4151	214.9					0.2149			3.2/9.6	0.2	107.44		
CCV-2	"		88.2	0.4151	212.5					0.2125			3.2/9.6	0.2	106.24		
CCV-3	"		86.7	0.4151	208.9					0.2089			3.2/9.6	0.2	104.43		
CCV-4	"		89	0.4151	214.4					0.2144			3.2/9.6	0.2	107.20		
CCV-5	"		89	0.4151	214.4					0.2144			3.2/9.6	0.2	107.20		
CCV-6	"		88.7	0.4151	213.7					0.2137			3.2/9.6	0.2	106.84		
CCV-7	"		88.8	0.4151	213.9					0.2139			3.2/9.6	0.2	106.96		
CCV-8	"		88.5	0.4151	213.2					0.2132			3.2/9.6	0.2	106.60		
CCV-9	"		87.2	0.4151	210.1					0.2101			3.2/9.6	0.2	105.03		
CCV-10	"		88.4	0.4151	213.0					0.2130			3.2/9.6	0.2	106.48		
CCV-11	"		82.4	0.4151	198.5					0.1985			3.2/9.6	0.2	99.25		
CCV-12	"		89.3	0.4151	215.1					0.2151			3.2/9.6	0.2	107.56		
CCV-13	"		86.9	0.4151	209.3					0.2093			3.2/9.6	0.2	104.67		
CCV-14	"		87.7	0.4151	211.3					0.2113			3.2/9.6	0.2	105.64		
CCV-15	"		88.1	0.4151	212.2					0.2122			3.2/9.6	0.2	106.12		
CCV-16	"		87.1	0.4151	209.8					0.2098			3.2/9.6	0.2	104.91		
CCV-17	"		85.9	0.4151	206.9					0.2069			3.2/9.6	0.2	103.47		
CCV-18	"		86	0.4151	207.2					0.2072			3.2/9.6	0.2	103.59		
CCV-19	"		85.7	0.4151	206.5					0.2065			3.2/9.6	0.2	103.23		
CCV-20	"		85.7	0.4151	206.5					0.2065		0.211	3.2/9.6	0.2	103.23	0.0041	1.94

RR = Rerun

10 % Recalculated Results for Total Mercury in Fish Tissue

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-14-00 Checked by NJS 4-14-00

Sample	QC Batch	Qualifier Note	Instrument Reading Peak Height	Slope	Hg Concentration (ppt)	Vol. added (mL)	Average Reagent Blank (ng)	Corrected Hg Concentration (ng)	Weight of fish (g)	Hg Concentration ppb	Averaged Result ppb	Averaged Reported Result (ppb)	Detection Limit/*3 ppb	SPK CONC (ppb)	R%	Standard Deviation	Relative Standard Deviation
M5-693FSF-1	HG02KF1	r^2=0.9996	60.2	0.4174	144.2	1	0.3325	8.75	0.0549	159.45			3.2/9.6				
	"		64.5	0.4174	154.5	1	0.3325	9.40	0.0549	171.27	165.36		3.2/9.6				
M5-693FSF-2	"		69.7	0.4174	167.0	1	0.0875	10.43	0.0425	245.47			3.2/9.6				
	"		70	0.4174	167.7	1	0.0875	10.48	0.0425	246.54	246.01		3.2/9.6				
M5-693FSF-3	"		30.2	0.4174	72.4	1	0.0875	4.47	0.0373	119.86			3.2/9.6				
	"		31.6	0.4174	75.7	1	0.0875	4.68	0.0373	125.52	122.69		3.2/9.6				
M5-693FSF-4	"		5.5	0.4174	13.2	1	0.0875	0.74	0.0095	78.17			3.2/9.6				
	"		5.6	0.4174	13.4	1	0.0875	0.76	0.0095	79.76	78.97		3.2/9.6				
M5-693FSF-5	"		68.5	0.4174	164.1	1	0.0875	10.25	0.0608	168.61			3.2/9.6				
	"		74.5	0.4174	178.5	1	0.0875	11.16	0.0608	183.51	176.06		3.2/9.6				
M5-693FSF-6	"		37.1	0.4174	88.9	1	0.0875	5.51	0.0397	138.85			3.2/9.6				
	"		37.2	0.4174	89.1	1	0.0875	5.53	0.0397	139.23	139.04		3.2/9.6				
M5-693FSF-7	"		51.8	0.4174	124.1	1	0.0875	7.73	0.0699	110.60			3.2/9.6				
	"		54.6	0.4174	130.8	1	0.0875	8.15	0.0699	116.65	113.62	148.82	3.2/9.6				
METHOD BLK-1	"		0.5	0.4174	1.20	1		0.08					3.2/9.6				
	"		0.4	0.4174	0.96	1		0.06			0.07		3.2/9.6				
METHOD BLK-0.5	"		0.3	0.4174	0.72	0.5		0.09					3.2/9.6				
	"		0.4	0.4174	0.96	0.5		0.12			0.10	0.086	3.2/9.6				
Instrument Blank	"		0.6	0.4174	1.44	1		0.09		0.0014	0.0014	0.0014	3.2/9.6				
DORM 31	"		40.3	0.4174	96.6	0.1	0.0875	58.13	0.0126	4613.7			3.2/9.6	4600	100.30		
DORM 31	"		40	0.4174	95.8	0.1	0.0875	57.70	0.0126	4579.3	4596.47	4596.47	3.2/9.6	4600	99.55	24.32	0.5
CCV-1	"		83.8	0.4174	200.8					0.2008			3.2/9.6	0.2	100.38		
CCV-2	"		85.1	0.4174	203.9					0.2039			3.2/9.6	0.2	101.94		
CCV-3	"		79.7	0.4174	190.9					0.1909			3.2/9.6	0.2	95.47		
CCV-4	"		86	0.4174	206.0					0.2060			3.2/9.6	0.2	103.02		
CCV-5	"		86.7	0.4174	207.7					0.2077			3.2/9.6	0.2	103.86		
CCV-6	"		86.9	0.4174	208.2					0.2082			3.2/9.6	0.2	104.10		
CCV-7	"		82.3	0.4174	197.2					0.1972			3.2/9.6	0.2	98.59		
CCV-8	"		88.1	0.4174	211.1					0.2111			3.2/9.6	0.2	105.53		
CCV-9	"		89.4	0.4174	214.2					0.2142			3.2/9.6	0.2	107.09		
CCV-10	"		89.7	0.4174	214.9					0.2149			3.2/9.6	0.2	107.45		
CCV-11	"		88.2	0.4174	211.3					0.2113			3.2/9.6	0.2	105.65		
CCV-12	"		91.3	0.4174	218.7					0.2187			3.2/9.6	0.2	109.37		
CCV-13	"		95.4	0.4174	228.6					0.2286			3.2/9.6	0.2	114.28		
CCV-14	"		95.3	0.4174	228.3					0.2283			3.2/9.6	0.2	114.16		
CCV-15	"		86.5	0.4174	207.2					0.2072			3.2/9.6	0.2	103.62		
CCV-16	"		95	0.4174	227.6					0.2276		0.211	3.2/9.6	0.2	113.80	0.0109	5.15

10 % Recalculated Results for Total Mercury in Fish Tissue

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-14-00 Checked by NJS 4-14-00

Sample	QC Batch	Qualifier Note	Instrument Reading Peak Height	Slope	Hg Concentration (ppt)	Vol. added (mL)	Average Reagent Blank (ng)	Corrected Hg Concentration (ng)	Weight of fish (g)	Hg Concentration ppb	Averaged Result ppb	Averaged Reported Result (ppb)	Detection Limit/*3 ppb	SPK CONC (ppb)	R%	Standard Deviation	Relative Standard Deviation
M5-703FSF-1	HG05KF1	r^2=0.9991	38.4	0.3956	97.1	0.1	0.1025	58.43	0.2116	276.13			3.2/9.6				
	"		38.7	0.3956	97.8	0.1	0.1025	58.89	0.2116	278.29	277.21		3.2/9.6				
M5-703FSF-2	"		46.9	0.3956	118.6	0.1	0.1025	71.39	0.223	320.11			3.2/9.6				
	"		47	0.3956	118.8	0.1	0.1025	71.54	0.223	320.80	320.46		3.2/9.6				
M5-703FSF-3	"		58.7	0.3956	148.4	0.5	0.1025	18.15	0.0806	225.17			3.2/9.6				
	"		59.1	0.3956	149.4	0.5	0.1025	18.27	0.0806	226.71	225.94		3.2/9.6				
M5-703FSF-4	"		97.7	0.3956	247.0	0.5	0.1025	30.27	0.103	293.93			3.2/9.6				
	"		97.7	0.3956	247.0	0.5	0.1025	30.27	0.103	293.93	293.93		3.2/9.6				
M5-703FSF-5	"		52.7	0.3956	133.2	0.5	0.1025	16.28	0.0736	221.24			3.2/9.6				
	"		54.7	0.3956	138.3	0.5	0.1025	16.90	0.0736	229.69	225.46		3.2/9.6				
M5-703FSF-6	"		92.5	0.3956	233.8	1	0.1025	14.63	0.0796	183.77			3.2/9.6				
	"		93.2	0.3956	235.6	1	0.1025	14.74	0.0796	185.17	184.47		3.2/9.6				
M5-703FSF-7	"		84.8	0.3956	214.4	1	0.1025	13.40	0.0849	157.86			3.2/9.6				
	"		84.5	0.3956	213.6	1	0.1025	13.35	0.0849	157.29	157.58	240.72	3.2/9.6				
METHOD BLK-1	"		0.6	0.3956	1.52	1		0.10					3.2/9.6				
	"		0.8	0.3956	2.02	1		0.13			0.11		3.2/9.6				
METHOD BLK-0.5	"		0.3	0.3956	0.76	0.5		0.09					3.2/9.6				
	"		0.3	0.3956	0.76	0.5		0.09			0.09	0.102	3.2/9.6				
Instrument Blank	"		0.35	0.3956	0.88	1		0.06		0.0009	0.0009	0.0009	3.2/9.6				
DORM 29	"		33.4	0.3956	84.4	0.1	0.1025	50.81	0.0109	4661.3			3.2/9.6	4600	101.33		
DORM 29	"		33.2	0.3956	83.9	0.1	0.1025	50.50	0.0109	4633.3	4647.30	4647.30	3.2/9.6	4600	100.72	19.78	0.4
CCV-1	"		79.2	0.3956	200.2					0.2002			3.2/9.6	0.2	100.10		
CCV-2	"		79.5	0.3956	201.0					0.2010			3.2/9.6	0.2	100.48		
CCV-3	"		85	0.3956	214.9					0.2149			3.2/9.6	0.2	107.43		
CCV-4	"		85.2	0.3956	215.4					0.2154			3.2/9.6	0.2	107.68		
CCV-5	"		83.9	0.3956	212.1					0.2121			3.2/9.6	0.2	106.04		
CCV-6	"		85	0.3956	214.9					0.2149			3.2/9.6	0.2	107.43		
CCV-7	"		85.5	0.3956	216.1					0.2161			3.2/9.6	0.2	108.06		
CCV-8	"		82.6	0.3956	208.8					0.2088			3.2/9.6	0.2	104.40		
CCV-9	"		84.8	0.3956	214.4					0.2144			3.2/9.6	0.2	107.18		
CCV-10	"		83	0.3956	209.8					0.2098			3.2/9.6	0.2	104.90		
CCV-11	"		83.6	0.3956	211.3					0.2113			3.2/9.6	0.2	105.66		
CCV-12	"		86.3	0.3956	218.1					0.2181			3.2/9.6	0.2	109.07		
CCV-13	"		84.2	0.3956	212.8					0.2128			3.2/9.6	0.2	106.42		
CCV-14	"		85.3	0.3956	215.6					0.2156			3.2/9.6	0.2	107.81		
CCV-15	"		83.3	0.3956	210.6					0.2106			3.2/9.6	0.2	105.28		
CCV-16	"		83.9	0.3956	212.1					0.2121			3.2/9.6	0.2	106.04		
CCV-17	"		82.9	0.3956	209.6					0.2096			3.2/9.6	0.2	104.78		
CCV-18	"		83.6	0.3956	211.3					0.2113			3.2/9.6	0.2	105.66		
CCV-19	"		83	0.3956	209.8					0.2098			3.2/9.6	0.2	104.90		
CCV-20	"		83.3	0.3956	210.6					0.2106		0.211	3.2/9.6	0.2	105.28	0.0045	2.14

10 % Recalculated Results for Total Mercury in Fish Tissue

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-14-00 Checked by NJS 4-14-00

Sample	QC Batch	Qualifier Note	Instrument Reading Peak Height	Slope	Hg Concentration (ppt)	Vol. added (mL)	Average Reagent Blank (ng)	Corrected Hg Concentration (ng)	Weight of fish (g)	Hg Concentration ppb	Averaged Result ppb	Averaged Reported Result (ppb)	Detection Limit/*3 ppb	SPK CONC (ppb)	R%	Standard Deviation	Relative Standard Deviation
M5-703FSF-1	HG09KF1	r^2=0.9999	35.3	0.3845	91.8	0.1	0.0900	55.27	0.2116	261.20			3.2/9.6				
	"		36.4	0.3845	94.7	0.1	0.0900	57.00	0.2116	269.35	265.28		3.2/9.6				
M5-703FSF-2	"		41.4	0.3845	107.7	0.1	0.0900	64.84	0.223	290.75			3.2/9.6				
	"		41.3	0.3845	107.4	0.1	0.0900	64.68	0.223	290.04	290.39		3.2/9.6				
M5-703FSF-3	"		58.2	0.3845	151.4	0.5	0.0900	18.53	0.0806	229.88			3.2/9.6				
	"		58	0.3845	150.8	0.5	0.0900	18.46	0.0806	229.08	229.48		3.2/9.6				
M5-703FSF-4	"		104.9	0.3845	272.8	0.5	0.0900	33.47	0.103	324.92			3.2/9.6				
	"		103.5	0.3845	269.2	0.5	0.0900	33.02	0.103	320.58	322.75		3.2/9.6				
M5-703FSF-5	"		54.5	0.3845	141.7	0.5	0.0900	17.34	0.0736	235.66			3.2/9.6				
	"		54	0.3845	140.4	0.5	0.0900	17.18	0.0736	233.48	234.57		3.2/9.6				
M5-703FSF-6	"		92.3	0.3845	240.1	1	0.0900	15.03	0.0796	188.86			3.2/9.6				
	"		93.3	0.3845	242.7	1	0.0900	15.20	0.0796	190.92	189.89	255.39	3.2/9.6				
METHOD BLK-1	"		0.5	0.3845	1.30	1		0.08					3.2/9.6				
	"		0.3	0.3845	0.78	1		0.05			0.07		3.2/9.6				
METHOD BLK-0.5	"		0.3	0.3845	0.78	0.5		0.10					3.2/9.6				
	"		0.4	0.3845	1.04	0.5		0.13			0.11	0.089	3.2/9.6				
Instrument Blank	"		0.3	0.3845	0.78	1		0.05		0.0008	0.0008	0.0008	3.2/9.6				
DORM 37	"		42.2	0.3845	109.8	0.1	0.0900	66.09	0.014	4720.8			3.2/9.6	4600	102.63		
DORM 37	"		42.6	0.3845	110.8	0.1	0.0900	66.72	0.014	4765.6	4743.19	4743.19	3.2/9.6	4600	103.60	31.68	0.7
CCV-1	"		75.7	0.3845	196.9					0.1969			3.2/9.6	0.2	98.44		
CCV-2	"		76.2	0.3845	198.2					0.1982			3.2/9.6	0.2	99.09		
CCV-3	"		82.5	0.3845	214.6					0.2146			3.2/9.6	0.2	107.28		
CCV-4	"		83.1	0.3845	216.1					0.2161			3.2/9.6	0.2	108.06		
CCV-5	"		83.6	0.3845	217.4					0.2174			3.2/9.6	0.2	108.71		
CCV-6	"		83.8	0.3845	217.9					0.2179			3.2/9.6	0.2	108.97		
CCV-7	"		82.8	0.3845	215.3					0.2153			3.2/9.6	0.2	107.67		
CCV-8	"		83.6	0.3845	217.4					0.2174			3.2/9.6	0.2	108.71		
CCV-9	"		80.7	0.3845	209.9					0.2099			3.2/9.6	0.2	104.94		
CCV-10	"		80.8	0.3845	210.1					0.2101			3.2/9.6	0.2	105.07		
CCV-11	"		73.6	0.3845	191.4					0.1914			3.2/9.6	0.2	95.71		
CCV-12	"		74.6	0.3845	194.0					0.1940			3.2/9.6	0.2	97.01		
CCV-13	"		72.3	0.3845	188.0					0.1880			3.2/9.6	0.2	94.02		
CCV-14	"		72.3	0.3845	188.0					0.1880			3.2/9.6	0.2	94.02		
CCV-15	"		72.9	0.3845	189.6					0.1896			3.2/9.6	0.2	94.80		
CCV-16	"		73.9	0.3845	192.2					0.1922			3.2/9.6	0.2	96.10		
CCV-17	"		0.7	0.3845	1.8					0.0018			3.2/9.6	0.2	0.91		
CCV-18	"		0.5	0.3845	1.3					0.0013			3.2/9.6	0.2	0.65		
CCV-19	"		0.5	0.3845	1.3					0.0013			3.2/9.6	0.2	0.65		
CCV-20	"		0.1	0.3845	0.3					0.0003			3.2/9.6	0.2	0.13		
CCV-21	"		75.8	0.3845	197.1					0.1971			3.2/9.6	0.2	98.57		
CCV-22	"		77.2	0.3845	200.8					0.2008		0.166	3.2/9.6	0.2	100.39	0.0804	48.31

10 % Recalculated Results for Total Mercury in Fish Tissue

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-14-00 Checked by NJS 4-14-00

Sample	QC Batch	Qualifier Note	Instrument Reading Peak Height	Slope	Hg Concentration (ppt)	Vol. added (mL)	Average Reagent Blank (ng)	Corrected Hg Concentration (ng)	Weight of fish (g)	Hg Concentration ppb	Averaged Result ppb	Averaged Reported Result (ppb)	Detection Limit/*3 ppb	SPK CONC (ppb)	R%	Standard Deviation	Relative Standard Deviation
M5-714FSF-1	HG05KF1	r^2=0.9991	119.5	0.3956	302.1	0.5	0.1025	37.05	0.1451	255.36			3.2/9.6				
	"		120.4	0.3956	304.3	0.5	0.1025	37.33	0.1451	257.29	256.32		3.2/9.6				
M5-714FSF-2	"		86.9	0.3956	219.7	0.5	0.1025	26.92	0.1138	236.52			3.2/9.6				
	"		86.7	0.3956	219.2	0.5	0.1025	26.85	0.1138	235.98	236.25		3.2/9.6				
M5-714FSF-3	"		71.6	0.3956	181.0	0.5	0.1025	22.16	0.1586	139.72			3.2/9.6				
	"		70.1	0.3956	177.2	0.5	0.1025	21.69	0.1586	136.78	138.25		3.2/9.6				
M5-714FSF-4	"		99.9	0.3956	252.5	0.5	0.1025	30.96	0.1611	192.17			3.2/9.6				
	"		101	0.3956	255.3	0.5	0.1025	31.30	0.1611	194.29	193.23		3.2/9.6				
M5-714FSF-5	"		121.2	0.3956	306.4	0.5	0.1025	37.58	0.1041	361.01			3.2/9.6				
	"		120.3	0.3956	304.1	0.5	0.1025	37.30	0.1041	358.32	359.66		3.2/9.6				
M5-714FSF-6	"		119.4	0.3956	301.8	1	0.1025	18.91	0.0880	214.91			3.2/9.6				
	"		118.9	0.3956	300.6	1	0.1025	18.83	0.0880	214.01	214.46		3.2/9.6				
M5-714FSF-7	"		111.2	0.3956	281.1	1	0.1025	17.61	0.0649	271.28			3.2/9.6				
	"		110	0.3956	278.1	1	0.1025	17.42	0.0649	268.34	269.81	238.28	3.2/9.6				
METHOD BLK-1	"		0.6	0.3956	1.52	1		0.10					3.2/9.6				
	"		0.8	0.3956	2.02	1		0.13			0.11		3.2/9.6				
METHOD BLK-0.5	"		0.3	0.3956	0.76	0.5		0.09					3.2/9.6				
	"		0.3	0.3956	0.76	0.5		0.09			0.09	0.102	3.2/9.6				
Instrument Blank	"		0.35	0.3956	0.88	1		0.06		0.0009	0.0009	0.0009	3.2/9.6				
DORM 29	"		33.4	0.3956	84.4	0.1	0.1025	50.81	0.0109	4661.3			3.2/9.6	4600	101.33		
DORM 29	"		33.2	0.3956	83.9	0.1	0.1025	50.50	0.0109	4633.3	4647.30	4647.30	3.2/9.6	4600	100.72	19.78	0.4
CCV-1	"		79.2	0.3956	200.2					0.2002			3.2/9.6	0.2	100.10		
CCV-2	"		79.5	0.3956	201.0					0.2010			3.2/9.6	0.2	100.48		
CCV-3	"		85	0.3956	214.9					0.2149			3.2/9.6	0.2	107.43		
CCV-4	"		85.2	0.3956	215.4					0.2154			3.2/9.6	0.2	107.68		
CCV-5	"		83.9	0.3956	212.1					0.2121			3.2/9.6	0.2	106.04		
CCV-6	"		85	0.3956	214.9					0.2149			3.2/9.6	0.2	107.43		
CCV-7	"		85.5	0.3956	216.1					0.2161			3.2/9.6	0.2	108.06		
CCV-8	"		82.6	0.3956	208.8					0.2088			3.2/9.6	0.2	104.40		
CCV-9	"		84.8	0.3956	214.4					0.2144			3.2/9.6	0.2	107.18		
CCV-10	"		83	0.3956	209.8					0.2098			3.2/9.6	0.2	104.90		
CCV-11	"		83.6	0.3956	211.3					0.2113			3.2/9.6	0.2	105.66		
CCV-12	"		86.3	0.3956	218.1					0.2181			3.2/9.6	0.2	109.07		
CCV-13	"		84.2	0.3956	212.8					0.2128			3.2/9.6	0.2	106.42		
CCV-14	"		85.3	0.3956	215.6					0.2156			3.2/9.6	0.2	107.81		
CCV-15	"		83.3	0.3956	210.6					0.2106			3.2/9.6	0.2	105.28		
CCV-16	"		83.9	0.3956	212.1					0.2121			3.2/9.6	0.2	106.04		
CCV-17	"		82.9	0.3956	209.6					0.2096			3.2/9.6	0.2	104.78		
CCV-18	"		83.6	0.3956	211.3					0.2113			3.2/9.6	0.2	105.66		
CCV-19	"		83	0.3956	209.8					0.2098			3.2/9.6	0.2	104.90		
CCV-20	"		83.3	0.3956	210.6					0.2106		0.211	3.2/9.6	0.2	105.28	0.0045	2.14

10 % Recalculated Results for Total Mercury in Fish Tissue

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-14-00 Checked by NJS 4-14-00

Sample	QC Batch	Qualifier Note	Instrument Reading Peak Height	Slope	Hg Concentration (ppt)	Vol. added (mL)	Average Reagent Blank (ng)	Corrected Hg Concentration (ng)	Weight of fish (g)	Hg Concentration ppb	Averaged Result ppb	Averaged Reported Result (ppb)	Detection Limit/*3 ppb	SPK CONC (ppb)	R%	Standard Deviation	Relative Standard Deviation
M5-714FSF-1	HG09KF1	r^2=0.9999	129.5	0.3845	336.8	0.5	0.0900	41.34	0.1451	284.88			3.2/9.6				
	"		133.1	0.3845	346.2	0.5	0.0900	42.49	0.1451	292.82	288.85		3.2/9.6				
M5-714FSF-2	"		88.5	0.3845	230.2	0.5	0.0900	28.22	0.1138	247.99			3.2/9.6				
	"		89	0.3845	231.5	0.5	0.0900	28.38	0.1138	249.39	248.69		3.2/9.6				
M5-714FSF-3	"		82.4	0.3845	214.3	0.5	0.0900	26.27	0.1586	165.63			3.2/9.6				
	"		82	0.3845	213.3	0.5	0.0900	26.14	0.1586	164.83	165.23		3.2/9.6				
M5-714FSF-4	"		111.7	0.3845	290.5	0.5	0.0900	35.64	0.1611	221.24			3.2/9.6				
	"		108.9	0.3845	283.2	0.5	0.0900	34.75	0.1611	215.68	218.46		3.2/9.6				
M5-714FSF-5	"		121	0.3845	314.7	0.5	0.0900	38.62	0.1041	370.96			3.2/9.6				
	"		124.6	0.3845	324.1	0.5	0.0900	39.77	0.1041	382.03	376.50		3.2/9.6				
M5-714FSF-6	"		129.5	0.3845	336.8	1	0.0900	21.13	0.0880	240.10			3.2/9.6				
	"		127.8	0.3845	332.4	1	0.0900	20.85	0.0880	236.93	238.51		3.2/9.6				
M5-714FSF-7	"		110.4	0.3845	287.1	1	0.0900	18.00	0.0649	277.33			3.2/9.6				
	"		108	0.3845	280.9	1	0.0900	17.61	0.0649	271.27	274.30	258.65	3.2/9.6				
METHOD BLK-1	"		0.5	0.3845	1.30	1		0.08					3.2/9.6				
	"		0.3	0.3845	0.78	1		0.05			0.07		3.2/9.6				
METHOD BLK-0.5	"		0.3	0.3845	0.78	0.5		0.10					3.2/9.6				
	"		0.4	0.3845	1.04	0.5		0.13			0.11	0.089	3.2/9.6				
Instrument Blank	"		0.3	0.3845	0.78	1		0.05		0.0008	0.0008	0.0008	3.2/9.6				
DORM 37	"		42.2	0.3845	109.8	0.1	0.0900	66.09	0.014	4720.8			3.2/9.6	4600	102.63		
DORM 37	"		42.6	0.3845	110.8	0.1	0.0900	66.72	0.014	4765.6	4743.19	4743.19	3.2/9.6	4600	103.60	31.68	0.7
CCV-1	"		75.7	0.3845	196.9					0.1969			3.2/9.6	0.2	98.44		
CCV-2	"		76.2	0.3845	198.2					0.1982			3.2/9.6	0.2	99.09		
CCV-3	"		82.5	0.3845	214.6					0.2146			3.2/9.6	0.2	107.28		
CCV-4	"		83.1	0.3845	216.1					0.2161			3.2/9.6	0.2	108.06		
CCV-5	"		83.6	0.3845	217.4					0.2174			3.2/9.6	0.2	108.71		
CCV-6	"		83.8	0.3845	217.9					0.2179			3.2/9.6	0.2	108.97		
CCV-7	"		82.8	0.3845	215.3					0.2153			3.2/9.6	0.2	107.67		
CCV-8	"		83.6	0.3845	217.4					0.2174			3.2/9.6	0.2	108.71		
CCV-9	"		80.7	0.3845	209.9					0.2099			3.2/9.6	0.2	104.94		
CCV-10	"		80.8	0.3845	210.1					0.2101			3.2/9.6	0.2	105.07		
CCV-11	"		73.6	0.3845	191.4					0.1914			3.2/9.6	0.2	95.71		
CCV-12	"		74.6	0.3845	194.0					0.1940			3.2/9.6	0.2	97.01		
CCV-13	"		72.3	0.3845	188.0					0.1880			3.2/9.6	0.2	94.02		
CCV-14	"		72.3	0.3845	188.0					0.1880			3.2/9.6	0.2	94.02		
CCV-15	"		72.9	0.3845	189.6					0.1896			3.2/9.6	0.2	94.80		
CCV-16	"		73.9	0.3845	192.2					0.1922			3.2/9.6	0.2	96.10		
CCV-21	"		75.8	0.3845	197.1					0.1971			3.2/9.6	0.2	98.57		
CCV-22	"		77.2	0.3845	200.8					0.2008		0.203	3.2/9.6	0.2	100.39	0.0115	5.67

10 % Recalculated Results for Total Mercury in Fish Tissue

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-14-00 Checked by NJS 4-14-00

Sample	QC Batch	Qualifier Note	Instrument Reading Peak Height	Slope	Hg Concentration (ppt)	Vol. added (mL)	Average Reagent Blank (ng)	Corrected Hg Concentration (ng)	Weight of fish (g)	Hg Concentration ppb	Averaged Result ppb	Averaged Reported Result (ppb)	Detection Limit/*3 ppb	SPK CONC (ppb)	R%	Standard Deviation	Relative Standard Deviation
M5-726FSF-1	HG09KF1	r^2=0.9999	56.8	0.3845	147.7	0.2	0.0900	44.67	0.2004	222.91			3.2/9.6				
	"		57.4	0.3845	149.3	0.2	0.0900	45.14	0.2004	225.27	224.09		3.2/9.6				
M5-726FSF-2	"		26.3	0.3845	68.4	0.2	0.0900	20.64	0.1542	133.82			3.2/9.6				
	"		26.4	0.3845	68.7	0.2	0.0900	20.71	0.1542	134.33	134.08		3.2/9.6				
M5-726FSF-3	"		44.1	0.3845	114.7	0.2	0.0900	34.66	0.1778	194.95			3.2/9.6				
	"		44.3	0.3845	115.2	0.2	0.0900	34.82	0.1778	195.84	195.39		3.2/9.6				
M5-726FSF-4	"		21.3	0.3845	55.4	1	0.0900	3.40	0.1063	31.98			3.2/9.6				
	"		21.1	0.3845	54.9	1	0.0900	3.37	0.1063	31.68	31.83		3.2/9.6				
M5-726FSF-5	"		38.5	0.3845	100.1	1	0.0900	6.22	0.0667	93.23			3.2/9.6				
	"		40.7	0.3845	105.9	1	0.0900	6.58	0.0667	98.63	95.93		3.2/9.6				
M5-726FSF-6	"		15	0.3845	39.0	1	0.0900	2.37	0.0720	32.89			3.2/9.6				
	"		14.8	0.3845	38.5	1	0.0900	2.33	0.0720	32.43	32.66		3.2/9.6				
M5-726FSF-7	"		22.4	0.3845	58.3	1	0.0900	3.58	0.1005	35.62			3.2/9.6				
	"		22.3	0.3845	58.0	1	0.0900	3.56	0.1005	35.46	35.54	107.07	3.2/9.6				
METHOD BLK-1	"		0.5	0.3845	1.30	1		0.08					3.2/9.6				
	"		0.3	0.3845	0.78	1		0.05			0.07		3.2/9.6				
METHOD BLK-0.5	"		0.3	0.3845	0.78	0.5		0.10					3.2/9.6				
	"		0.4	0.3845	1.04	0.5		0.13			0.11	0.089	3.2/9.6				
Instrument Blank	"		0.3	0.3845	0.78	1		0.05		0.0008	0.0008	0.0008	3.2/9.6				
DORM 37	"		42.2	0.3845	109.8	0.1	0.0900	66.09	0.014	4720.8			3.2/9.6	4600	102.63		
DORM 37	"		42.6	0.3845	110.8	0.1	0.0900	66.72	0.014	4765.6	4743.19	4743.19	3.2/9.6	4600	103.60	31.68	0.7
CCV-1	"		75.7	0.3845	196.9					0.1969			3.2/9.6	0.2	98.44		
CCV-2	"		76.2	0.3845	198.2					0.1982			3.2/9.6	0.2	99.09		
CCV-3	"		82.5	0.3845	214.6					0.2146			3.2/9.6	0.2	107.28		
CCV-4	"		83.1	0.3845	216.1					0.2161			3.2/9.6	0.2	108.06		
CCV-5	"		83.6	0.3845	217.4					0.2174			3.2/9.6	0.2	108.71		
CCV-6	"		83.8	0.3845	217.9					0.2179			3.2/9.6	0.2	108.97		
CCV-7	"		82.8	0.3845	215.3					0.2153			3.2/9.6	0.2	107.67		
CCV-8	"		83.6	0.3845	217.4					0.2174			3.2/9.6	0.2	108.71		
CCV-9	"		80.7	0.3845	209.9					0.2099			3.2/9.6	0.2	104.94		
CCV-10	"		80.8	0.3845	210.1					0.2101			3.2/9.6	0.2	105.07		
CCV-11	"		73.6	0.3845	191.4					0.1914			3.2/9.6	0.2	95.71		
CCV-12	"		74.6	0.3845	194.0					0.1940			3.2/9.6	0.2	97.01		
CCV-13	"		72.3	0.3845	188.0					0.1880			3.2/9.6	0.2	94.02		
CCV-14	"		72.3	0.3845	188.0					0.1880			3.2/9.6	0.2	94.02		
CCV-15	"		72.9	0.3845	189.6					0.1896			3.2/9.6	0.2	94.80		
CCV-16	"		73.9	0.3845	192.2					0.1922			3.2/9.6	0.2	96.10		
CCV-21	"		75.8	0.3845	197.1					0.1971			3.2/9.6	0.2	98.57		
CCV-22	"		77.2	0.3845	200.8					0.2008		0.203	3.2/9.6	0.2	100.39	0.0115	5.67

10 % Recalculated Results for Total Mercury in Fish Tissue

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-14-00 Checked by NJS 4-14-00

Sample	QC Batch	Qualifier Note	Instrument Reading Peak Height	Slope	Hg Concentration (ppt)	Vol. added (mL)	Average Reagent Blank (ng)	Corrected Hg Concentration (ng)	Weight of fish (g)	Hg Concentration ppb	Averaged Result ppb	Averaged Reported Result (ppb)	Detection Limit/*3 ppb	SPK CONC (ppb)	R%	Standard Deviation	Relative Standard Deviation
M5-738FSF-1	HG12KF1	r^2=0.9994	21.5	0.4012	53.6	0.5	0.1275	6.46	0.1478	43.73			3.2/9.6				
	"		21.1	0.4012	52.6	0.5	0.1275	6.34	0.1478	42.90	43.32		3.2/9.6				
M5-738FSF-2	"		17.9	0.4012	44.6	0.5	0.1275	5.36	0.1318	40.67			3.2/9.6				
	"		18.2	0.4012	45.4	0.5	0.1275	5.45	0.1318	41.37	41.02		3.2/9.6				
M5-738FSF-3	"		18.7	0.4012	46.6	0.2	0.1275	14.00	0.1996	70.12			3.2/9.6				
	"		18.7	0.4012	46.6	0.2	0.1275	14.00	0.1996	70.12	70.12		3.2/9.6				
M5-738FSF-4	"		18.9	0.4012	47.1	1	0.1275	2.84	0.0906	31.35			3.2/9.6				
	"		18.9	0.4012	47.1	1	0.1275	2.84	0.0906	31.35	31.35		3.2/9.6				
M5-738FSF-5	"		29.8	0.4012	74.3	1	0.1275	4.55	0.1315	34.62			3.2/9.6				
	"		29.8	0.4012	74.3	1	0.1275	4.55	0.1315	34.62	34.62		3.2/9.6				
M5-738FSF-6	"		28	0.4012	69.8	1	0.1275	4.27	0.0317	134.68			3.2/9.6				
	"		28.1	0.4012	70.0	1	0.1275	4.29	0.0317	135.17	134.93		3.2/9.6				
M5-738FSF-7	"		15.4	0.4012	38.4	1	0.1275	2.29	0.0707	32.40			3.2/9.6				
	"		14.9	0.4012	37.1	1	0.1275	2.21	0.0707	31.29	31.85	55.31	3.2/9.6				
METHOD BLK-1	"		1.2	0.4012	2.99	1		0.19					3.2/9.6				
	"		0.9	0.4012	2.24	1		0.14			0.16		3.2/9.6				
METHOD BLK-0.5	"		0.1	0.4012	0.25	0.5		0.03					3.2/9.6				
	"		0.5	0.4012	1.25	0.5		0.15			0.09	0.128	3.2/9.6				
Instrument Blank	"		0.5	0.4012	1.25	1		0.08		0.0012	0.0012	0.0012	3.2/9.6				
DORM 47	"		48.3	0.4012	120.4	0.1	0.1275	72.47	0.0152	4767.6			3.2/9.6	4600	103.64		
DORM 47	"		48.5	0.4012	120.9	0.1	0.1275	72.77	0.0152	4787.3	4777.45	4777.45	3.2/9.6	4600	104.07	13.98	0.3
CCV-1	"		80.2	0.4012	199.9					0.1999			3.2/9.6	0.2	99.95		
CCV-2	"		80.8	0.4012	201.4					0.2014			3.2/9.6	0.2	100.70		
CCV-3	"		75.5	0.4012	188.2					0.1882			3.2/9.6	0.2	94.09		
CCV-4	"		79.8	0.4012	198.9					0.1989			3.2/9.6	0.2	99.45		
CCV-5	"		79.8	0.4012	198.9					0.1989			3.2/9.6	0.2	99.45		
CCV-6	"		79.6	0.4012	198.4					0.1984			3.2/9.6	0.2	99.20		
CCV-7	"		79.5	0.4012	198.2					0.1982			3.2/9.6	0.2	99.08		
CCV-8	"		79.8	0.4012	198.9					0.1989			3.2/9.6	0.2	99.45		
CCV-9	"		76.8	0.4012	191.4					0.1914			3.2/9.6	0.2	95.71		
CCV-10	"		80	0.4012	199.4					0.1994			3.2/9.6	0.2	99.70		
CCV-11	"		79.2	0.4012	197.4					0.1974			3.2/9.6	0.2	98.70		
CCV-12	"		79.6	0.4012	198.4					0.1984			3.2/9.6	0.2	99.20		
CCV-13	"		74.9	0.4012	186.7					0.1867			3.2/9.6	0.2	93.34		
CCV-14	"		79.8	0.4012	198.9					0.1989			3.2/9.6	0.2	99.45		
CCV-15	"		78.1	0.4012	194.7					0.1947			3.2/9.6	0.2	97.33		
CCV-16	"		78	0.4012	194.4					0.1944			3.2/9.6	0.2	97.21		
CCV-17	"		76.9	0.4012	191.7					0.1917			3.2/9.6	0.2	95.84		
CCV-18	"		78.4	0.4012	195.4					0.1954		0.196	3.2/9.6	0.2	97.71	0.0042	2.14

10 % Recalculated Results for Total Mercury in Fish Tissue

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-14-00 Checked by NJS 4-14-00

Sample	QC Batch	Qualifier Note	Instrument Reading Peak Height	Slope	Hg Concentration (ppt)	Vol. added (mL)	Average Reagent Blank (ng)	Corrected Hg Concentration (ng)	Weight of fish (g)	Hg Concentration ppb	Averaged Result ppb	Averaged Reported Result (ppb)	Detection Limit/*3 ppb	SPK CONC (ppb)	R%	Standard Deviation	Relative Standard Deviation
M5-828FSF-1	HG16KF1	r^2=1.0000	83.9	0.4302	195.0	0.5	0.0850	23.90	0.1472	162.39			3.2/9.6				
	"		84.1	0.4302	195.5	0.5	0.0850	23.96	0.1472	162.77	162.58		3.2/9.6				
M5-828FSF-2	"		24.8	0.4302	57.6	0.1	0.0850	34.68	0.2229	155.57			3.2/9.6				
	"		24.6	0.4302	57.2	0.1	0.0850	34.40	0.2229	154.31	154.94		3.2/9.6				
M5-828FSF-3	"		54.1	0.4302	125.8	0.1	0.0850	75.75	0.2527	299.74			3.2/9.6				
	"		53	0.4302	123.2	0.1	0.0850	74.20	0.2527	293.64	296.69		3.2/9.6				
M5-828FSF-4	"		152.9	0.4302	355.4	1	0.0850	22.31	0.0928	240.37			3.2/9.6				
	"		152.9	0.4302	355.4	1	0.0850	22.31	0.0928	240.37	240.37		3.2/9.6				
M5-828FSF-5	"		67.8	0.4302	157.6	1	0.0850	9.84	0.0316	311.51			3.2/9.6				
	"		67.7	0.4302	157.4	1	0.0850	9.83	0.0316	311.05	311.28		3.2/9.6				
M5-828FSF-6	"		107.9	0.4302	250.8	0.5	0.0850	30.77	0.1142	269.40			3.2/9.6				
	"		108.6	0.4302	252.4	0.5	0.0850	30.97	0.1142	271.15	270.27	239.36	3.2/9.6				
METHOD BLK-1	"		1	0.4302	2.32	1		0.15					3.2/9.6				
	"		0.5	0.4302	1.16	1		0.07			0.11		3.2/9.6				
METHOD BLK-0.5	"		0.2	0.4302	0.46	0.5		0.06					3.2/9.6				
	"		0.2	0.4302	0.46	0.5		0.06			0.06	0.084	3.2/9.6				
Instrument Blank	"		0.55	0.4302	1.28	1		0.08		0.0013	0.0013	0.0013	3.2/9.6				
DORM 49	"		49.5	0.4302	115.1	0.1	0.0850	69.30	0.016	4331.1			3.2/9.6	4600	94.15		
DORM 49	"		49.8	0.4302	115.8	0.1	0.0850	69.72	0.016	4357.4	4344.26	4344.26	3.2/9.6	4600	94.73	18.58	0.4
CCV-1	"		85.7	0.4302	199.2					0.1992			3.2/9.6	0.2	99.60		
CCV-2	"		86.6	0.4302	201.3					0.2013			3.2/9.6	0.2	100.65		
CCV-3	"		85.8	0.4302	199.4					0.1994			3.2/9.6	0.2	99.72		
CCV-4	"		86.1	0.4302	200.1					0.2001			3.2/9.6	0.2	100.07		
CCV-5	"		84.8	0.4302	197.1					0.1971			3.2/9.6	0.2	98.56		
CCV-6	"		84.6	0.4302	196.7					0.1967			3.2/9.6	0.2	98.33		
CCV-7	"		82.3	0.4302	191.3					0.1913			3.2/9.6	0.2	95.65		
CCV-8	"		85.4	0.4302	198.5					0.1985			3.2/9.6	0.2	99.26		
CCV-9	"		87.7	0.4302	203.9					0.2039			3.2/9.6	0.2	101.93		
CCV-10	"		88.1	0.4302	204.8					0.2048			3.2/9.6	0.2	102.39		
CCV-11	"		86.1	0.4302	200.1					0.2001			3.2/9.6	0.2	100.07		
CCV-12	"		90	0.4302	209.2					0.2092			3.2/9.6	0.2	104.60		
CCV-13	"		90	0.4302	209.2					0.2092			3.2/9.6	0.2	104.60		
CCV-14	"		90.7	0.4302	210.8					0.2108			3.2/9.6	0.2	105.42		
CCV-15	"		86.1	0.4302	200.1					0.2001			3.2/9.6	0.2	100.07		
CCV-16	"		89.8	0.4302	208.7					0.2087			3.2/9.6	0.2	104.37		
CCV-17	"		90.4	0.4302	210.1					0.2101			3.2/9.6	0.2	105.07		
CCV-18	"		90.9	0.4302	211.3					0.2113			3.2/9.6	0.2	105.65		
CCV-19	"		86.8	0.4302	201.8					0.2018			3.2/9.6	0.2	100.88		
CCV-20	"		90.8	0.4302	211.1					0.2111			3.2/9.6	0.2	105.53		
CCV-21	"		89.6	0.4302	208.3					0.2083			3.2/9.6	0.2	104.14		
CCV-22	"		89.7	0.4302	208.5					0.2085			3.2/9.6	0.2	104.25		
CCV-23	"		89.6	0.4302	208.3					0.2083			3.2/9.6	0.2	104.14		
CCV-24	"		89.5	0.4302	208.0					0.2080		0.204	3.2/9.6	0.2	104.02	0.0056	2.76

10 % Recalculated Results for Total Mercury in Fish Tissue

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-14-00 Checked by NJS 4-14-00

Sample	QC Batch	Qualifier Note	Instrument Reading Peak Height	Slope	Hg Concentration (ppt)	Vol. added (mL)	Average Reagent Blank (ng)	Corrected Hg Concentration (ng)	Weight of fish (g)	Hg Concentration ppb	Averaged Result ppb	Averaged Reported Result (ppb)	Detection Limit/*3 ppb	SPK CONC (ppb)	R%	Standard Deviation	Relative Standard Deviation
M5-944FSF-1	HG15KF1	r^2=0.9999	98	0.4179	234.5	1	0.2625	14.51	0.0872	166.41			3.2/9.6				
	"		96.1	0.4179	230.0	1	0.2625	14.22	0.0872	163.13	164.77		3.2/9.6				
M5-944FSF-2	"		38.6	0.4179	92.4	1	0.2625	5.56	0.0773	71.88			3.2/9.6				
	"		39.8	0.4179	95.2	1	0.2625	5.74	0.0773	74.22	73.05		3.2/9.6				
M5-944FSF-3	"		77.6	0.4179	185.7	1	0.2625	11.44	0.0754	151.67			3.2/9.6				
	"		77.6	0.4179	185.7	1	0.2625	11.44	0.0754	151.67	151.67		3.2/9.6				
M5-944FSF-4	"		66.3	0.4179	158.7	1	0.2625	9.73	0.0842	115.59			3.2/9.6				
	"		70.1	0.4179	167.7	1	0.2625	10.31	0.0842	122.39	118.99		3.2/9.6				
M5-944FSF-5	"		36.7	0.4179	87.8	1	0.2625	5.27	0.0389	135.48			3.2/9.6				
	"		36.8	0.4179	88.1	1	0.2625	5.29	0.0389	135.87	135.67		3.2/9.6				
M5-944FSF-6	"		30.5	0.4179	73.0	1	0.2625	4.34	0.0434	99.90			3.2/9.6				
	"		32.6	0.4179	78.0	1	0.2625	4.65	0.0434	107.19	103.54		3.2/9.6				
M5-944FSF7	"		23.3	0.4179	55.8	1	0.2625	3.25	0.0361	90.03			3.2/9.6				
	"		23.5	0.4179	56.2	1	0.2625	3.28	0.0361	90.86	90.45	119.74	3.2/9.6				
METHOD BLK-1	"		2.8	0.4179	6.70	1		0.42					3.2/9.6				
	"		2.6	0.4179	6.22	1		0.39			0.41		3.2/9.6				
METHOD BLK-0.5	"		0.4	0.4179	0.96	0.5		0.12					3.2/9.6				
	"		0.4	0.4179	0.96	0.5		0.12			0.12	0.262	3.2/9.6				
Instrument Blank	"		0.6	0.4179	1.44	1		0.09		0.0014	0.0014	0.0014	3.2/9.6				
DORM 52	"		41.3	0.4179	98.8	0.1	0.2625	59.33	0.0136	4362.5			3.2/9.6	4600	94.84		
DORM 52	"		40.7	0.4179	97.4	0.1	0.2625	58.46	0.0136	4298.9	4330.70	4330.70	3.2/9.6	4600	93.45	45.01	1.0
CCV-1	"		83.4	0.4179	199.6					0.1996			3.2/9.6	0.2	99.78		
CCV-2	"		84.2	0.4179	201.5					0.2015			3.2/9.6	0.2	100.74		
CCV-3	"		79.2	0.4179	189.5					0.1895			3.2/9.6	0.2	94.76		
CCV-4	"		82.9	0.4179	198.4					0.1984			3.2/9.6	0.2	99.19		
CCV-5	"		83.2	0.4179	199.1					0.1991			3.2/9.6	0.2	99.55		
CCV-6	"		82.5	0.4179	197.4					0.1974			3.2/9.6	0.2	98.71		
CCV-7	"		82	0.4179	196.2					0.1962			3.2/9.6	0.2	98.11		
CCV-8	"		82.3	0.4179	196.9					0.1969			3.2/9.6	0.2	98.47		
CCV-9	"		82.3	0.4179	196.9					0.1969			3.2/9.6	0.2	98.47		
CCV-10	"		82.7	0.4179	197.9					0.1979			3.2/9.6	0.2	98.95		
CCV-11	"		82	0.4179	196.2					0.1962			3.2/9.6	0.2	98.11		
CCV-12	"		82.8	0.4179	198.1					0.1981			3.2/9.6	0.2	99.07		
CCV-13	"		81.1	0.4179	194.1					0.1941			3.2/9.6	0.2	97.03		
CCV-14	"		81.8	0.4179	195.7					0.1957			3.2/9.6	0.2	97.87		
CCV-15	"		78.9	0.4179	188.8					0.1888			3.2/9.6	0.2	94.40		
CCV-16	"		80	0.4179	191.4					0.1914			3.2/9.6	0.2	95.72		
CCV-17	"		80.1	0.4179	191.7					0.1917			3.2/9.6	0.2	95.84		
CCV-18	"		79.7	0.4179	190.7					0.1907		0.196	3.2/9.6	0.2	95.36	0.0037	1.89

10 % Recalculated Results for Total Mercury in Fish Tissue

Analyzed by Florida International University (SERC) for the September 1999 Wet Season (M5)

Entered by mwb 4-14-00 Checked by NJS 4-14-00

Sample	QC Batch	Qualifier Note	Instrument Reading Peak Height	Slope	Hg Concentration (ppt)	Vol. added (mL)	Average Reagent Blank (ng)	Corrected Hg Concentration (ng)	Weight of fish (g)	Hg Concentration ppb	Averaged Result ppb	Averaged Reported Result (ppb)	Detection Limit/*3 ppb	SPK CONC (ppb)	R%	Standard Deviation	Relative Standard Deviation
M5-944FSF-1	HG15KF1	r^2=0.9999	98	0.4179	234.5	1	0.2625	14.51	0.0872	166.41			3.2/9.6				
	"		96.1	0.4179	230.0	1	0.2625	14.22	0.0872	163.13	164.77		3.2/9.6				
M5-944FSF-2	"		38.6	0.4179	92.4	1	0.2625	5.56	0.0773	71.88			3.2/9.6				
	"		39.8	0.4179	95.2	1	0.2625	5.74	0.0773	74.22	73.05		3.2/9.6				
M5-944FSF-3	"		77.6	0.4179	185.7	1	0.2625	11.44	0.0754	151.67			3.2/9.6				
	"		77.6	0.4179	185.7	1	0.2625	11.44	0.0754	151.67	151.67		3.2/9.6				
M5-944FSF-4	"		66.3	0.4179	158.7	1	0.2625	9.73	0.0842	115.59			3.2/9.6				
	"		70.1	0.4179	167.7	1	0.2625	10.31	0.0842	122.39	118.99		3.2/9.6				
M5-944FSF-5	"		36.7	0.4179	87.8	1	0.2625	5.27	0.0389	135.48			3.2/9.6				
	"		36.8	0.4179	88.1	1	0.2625	5.29	0.0389	135.87	135.67		3.2/9.6				
M5-944FSF-6	"		30.5	0.4179	73.0	1	0.2625	4.34	0.0434	99.90			3.2/9.6				
	"		32.6	0.4179	78.0	1	0.2625	4.65	0.0434	107.19	103.54		3.2/9.6				
M5-944FSF7	"		23.3	0.4179	55.8	1	0.2625	3.25	0.0361	90.03			3.2/9.6				
	"		23.5	0.4179	56.2	1	0.2625	3.28	0.0361	90.86	90.45	119.74	3.2/9.6				
METHOD BLK-1	"		2.8	0.4179	6.70	1		0.42					3.2/9.6				
	"		2.6	0.4179	6.22	1		0.39			0.41		3.2/9.6				
METHOD BLK-0.5	"		0.4	0.4179	0.96	0.5		0.12					3.2/9.6				
	"		0.4	0.4179	0.96	0.5		0.12			0.12	0.262	3.2/9.6				
Instrument Blank	"		0.6	0.4179	1.44	1		0.09		0.0014	0.0014	0.0014	3.2/9.6				
DORM 52	"		41.3	0.4179	98.8	0.1	0.2625	59.33	0.0136	4362.5			3.2/9.6	4600	94.84		
DORM 52	"		40.7	0.4179	97.4	0.1	0.2625	58.46	0.0136	4298.9	4330.70	4330.70	3.2/9.6	4600	93.45	45.01	1.0
CCV-1	"		83.4	0.4179	199.6					0.1996			3.2/9.6	0.2	99.78		
CCV-2	"		84.2	0.4179	201.5					0.2015			3.2/9.6	0.2	100.74		
CCV-3	"		79.2	0.4179	189.5					0.1895			3.2/9.6	0.2	94.76		
CCV-4	"		82.9	0.4179	198.4					0.1984			3.2/9.6	0.2	99.19		
CCV-5	"		83.2	0.4179	199.1					0.1991			3.2/9.6	0.2	99.55		
CCV-6	"		82.5	0.4179	197.4					0.1974			3.2/9.6	0.2	98.71		
CCV-7	"		82	0.4179	196.2					0.1962			3.2/9.6	0.2	98.11		
CCV-8	"		82.3	0.4179	196.9					0.1969			3.2/9.6	0.2	98.47		
CCV-9	"		82.3	0.4179	196.9					0.1969			3.2/9.6	0.2	98.47		
CCV-10	"		82.7	0.4179	197.9					0.1979			3.2/9.6	0.2	98.95		
CCV-11	"		82	0.4179	196.2					0.1962			3.2/9.6	0.2	98.11		
CCV-12	"		82.8	0.4179	198.1					0.1981			3.2/9.6	0.2	99.07		
CCV-13	"		81.1	0.4179	194.1					0.1941			3.2/9.6	0.2	97.03		
CCV-14	"		81.8	0.4179	195.7					0.1957			3.2/9.6	0.2	97.87		
CCV-15	"		78.9	0.4179	188.8					0.1888			3.2/9.6	0.2	94.40		
CCV-16	"		80	0.4179	191.4					0.1914			3.2/9.6	0.2	95.72		
CCV-17	"		80.1	0.4179	191.7					0.1917			3.2/9.6	0.2	95.84		
CCV-18	"		79.7	0.4179	190.7					0.1907		0.196	3.2/9.6	0.2	95.36	0.0037	1.89

September (M5) 1999 Samples for Critical Parameters Analyzed by SERC With the 10% Full QA/QC Review

Station ID **M5-622-FSF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	X	X	X
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	09/30/99	09/30/99	09/30/99
Digestion Date	10/25/99	10/13/99	10/13/99
Analysis Date	10/25/99	10/13/99	10/13/99
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume	7 Fish	NA	NA
Total Volume	7 Fish	7 Fish	7 Fish
Sample Volume Analyzed	Aliquot	7 Fish	7 Fish
Dilution	500-1000	NA	NA
Results	170	See Database	See Database
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	FDL/JL	MB	MB
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	No	No
All Calculation Checked	Yes (FTN Associates)	NA	NA
Holding Time Met	Yes	Yes	Yes
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID	HG21JF1	By: Date/Time	By: Date/Time
Method Blanks	All Good	NA	NA
Instrument Blanks	Good	NA	NA
Duplicates (RSD)	<20 RSD	NA	NA
Matrix Spike Recoveries (75-125)	All Good	NA	NA
Blank Spike/CCV Recoveries	All Good	NA	NA
Detection Range	3.2 ppb and >	NA	NA
Correlation Coefficient (>0.995)	0.9989	NA	NA
QC Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	NA	NA
All Calculation Checked	Yes (FTN Associates)	NA	NA
QC Limits Met			

Notes

No descriptive narratives were provided by FIU.

Holding time goals set for the length and weight measurements are guidelines only.

Documentation on how the fish samples were stored (Frozen, 4 C, preserved) was not provided.

Station ID **M5-622-FSF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	X	X	X
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September (M5) 1999 Samples for Critical Parameters Analyzed by SERC With the 10% Full QA/QC Review

Station ID **M5-633-FSF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	X	X	X
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	09/29/99	09/29/99	09/29/99
Digestion Date	10/26/99	10/14/99	10/14/99
Analysis Date	10/26/99	10/14/99	10/14/99
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume	7 Fish	NA	NA
Total Volume	7 Fish	7 Fish	7 Fish
Sample Volume Analyzed	Aliquot	7 Fish	7 Fish
Dilution	500-1000	NA	NA
Results	41	See Database	See Database
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	FDL/JL	MB	MB
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	No	No
All Calculation Checked	Yes (FTN Associates)	NA	NA
Holding Time Met	Yes	No (Goal Only)	No (Goal Only)
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID	HG22JF1	By: Date/Time	By: Date/Time
Method Blanks	All Good	NA	NA
Instrument Blanks	Good	NA	NA
Duplicates (RSD)	<20 RSD	NA	NA
Matrix Spike Recoveries (75-125)	65.48%	NA	NA
Blank Spike/CCV Recoveries	All Good	NA	NA
Detection Range	3.2 ppb and >	NA	NA
Correlation Coefficient (>0.995)	1	NA	NA
QC Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	NA	NA
All Calculation Checked	Yes (FTN Associates)	NA	NA
QC Limits Met	"M"		

Notes

No descriptive narratives were provided by FIU.

Holding time goals set for the length and weight measurements are guidelines only.

Documentation on how the fish samples were stored (Frozen, 4 C, preserved) was not provided.

Station ID **M5-633-FSF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	X	X	X
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers	"M"		

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September (M5) 1999 Samples for Critical Parameters Analyzed by SERC With the 10% Full QA/QC Review

Station ID **M5-643-FSF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	X	X	X
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	09/29/99	09/29/99	09/29/99
Digestion Date	10/27/99	10/19/99	10/19/99
Analysis Date	10/27/99	10/19/99	10/19/99
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume	7 Fish	NA	NA
Total Volume	7 Fish	7 Fish	7 Fish
Sample Volume Analyzed	Aliquot	7 Fish	7 Fish
Dilution	500-1000	NA	NA
Results	45	See Database	See Database
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	FDL/JL	MB	MB
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	No	No
All Calculation Checked	Yes (FTN Associates)	NA	NA
Holding Time Met	Yes	No (Goal Only)	No (Goal Only)
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID	HG26JF1	By: Date/Time	By: Date/Time
Method Blanks	All Good	NA	NA
Instrument Blanks	Good	NA	NA
Duplicates (RSD)	<20 RSD	NA	NA
Matrix Spike Recoveries (75-125)	All Good	NA	NA
Blank Spike/CCV Recoveries	All Good	NA	NA
Detection Range	3.2 ppb and >	NA	NA
Correlation Coefficient (>0.995)	0.9998	NA	NA
QC Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	NA	NA
All Calculation Checked	Yes (FTN Associates)	NA	NA
QC Limits Met			

Notes

No descriptive narratives were provided by FIU.

Holding time goals set for the length and weight measurements are guidelines only.

Documentation on how the fish samples were stored (Frozen, 4 C, preserved) was not provided.

Station ID **M5-643-FSF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	X	X	X
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September (M5) 1999 Samples for Critical Parameters Analyzed by SERC With the 10% Full QA/QC Review

Station ID **M5-653-FSF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	X	X	X
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	09/28/99	09/28/99	09/28/99
Digestion Date	10/27/99	10/21/99	10/21/99
Analysis Date	10/27/99	10/21/99	10/21/99
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume	7 Fish	NA	NA
Total Volume	7 Fish	7 Fish	7 Fish
Sample Volume Analyzed	Aliquot	7 Fish	7 Fish
Dilution	500-1000	NA	NA
Results	8.2	See Database	See Database
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	FDL/JL	MB	MB
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	No	No
All Calculation Checked	Yes (FTN Associates)	NA	NA
Holding Time Met	Yes	No (Goal Only)	No (Goal Only)
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID	HG28JF1	By: Date/Time	By: Date/Time
Method Blanks	All Good	NA	NA
Instrument Blanks	Good	NA	NA
Duplicates (RSD)	<20 RSD	NA	NA
Matrix Spike Recoveries (75-125)	All Good	NA	NA
Blank Spike/CCV Recoveries	All Good	NA	NA
Detection Range	3.2 ppb and >	NA	NA
Correlation Coefficient (>0.995)	0.9982	NA	NA
QC Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	NA	NA
All Calculation Checked	Yes (FTN Associates)	NA	NA
QC Limits Met			

Notes

No descriptive narratives were provided by FIU.

Holding time goals set for the length and weight measurements are guidelines only.

Documentation on how the fish samples were stored (Frozen, 4 C, preserved) was not provided.

Station ID **M5-653-FSF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	X	X	X
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September (M5) 1999 Samples for Critical Parameters Analyzed by SERC With the 10% Full QA/QC Review

Station ID **M5-663-FSF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	X	X	X
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	09/27/99	09/27/99	09/27/99
Digestion Date	11/02/99	11/22/99	11/22/99
Analysis Date	11/02/99	11/22/99	11/22/99
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume	7 Fish	NA	NA
Total Volume	7 Fish	7 Fish	7 Fish
Sample Volume Analyzed	Aliquot	7 Fish	7 Fish
Dilution	500-1000	NA	NA
Results	130	See Database	See Database
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	FDL/JL	MB	MB
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	No	No
All Calculation Checked	Yes (FTN Associates)	NA	NA
Holding Time Met	No (Goal Only)	No (Goal Only)	No (Goal Only)
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID	HG21JF1	By: Date/Time	By: Date/Time
Method Blanks	All Good	NA	NA
Instrument Blanks	Good	NA	NA
Duplicates (RSD)	<20 RSD	NA	NA
Matrix Spike Recoveries (75-125)	All Good	NA	NA
Blank Spike/CCV Recoveries	All Good	NA	NA
Detection Range	3.2 ppb and >	NA	NA
Correlation Coefficient (>0.995)	0.999	NA	NA
QC Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	NA	NA
All Calculation Checked	Yes (FTN Associates)	NA	NA
QC Limits Met	"H"		

Notes

No descriptive narratives were provided by FIU.

Holding time goals set for the length and weight measurements are guidelines only.

Documentation on how the fish samples were stored (Frozen, 4 C, preserved) was not provided.

Station ID **M5-663-FSF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	X	X	X
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers	"H"		

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September (M5) 1999 Samples for Critical Parameters Analyzed by SERC With the 10% Full QA/QC Review

Station ID **M5-673-FSF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	X	X	X
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	09/27/99	09/27/99	09/27/99
Digestion Date	11/08/99	10/25/99	10/25/99
Analysis Date	11/08/99	10/25/99	10/25/99
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume	7 Fish	NA	NA
Total Volume	7 Fish	7 Fish	7 Fish
Sample Volume Analyzed	Aliquot	7 Fish	7 Fish
Dilution	500-1000	NA	NA
Results	170	See Database	See Database
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	FDL/JL	MB	MB
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	No	No
All Calculation Checked	Yes (FTN Associates)	NA	NA
Holding Time Met	No (Goal Only)	No (Goal Only)	No (Goal Only)
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID	HG29JF1	By: Date/Time	By: Date/Time
Method Blanks	All Good	NA	NA
Instrument Blanks	Good	NA	NA
Duplicates (RSD)	<20 RSD	NA	NA
Matrix Spike Recoveries (75-125)	All Good	NA	NA
Blank Spike/CCV Recoveries	All Good	NA	NA
Detection Range	3.2 ppb and >	NA	NA
Correlation Coefficient (>0.995)	0.9993	NA	NA
QC Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	NA	NA
All Calculation Checked	Yes (FTN Associates)	NA	NA
QC Limits Met			

Notes

No descriptive narratives were provided by FIU.

Holding time goals set for the length and weight measurements are guidelines only.

Documentation on how the fish samples were stored (Frozen, 4 C, preserved) was not provided.

Station ID **M5-673-FSF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	X	X	X
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers			

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September (M5) 1999 Samples for Critical Parameters Analyzed by SERC With the 10% Full QA/QC Review

Station ID **M5-683-FSF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	X	X	X
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	09/26/99	09/26/99	09/26/99
Digestion Date	11/04/99	10/27/99	10/27/99
Analysis Date	11/04/99	10/27/99	10/27/99
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume	7 Fish	NA	NA
Total Volume	7 Fish	7 Fish	7 Fish
Sample Volume Analyzed	Aliquot	7 Fish	7 Fish
Dilution	500-1000	NA	NA
Results	220	See Database	See Database
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	FDL/JL	MB	MB
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	No	No
All Calculation Checked	Yes (FTN Associates)	NA	NA
Holding Time Met	No (Goal Only)	No (Goal Only)	No (Goal Only)
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID	HG01JF1	By: Date/Time	By: Date/Time
Method Blanks	All Good	NA	NA
Instrument Blanks	Good	NA	NA
Duplicates (RSD)	<20 RSD	NA	NA
Matrix Spike Recoveries (75-125)	All Good	NA	NA
Blank Spike/CCV Recoveries	All Good	NA	NA
Detection Range	3.2 ppb and >	NA	NA
Correlation Coefficient (>0.995)	0.9998	NA	NA
QC Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	NA	NA
All Calculation Checked	Yes (FTN Associates)	NA	NA
QC Limits Met	"H"		

Notes

No descriptive narratives were provided by FIU.

Holding time goals set for the length and weight measurements are guidelines only.

Documentation on how the fish samples were stored (Frozen, 4 C, preserved) was not provided.

Station ID **M5-683-FSF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	X	X	X
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers	"H"		

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September (M5) 1999 Samples for Critical Parameters Analyzed by SERC With the 10% Full QA/QC Review

Station ID **M5-693-FSF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	X	X	X
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	09/25/99	09/25/99	09/25/99
Digestion Date	10/27/99	10/27/99	10/27/99
Analysis Date	10/27/99	10/27/99	10/27/99
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume	7 Fish	NA	NA
Total Volume	7 Fish	7 Fish	7 Fish
Sample Volume Analyzed	Aliquot	7 Fish	7 Fish
Dilution	500-1000	NA	NA
Results	150	See Database	See Database
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	FDL/JL	MB	MB
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	No	No
All Calculation Checked	Yes (FTN Associates)	NA	NA
Holding Time Met	No (Goal Only)	No (Goal Only)	No (Goal Only)
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID	HG02KF1	By: Date/Time	By: Date/Time
Method Blanks	All Good	NA	NA
Instrument Blanks	Good	NA	NA
Duplicates (RSD)	<20 RSD	NA	NA
Matrix Spike Recoveries (75-125)	All Good	NA	NA
Blank Spike/CCV Recoveries	All Good	NA	NA
Detection Range	3.2 ppb and >	NA	NA
Correlation Coefficient (>0.995)	0.9996	NA	NA
QC Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	NA	NA
All Calculation Checked	Yes (FTN Associates)	NA	NA
QC Limits Met	"H"		

Notes

No descriptive narratives were provided by FIU.

Holding time goals set for the length and weight measurements are guidelines only.

Documentation on how the fish samples were stored (Frozen, 4 C, preserved) was not provided.

Station ID **M5-693-FSF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	X	X	X
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers	"H"		

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September (M5) 1999 Samples for Critical Parameters Analyzed by SERC With the 10% Full QA/QC Review

Station ID **M5-703-FSF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	X	X	X
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	09/24/99	09/24/99	09/24/99
Digestion Date	11/05/99	10/27/99	10/27/99
Analysis Date	11/05/99	10/27/99	10/27/99
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume	7 Fish	NA	NA
Total Volume	7 Fish	7 Fish	7 Fish
Sample Volume Analyzed	Aliquot	7 Fish	7 Fish
Dilution	500-1000	NA	NA
Results	250	See Database	See Database
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	FDL/JL	MB	MB
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	No	No
All Calculation Checked	Yes (FTN Associates)	NA	NA
Holding Time Met	No (Goal Only)	No (Goal Only)	No (Goal Only)
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID	HG05KF1	By: Date/Time	By: Date/Time
Method Blanks	All Good	NA	NA
Instrument Blanks	Good	NA	NA
Duplicates (RSD)	<20 RSD	NA	NA
Matrix Spike Recoveries (75-125)	All Good	NA	NA
Blank Spike/CCV Recoveries	All Good	NA	NA
Detection Range	3.2 ppb and >	NA	NA
Correlation Coefficient (>0.995)	0.9991	NA	NA
QC Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	NA	NA
All Calculation Checked	Yes (FTN Associates)	NA	NA
QC Limits Met	"H"		

Notes

No descriptive narratives were provided by FIU.

Holding time goals set for the length and weight measurements are guidelines only.

Documentation on how the fish samples were stored (Frozen, 4 C, preserved) was not provided.

Station ID **M5-703-FSF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	X	X	X
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers	"H"		

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September (M5) 1999 Samples for Critical Parameters Analyzed by SERC With the 10% Full QA/QC Review

Station ID **M5-714-FSF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	X	X	X
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	09/23/99	09/23/99	09/23/99
Digestion Date	10/25/99	11/01/99	11/01/99
Analysis Date	10/25/99	11/01/99	11/01/99
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume	7 Fish	NA	NA
Total Volume	7 Fish	7 Fish	7 Fish
Sample Volume Analyzed	Aliquot	7 Fish	7 Fish
Dilution	500-1000	NA	NA
Results	240	See Database	See Database
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	FDL/JL	MB	MB
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	No	No
All Calculation Checked	Yes (FTN Associates)	NA	NA
Holding Time Met	No (Goal Only)	No (Goal Only)	No (Goal Only)
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID	HG05KF1	By: Date/Time	By: Date/Time
Method Blanks	All Good	NA	NA
Instrument Blanks	Good	NA	NA
Duplicates (RSD)	<20 RSD	NA	NA
Matrix Spike Recoveries (75-125)	All Good	NA	NA
Blank Spike/CCV Recoveries	All Good	NA	NA
Detection Range	3.2 ppb and >	NA	NA
Correlation Coefficient (>0.995)	0.9991	NA	NA
QC Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	NA	NA
All Calculation Checked	Yes (FTN Associates)	NA	NA
QC Limits Met	"H"		

Notes

No descriptive narratives were provided by FIU.

Holding time goals set for the length and weight measurements are guidelines only.

Documentation on how the fish samples were stored (Frozen, 4 C, preserved) was not provided.

Station ID **M5-714-FSF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	X	X	X
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers	"H"		

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September (M5) 1999 Samples for Critical Parameters Analyzed by SERC With the 10% Full QA/QC Review

Station ID **M5-726-FSF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	X	X	X
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	09/23/99	09/23/99	09/23/99
Digestion Date	11/09/99	11/02/99	11/02/99
Analysis Date	11/09/99	11/02/99	11/02/99
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume	7 Fish	NA	NA
Total Volume	7 Fish	7 Fish	7 Fish
Sample Volume Analyzed	Aliquot	7 Fish	7 Fish
Dilution	110	NA	NA
Results	174.1	See Database	See Database
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	FDL/JL	MB	MB
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	No	No
All Calculation Checked	Yes (FTN Associates)	NA	NA
Holding Time Met	No (Goal Only)	No (Goal Only)	No (Goal Only)
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID	HG09KF1	By: Date/Time	By: Date/Time
Method Blanks	All Good	NA	NA
Instrument Blanks	Good	NA	NA
Duplicates (RSD)	<20 RSD	NA	NA
Matrix Spike Recoveries (75-125)	All Good	NA	NA
Blank Spike/CCV Recoveries	All Good	NA	NA
Detection Range	3.2 ppb and >	NA	NA
Correlation Coefficient (>0.995)	0.9999	NA	NA
QC Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	NA	NA
All Calculation Checked	Yes (FTN Associates)	NA	NA
QC Limits Met	"H"		

Notes

No descriptive narratives were provided by FIU.

Holding time goals set for the length and weight measurements are guidelines only.

Documentation on how the fish samples were stored (Frozen, 4 C, preserved) was not provided.

Station ID **M5-726-FSF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	X	X	X
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers	"H"		

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September (M5) 1999 Samples for Critical Parameters Analyzed by SERC With the 10% Full QA/QC Review

Station ID **M5-738-FSF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	X	X	X
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	09/22/99	09/22/99	09/22/99
Digestion Date	11/12/99	11/04/99	11/04/99
Analysis Date	11/12/99	11/04/99	11/04/99
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume	7 Fish	NA	NA
Total Volume	7 Fish	7 Fish	7 Fish
Sample Volume Analyzed	Aliquot	7 Fish	7 Fish
Dilution	500-1000	NA	NA
Results	57	See Database	See Database
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	FDL/JL	MB	MB
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	No	No
All Calculation Checked	Yes (FTN Associates)	NA	NA
Holding Time Met	No (Goal Only)	No (Goal Only)	No (Goal Only)
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID	HG12KF1	By: Date/Time	By: Date/Time
Method Blanks	All Good	NA	NA
Instrument Blanks	Good	NA	NA
Duplicates (RSD)	<20 RSD	NA	NA
Matrix Spike Recoveries (75-125)	All Good	NA	NA
Blank Spike/CCV Recoveries	All Good	NA	NA
Detection Range	3.2 ppb and >	NA	NA
Correlation Coefficient (>0.995)	0.9994	NA	NA
QC Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	NA	NA
All Calculation Checked	Yes (FTN Associates)	NA	NA
QC Limits Met	"H"		

Notes

No descriptive narratives were provided by FIU.

Holding time goals set for the length and weight measurements are guidelines only.

Documentation on how the fish samples were stored (Frozen, 4 C, preserved) was not provided.

Station ID **M5-738-FSF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	X	X	X
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers	"H"		

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September (M5) 1999 Samples for Critical Parameters Analyzed by SERC With the 10% Full QA/QC Review

Station ID **M5-828-FSF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	X	X	X
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	09/29/99	09/29/99	09/29/99
Digestion Date	11/16/99	11/08/99	11/08/99
Analysis Date	11/16/99	11/08/99	11/08/99
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume	7 Fish	NA	NA
Total Volume	7 Fish	7 Fish	7 Fish
Sample Volume Analyzed	Aliquot	7 Fish	7 Fish
Dilution	500-1000	NA	NA
Results	240	See Database	See Database
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	FDL/JL	MB	MB
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	No	No
All Calculation Checked	Yes (FTN Associates)	NA	NA
Holding Time Met	No (Goal Only)	No (Goal Only)	No (Goal Only)
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID	HG16KF1	By: Date/Time	By: Date/Time
Method Blanks	All Good	NA	NA
Instrument Blanks	Good	NA	NA
Duplicates (RSD)	<20 RSD	NA	NA
Matrix Spike Recoveries (75-125)	All Good	NA	NA
Blank Spike/CCV Recoveries	All Good	NA	NA
Detection Range	3.2 ppb and >	NA	NA
Correlation Coefficient (>0.995)	1	NA	NA
QC Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	NA	NA
All Calculation Checked	Yes (FTN Associates)	NA	NA
QC Limits Met	"H"		

Notes

No descriptive narratives were provided by FIU.

Holding time goals set for the length and weight measurements are guidelines only.

Documentation on how the fish samples were stored (Frozen, 4 C, preserved) was not provided.

Station ID **M5-828-FSF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	X	X	X
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers	"H"		

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

"H" Analysis digestion performed after holding times have expired.

"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

September (M5) 1999 Samples for Critical Parameters Analyzed by SERC With the 10% Full QA/QC Review

Station ID **M5-944-FSF** **Fish**

Laboratory Records	**Total Hg**	**Length**	**Weight**
Data Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sampling Location ID	X	X	X
Sample Type	FS = FI = Fh = Fish	FS = FI = Fh = Fish	FS = FI = Fh = Fish
Collection Date	09/22/99	09/22/99	09/22/99
Digestion Date	11/15/99	11/08/99	11/08/99
Analysis Date	11/15/99	11/08/99	11/08/99
QC Batch ID	The Batch Numbers are referenced by the Sample ID range. They are specific to this project.		
Digestion Volume	7 Fish	NA	NA
Total Volume	7 Fish	7 Fish	7 Fish
Sample Volume Analyzed	Aliquot	7 Fish	7 Fish
Dilution	500-1000	NA	NA
Results	120	See Database	See Database
Measuring Unit	ppb	mm	g
EPA Method	CVAF	Measurement	Measurement
Analyst	FDL/JL	MB	MB
Method Detection limit	3.2 ppb	NA	NA
Data Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	No	No
All Calculation Checked	Yes (FTN Associates)	NA	NA
Holding Time Met	No (Goal Only)	No (Goal Only)	No (Goal Only)
QC Report (Attached)			
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
QC Batch ID	HG15KF1	By: Date/Time	By: Date/Time
Method Blanks	All Good	NA	NA
Instrument Blanks	Good	NA	NA
Duplicates (RSD)	<20 RSD	NA	NA
Matrix Spike Recoveries (75-125)	All Good	NA	NA
Blank Spike/CCV Recoveries	All Good	NA	NA
Detection Range	3.2 ppb and >	NA	NA
Correlation Coefficient (>0.995)	0.9999	NA	NA
QC Report (Verified)			
Lab Data Entry Checked by Analyst	Yes (FTN Associates)	NA	NA
All Calculation Checked	Yes (FTN Associates)	NA	NA
QC Limits Met	"H"		

Notes

No descriptive narratives were provided by FIU.

Holding time goals set for the length and weight measurements are guidelines only.

Documentation on how the fish samples were stored (Frozen, 4 C, preserved) was not provided.

Station ID **M5-944-FSF** **Fish**

	Total Hg	Length	Weight
Narrative Description (Attached)	The Narrative Section will be written after all of the analyses are completed		
Total Samples/Matrix			
Methods/Parameters			
Range of Samples analyzed			
Holding Time Summary			
Analytical Problems			
QA/QC Acceptance Limits			
Integrity of Data Quality			
Deviations From SOP			
Observations			
Sample Management Records			
Sampling Location ID	X	X	X
Matrix	X	X	X
Preservative (Acid, Temp..)	Not Noted	Not Noted	Not Noted
Collection Data/Time	X	X	X
Laboratory ID Code	FIU/SERC Laboratory uses the same ID as the Sampling Station ID		
Sample Handling/Storage	Samples kept in the Hg laboratory area.		
Log-in Procedures	Internal COC	Internal COC	Internal COC
Raw Data (Attached)			
Sample Work Sheets	X	X	X
Sample Run Logs	X	X	X
Instrument Raw Data	X	X	X
Bench Sheets	Notes are the raw data and the instrument data.		
Sample Preparation Logs	X	X	X
Raw Data (Verified)			
Sample ID Transferred	X	X	X
All Calculation Checked	X	X	X
Measuring Unit	ppb	mm	g
PE Results (Attached)			
Organization	NA	NA	NA
Performance (Pass/Fail)	NA	NA	NA
Validation Criteria			
Applied Qualifiers	"H"		

X = Attached or Verified

Data Qualifiers

"J" Concentration reported should be considered an estimate. The data are acceptable for the use as determined by specific data users but certain QC criteria were not met

"Reject" Batch QC data did not meet DQO required accuracy/precision criteria required to allow use as stated

"R2" Correlation Coefficient is out of QAPP limits.

"M" Analyte exhibits potential matrix effect based on matrix spike recovery outside of 75 to 125% range.

"B" Analyte concentration in the associated blank was >3 times the MDL.

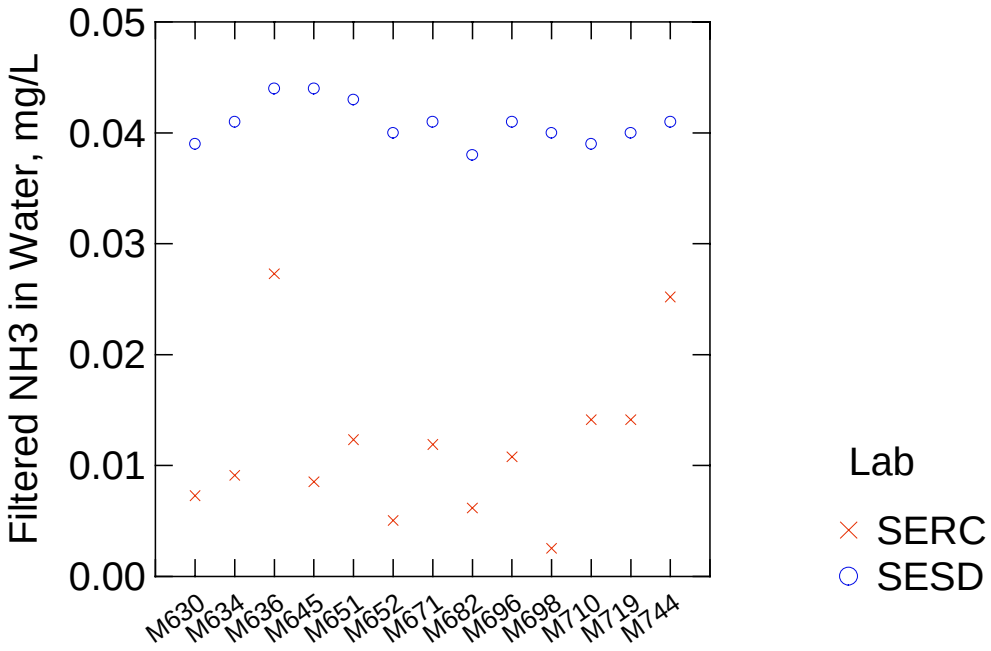
"H" Analysis digestion performed after holding times have expired.

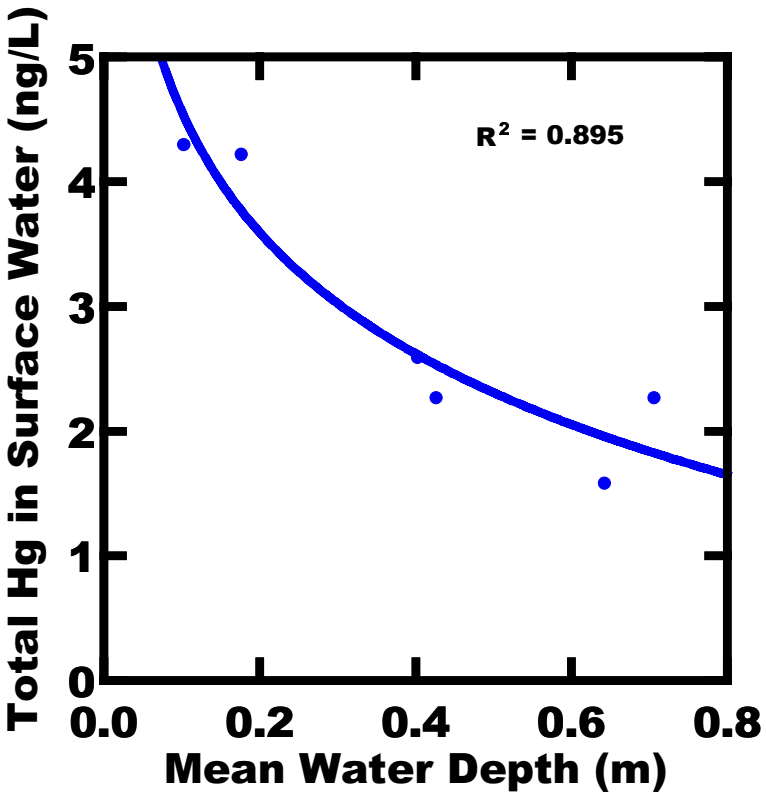
"NR" Data was unavailable for review.

"DQO" Precision and/or Accuracy results are out of the Data Quality Objective/QAPP control limits.

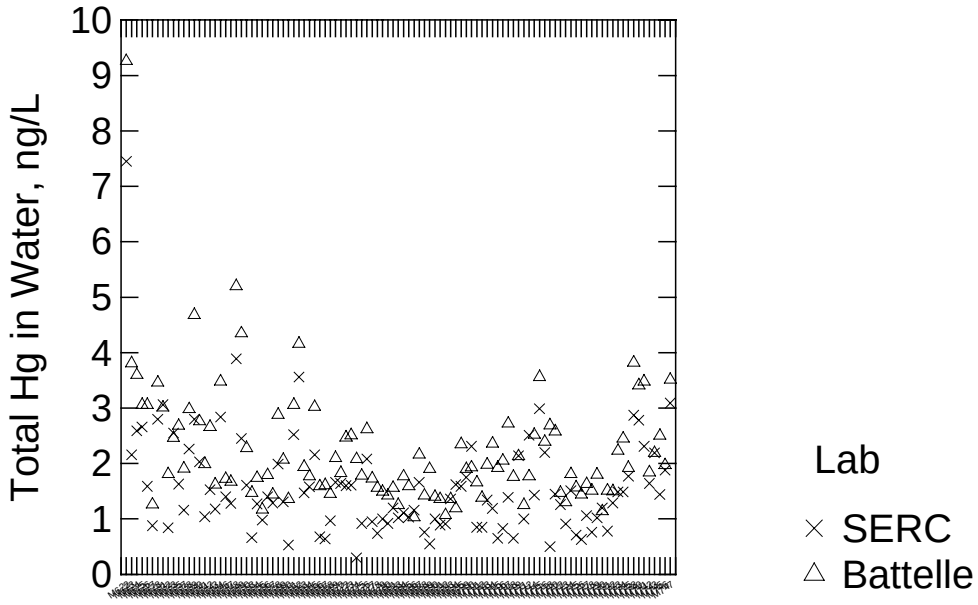
INTERLABORATORY COMPARISONS

September 1999 Samples

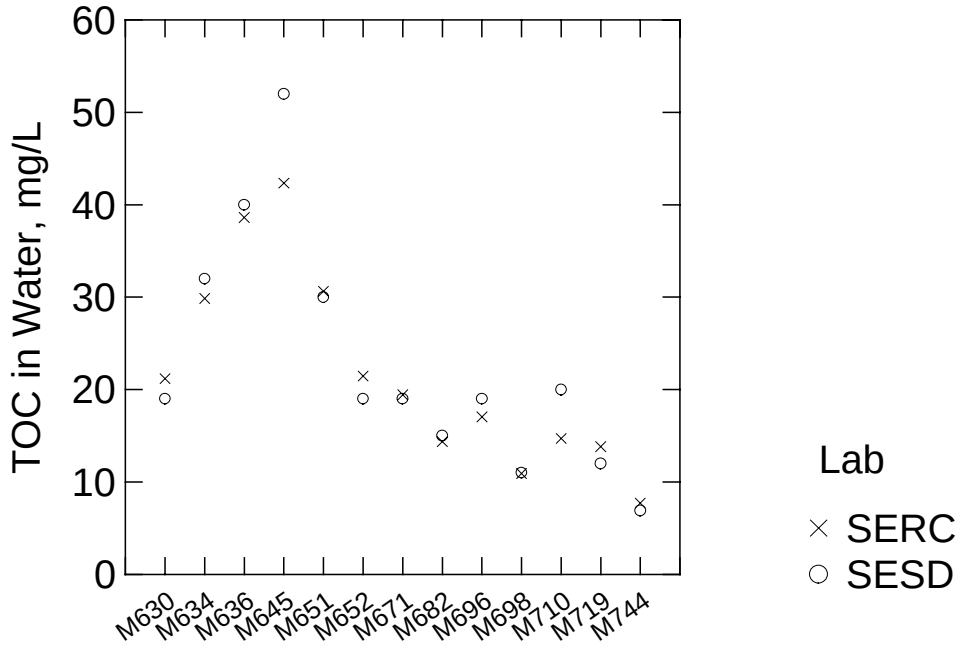



Total Hg in Surface Water (ng/L)
5
4
3
2
1
0
0.0
0.2
0.4
0.6
0.8
Mean Water Depth (m)
R2 = 0.895

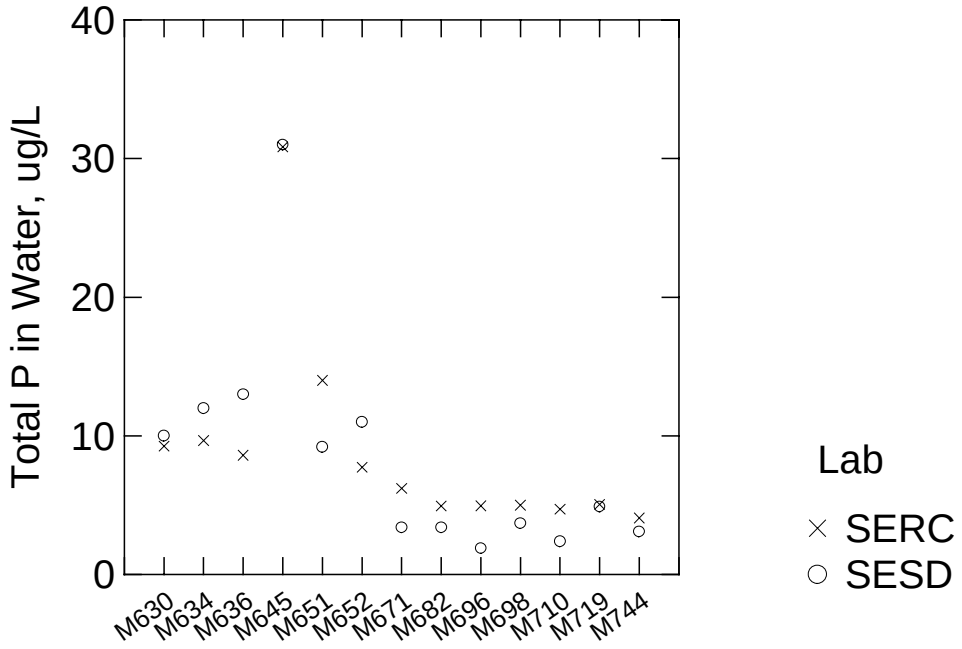
September 1999 Samples



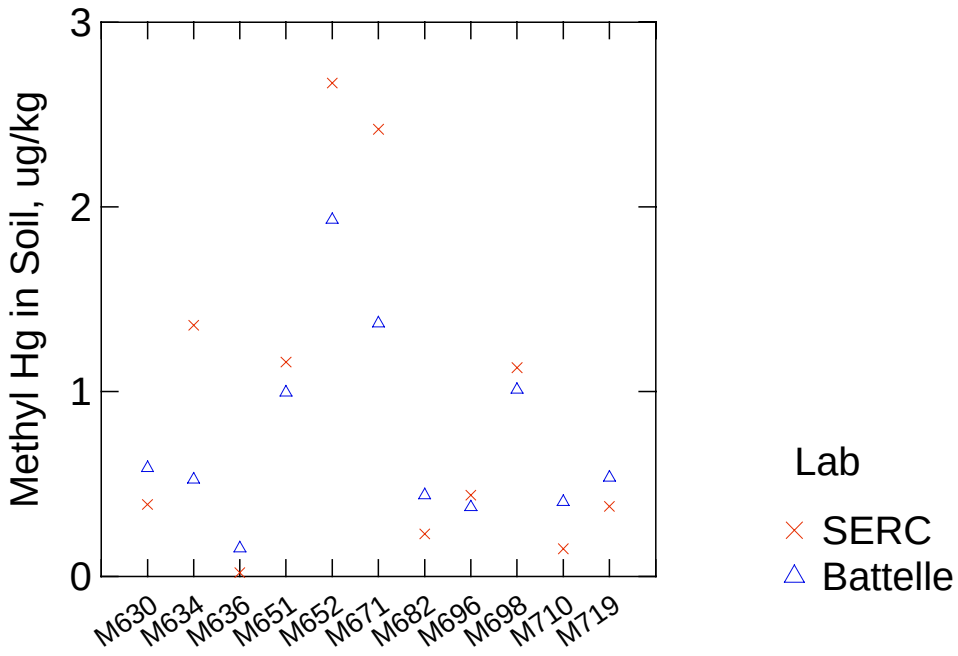
September 1999 Samples



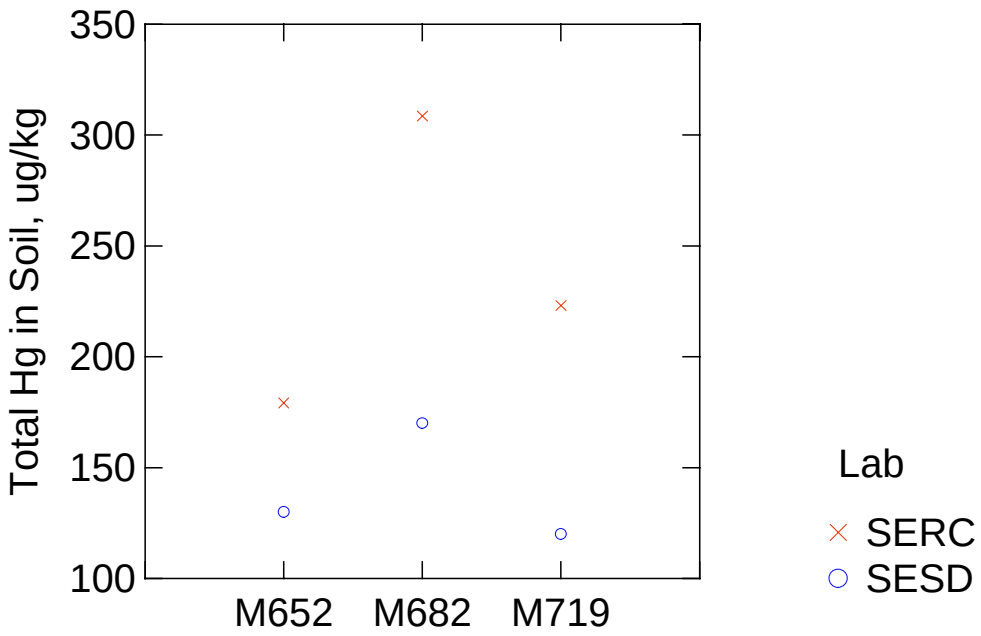
September 1999 Samples



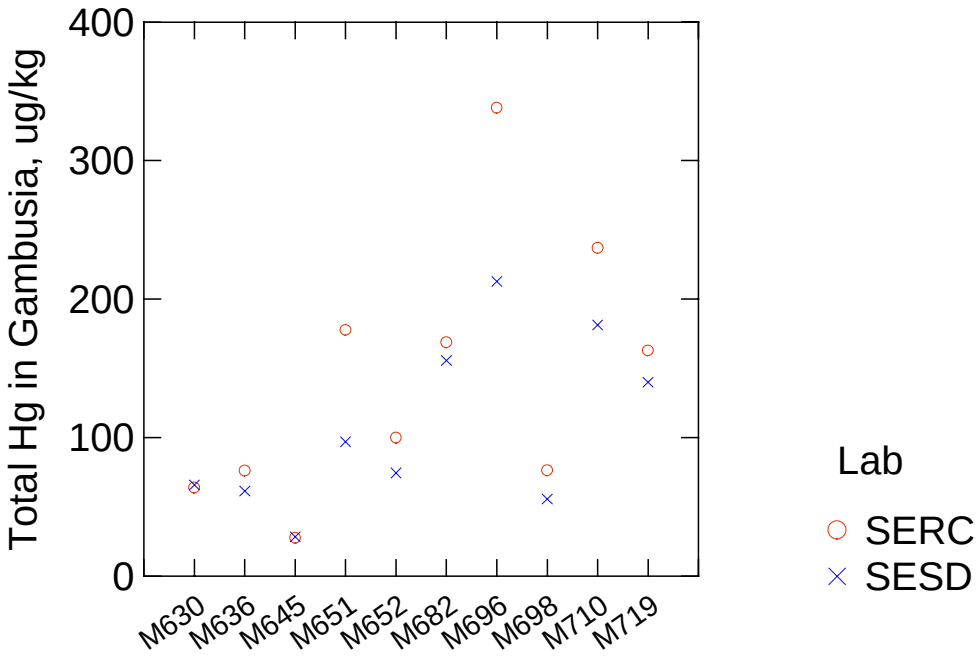
September 1999 Samples



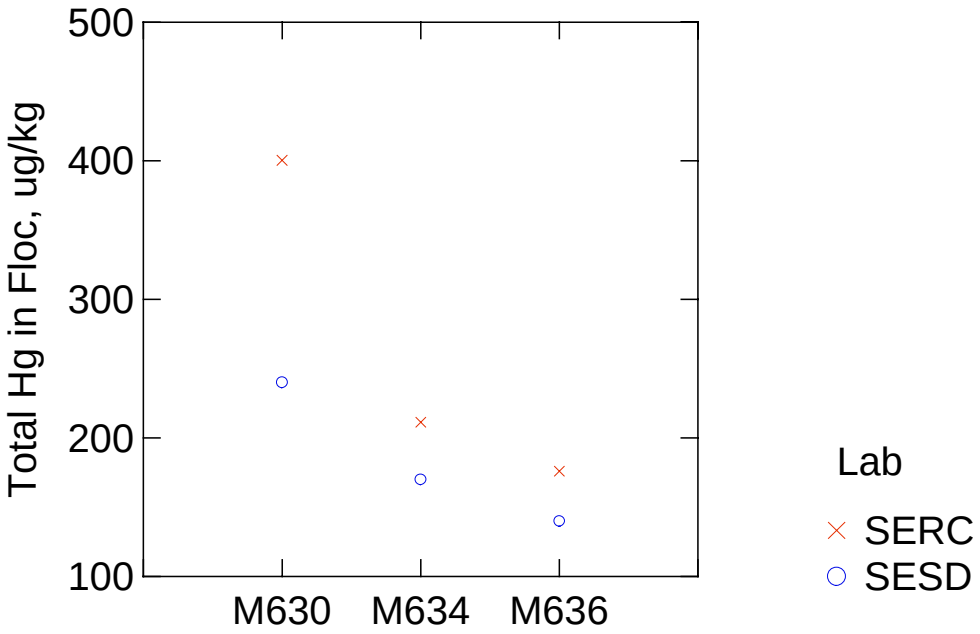
September 1999 Samples



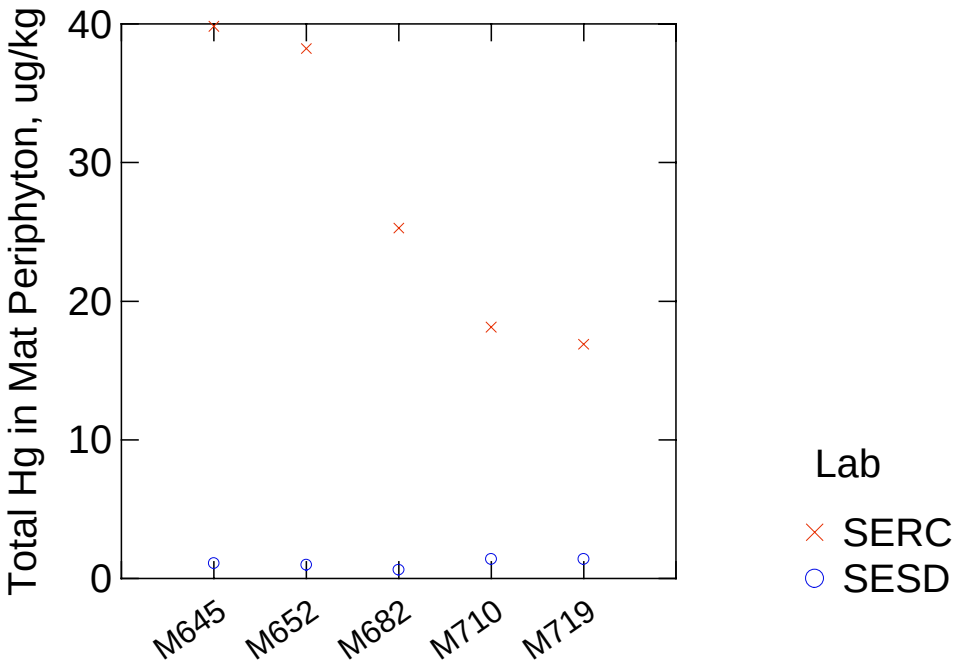
September 1999 Samples



September 1999 Samples



September 1999 Samples



APPENDIX D: Data Files

The following are Microsoft Excel spreadsheet files. These files should be able to load into a variety of other spreadsheet applications such Lotus 123, etc..You may need to specify within your application the file format (Excel *.xls).

P12join7FINAL.xls (multimedia chemistry data)

EPAM4M5.xls (diatom data)

NEWPAFIELD~JR1.xls (macrophyte presence / absence data)

ugacy45dom1.xls (aerial photo interp. of dominant vegetation (areas))

CYCLE4sec.xls (aerial photo secondary vegetation)

CYCLE5sec.xls (aerial photo secondary vegetation)

CYCLE4secP.xls (aerial photo percent secondary vegetation)

CYCLE5secP.xls (aerial photo percent secondary vegetation)

JRcljsagmorphclean.xls (macrophyte morphological data)

Guts_individual_fish.xls

The following files contain several 1 x 1 km map files (Adobe Acrobat pdf files)

Cycle 4, Everglades Agricultural Area (maps-cycle4-eaa.pdf)

Cycle 4, Everglades National Park (maps-cycle4-enp.pdf)

Cycle 4, Water Conservation Area 1 (maps-cycle4-wca1.pdf)

Cycle 4, Water Conservation Area 2 (maps-cycle4-wca2.pdf)

Cycle 4, Water Conservation Area 3 (maps-cycle4-wca3.pdf)

Cycle 5, Everglades Agricultural Area (maps-cycle5-eaa.pdf)

Cycle 5, Everglades National Park (maps-cycle5-enp.pdf)

Cycle 5, Water Conservation Area 1 (maps-cycle5-wca1.pdf)

Cycle 5, Water Conservation Area 2 (maps-cycle5-wca2.pdf)

Cycle 5, Water Conservation Area 3 (maps-cycle5-wca3.pdf)