

APPENDIX F
DATA ASSESSMENT REPORT

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**ASHLAND/NSP LAKEFRONT SITE
RI REPORT - APPENDIX F
WDNR BRRTS #02-02-00013
CERCLA DOCKET NO. V-W-04-C-764
USEPA ID# WISFN057952**

DATA ASSESSMENT REPORT

F.0 DATA QUALITY REVIEW

This Data Assessment Report (DAR) summarizes the usability of the analytical data generated for the Remedial Investigation (RI) at the Ashland/NSP Lakefront Superfund Site ("Site"). The Data Quality Indicators (DQIs) and Quality Assurance/Quality Control (QA/QC) objectives for field sampling and analysis relevant to the RI are documented in the following approved plans:

- Remedial Investigation/Feasibility Study (RI/FS) Work Plan Revision 02 Ashland/NSP Lakefront Superfund Site, Ashland, Wisconsin. (February 2005) ("RI/FS Work Plan");
- Quality Assurance Project Plan RI/FS Tasks, Revision 03, Ashland/NSP Lakefront Superfund Site, Ashland, Wisconsin. Remedial Investigation/Feasibility Study (February 2005) ("project QAPP"); and
- Field Sampling Plan, RI/FS Tasks, Revision 02, Ashland/NSP Lakefront Superfund Site, Ashland, Wisconsin. Remedial Investigation/Feasibility Study (February 2005) ("FSP").

In addition, the project QAPP was amended to include sampling protocols for smelt sampling and analysis (Addendum 01, April 2004), mobile laboratory analysis for reconnaissance activities (Addendum 02, May 2005), and ecological water and sediment studies (Addendum 03, June, revised October 2005). Sections of the aforementioned plans are referenced throughout this DAR to highlight the specific field sampling and analysis practices for the RI report.

The RI/FS Work Plan describes the field and laboratory activities for the following types of investigations for the Site:

- Surface and subsurface soil sampling
- Sediment reconnaissance sampling
- Fish sampling and tissue analysis
- Soil vapor probe and indoor air sampling (winter and summer events)
- Passive diffusion bag groundwater sampling
- Sediment sampling and forensic analysis
- Test pit and surface soil sampling
- Surface water sampling (low and high energy events)

Table F-1 provides a summary of the sampling periods and matrices.

The data quality and usability of the results for these investigations are summarized in this DAR. Data quality assessments for benthic invertebrate and physical/hydraulic parameter collection are not presented in this DAR since the data quality assessment process for these sampling programs is not part of United

States Environmental Protection Agency (USEPA) *Contract Laboratory Program National Functional Guidelines for Organic Data Review* (1999) and USEPA *Contract Laboratory Program National Functional Guidelines for Inorganic Data Review* (2004) and was not a specific requirement of the project QAPP.

The major components of the QA/QC program include the collection and analysis of QC samples, the use of standardized SOPs for field and laboratory activities, and data validation of analytical results. Sections F.1, F.2, and F.3 describe these components of the QA/QC program in greater detail. Sections F.4 through F.9 summarize the data validation results for each laboratory and sampling program for the RI. The collection program was evaluated by reviewing field logbooks. The review is described in Section F.10. The data usability/assessment for the entire data set is found in Section F.11. The data from the following analytical laboratories (and investigations) were evaluated for the RI:

- Northern Lake Services, Inc. (NLS), Crandon, Wisconsin – soil, sediment, groundwater (including diffusion bags), and surface water analyses;
- Environmental Chemistry Consulting Service (ECCS), Madison, Wisconsin – reconnaissance sediment analyses using an onsite mobile laboratory;
- Severn Trent Laboratory (STL), Knoxville, Tennessee – soil gas and vapor intrusion analyses
- STL, Burlington, Vermont – fish tissue and sediment analyses; geophysical analyses;
- Pace Analytical, Inc., Kimberly, Wisconsin and EnChem, Madison, Wisconsin laboratories (Pace/EnChem) – bioassay water and tissue analyses;
- Teledyne Brown Engineering (TBE) – radiochemistry age-dating of sediment cores;
- Woods Hole Group Environmental Laboratories (WHGEL), Raynham, Massachusetts - hydrocarbon characterization¹; and
- University of Kentucky (UK), Lexington, Kentucky - organic petrology measurements¹.

Other laboratories providing historic and/or air testing information for the RI report included STL (Chicago, Illinois), EnChem (Madison, Wisconsin), Enviroscan (Lancaster, Pennsylvania), Test America (Cedar Falls, Iowa), and Wisconsin Industrial Hygiene (Madison, Wisconsin). The data usability for the historic data is presented (where available) in the historical reports or other RI documents, but is not part of this DAR. A summary of the historic reports is found in Section 2.4 of the RI/FS Work Plan (URS, 2005c).

F.1 QUALITY CONTROL SAMPLE COLLECTION

Field and laboratory QC samples were collected and analyzed at different frequencies depending upon the sampling program and matrix collected. Field QC samples were collected for the various matrices based on specifications in the project QAPP and its addenda. Similarly, laboratory QC samples were analyzed based on the requirements of the reference analytical test method that was performed and the laboratory-specific standard operating procedures (SOPs). Field QC samples included equipment blanks, field blanks, trip blanks, methanol blanks, field duplicates and matrix spike/matrix spike duplicate (MS/MSD) samples. Laboratory QC samples included method blanks, laboratory control sample (LCS), and MS/MSD, and for metal analyses, contract required detection limit (CRDL) standards, serial dilutions, and post-digestion spikes (PDS). The collection frequency for field and laboratory QC samples followed

¹ QA/QC procedures for hydrocarbon characterization are found in Appendix A of the project QAPP (URS, 2004b). This DAR does not include a summary of the data usability associated with the hydrocarbon characterization of sediments. See Section F.3 for further details.

criteria specified in Section 4.2 (Measurement Performance Criteria - PARCC) and Section 5.13 (Collection of Quality Control Samples) of the project QAPP (URS, 2005b). Table F-2 shows the field QC samples collected or performed per matrix per laboratory (for the two-year groundwater sampling program and the 2005 soil/sediment/test pit programs). The laboratory QC samples analyzed per matrix per laboratory for the sampling programs are summarized in the laboratory SOPs provided in each of the QAPPs. Summaries of the findings associated with the field and laboratory QC analyses are found in Sections F.4 through F.9. These sections describe the individual laboratory performance for the RI work during the period from December 2003/January 2004 (initiation of groundwater sampling) through November 2005 (completion of RI sampling activities).

F.2 STANDARD OPERATING PROCEDURES

Field and laboratory SOPs for the RI are found in the RI/FS Work Plan (URS, 2005c), FSP (URS, 2005a) and QAPP and its addendums (URS, 2005b). Field SOPs were followed for sample collection, handling and decontamination, and the sediment and fish sampling conducted during the RI. Laboratory SOPs were followed for analytical, geotechnical, and toxicity testing. Analytical laboratories (i.e., those performing chemical testing) were required to meet analytical sensitivity requirements for the generation of data for most samples; geotechnical laboratories were required to follow standards procedures (e.g., grain size) to measure the physical nature of the matrix sample; and toxicity testing laboratories were required to follow specific protocols to assess potential ecological impact to aquatic/sediment habitats. These requirements were listed in the laboratory-specific SOPs. All SOPs for the analytical, geotechnical and toxicity testing were included in the reference documents listed in Section F.1.

F.3 LABORATORY DATA VALIDATION AND USABILITY ASSESSMENT

Data validation consists of evaluating the completeness, correctness, and conformance of a data set against the method, SOP, or contract requirements documented in the approved QAPP. Data assessment is the process of evaluating validated data to determine if they can be used for the purpose of the project (i.e., to answer the environmental questions or to make the environmental decision that must be made).

Validation of analytical data was based on principles found in the USEPA guidance documents, *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review* (1999) and *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review* (2004). Data validation is a comprehensive review and examination of the raw data and subsequent recalculation of results to verify the completeness of documentation and the accuracy of reported results. The validation also includes an assessment of sample handling, field QC sample analyses, and laboratory QC performance. Specifically, the items reviewed during the data validation process were:

- Analytical methods performed and test method references
- Sample Condition - review of log-in records for cooler temperature, presence of headspace, chemical preservation, etc.
- Holding times (comparison of collection, preparation, and analysis dates)
- Analytical results (units, values, significant figures, reporting limits, including any matrix interference problems, analyst, percent moisture)
- Sample traceability (Chain of Custody documentation)
- Method blank results and laboratory contamination
- Laboratory control sample (LCS) results and comparison to laboratory control limits
- Matrix spike/matrix spike duplicate (MS/MSD) results and comparison to laboratory control limits
- Field replicate/duplicate results and comparison to data review criteria

- Surrogate recoveries (where applicable) and comparison to laboratory control limits
- Internal standard values (where applicable) and comparison to laboratory control limits
- Instrument calibrations (initial and continuing)
- Chromatograms (form, structure, baseline)
- Quantitation reports (calculations)
- Mass spectra (GC/MS only)
- Internal standards
- Retention times
- Run logs/sequence logs
- Preparation/extraction logs
- Interference check samples (inorganics only)
- Instrument detection limits (inorganics only)
- Serial dilutions (inorganics only)

Data Validation Reports (DVRs) were prepared for all validations. Data validations were completed by Environmental Data Services (EDS), Concord, New Hampshire, and URS Corporation (URS), Chicago, Illinois. Each DVR contains a comprehensive listing of samples reviewed (field sample identifications and laboratory sample numbers), validation review elements, a summary of the individual method's performance, and the sample data sheets showing data validation qualifiers (and reasons for qualification) that were applied to the sample results. Edits were not made to the laboratory's sample data sheets if the performance and QC sample results were within laboratory or project criteria. When QC results indicated inadequate performance, the validation group applied data qualifiers to the results to inform the data user of the possible performance problem. These qualifiers are in addition to, or a revision of, the qualifiers provided by the laboratory. Data qualifying protocols were based on guidance from the CLP guidelines (USEPA, 1999 and USEPA, 2004a), the laboratory-specific SOPs, and SW846 guidance (USEPA, 2004b). The data qualifiers are presented on the laboratory analysis forms attached to the individual DVR included in Appendix G and are also shown on the final tables for the RI. The laboratories' original analysis reports are included in Appendix E.

On the basis of the validations, data were 1) accepted without qualification, 2) qualified as estimated ("J" for positive results, or "UJ" for non-detect results), or 3) rejected ("R"). A "J" qualifier was appended to those detected and undetected values that were usable, but should be considered estimated due to laboratory and/or field performance problems. In the case of rejected data, an "R" qualifier replaced the sample values, which were deemed unreliable, and therefore, not usable. Rejected data are data where the presence or absence of the result could not be determined nor supported by the results of laboratory method calibrations or other QC analyses.

Data qualifiers were added to the results in the electronic database by Newfields, Inc. (Newfields), Madison, Wisconsin, after completion of data validation. A printout of the final electronic data deliverable (EDD) for the RI, showing post-validation qualifiers and data revisions, is found in Tables 4-3 through 4-14. Consistent with the approach stated in the project QAPP (URS, 2005b), 100 percent of the analytical data was required to be validated for the RI. It should be noted that data validation was not performed on the analytical reports from the Woods Hole Group Environmental Laboratories (WHGEL), Raynham, Massachusetts, for hydrocarbon characterization and the University of Kentucky (UK), Lexington, Kentucky, for the organic petrology measurements due to an oversight. Data validation is currently in progress and will be reported in the June 2006 NSP/Lakefront Progress Report. Any impacts

and changes to the use of the data will be documented in the Progress Report. The WHGEL and UK laboratories' analysis reports are included in the Forensics Report found in Appendix D3 of this RI report.

Data assessment of the validated project data involves the evaluation of the data quality in terms of precision, accuracy (bias), [sample] representativeness, completeness, and comparability (PARCC). It also includes an evaluation of the method sensitivity and quantitation limits for each matrix. The data quality indicators were evaluated by assessing the following:

- Conformance to sample collection procedures
- Conformance to analytical methodologies
- Sample handling and chain-of-custody (COC) issues
- Adherence to sample holding times
- Results of field and laboratory QC samples
- Instrument calibration and tuning (where required)
- Analytical performance checks
- Analytical sensitivity of results
- Completeness, comparability, and representativeness of results
- Laboratory flags and codes

The following, Sections F.4 through F.9, describe the findings of the validations for the laboratories performing testing for the RI.

F.4 NORTHERN LAKE SERVICES, INC. (NLS)

NLS performed analysis of groundwater (including passive diffusion bags), surface soils, subsurface soils, test pit samples, surface water, and sediment analyses for the RI. Data validation was performed on all NLS analytical reports in accordance with the evaluation program described in Section F.3. This included a comprehensive review of the laboratory analysis reports for completeness, QC results, sample handling, hold time compliance, accuracy, precision, and reported detection limits. A summary of the results of the data validation for the NLS analytical reports is provided in Sections F.4.1 through F.4.11.

F.4.1 Conformance to the Sample Collection Procedures

Sample custody for the laboratory was in accordance with the FSP (URS, 2005a) and the project QAPP (URS, 2005b). Few deviations with respect to sample collection and/or custody were noted in the DVRs. The deviations that were found included incomplete COC records (e.g., the sample ID was left off of the COC form, e.g., NS-GW-MW7A) or revision of the analysis request (e.g., hexavalent chromium was shown on the COC but analysis was cancelled per project management request). The COCs that were completed for each sampling event were included in the laboratory analysis reports provided in Appendix E.

Table 9 of the project QAPP specifies the container and preservation requirements for groundwater, soil and sediment samples. Sample collection procedures for aqueous and solid matrices were in accordance with SOPs provided in the project FSP (URS, 2005a). For aqueous sample collection, 40-milliliter (mL) glass vials were used for VOCs, 1-Liter (L) amber glass jars were used for SVOCs, and 125 to 250-mL plastic bottles were used for inorganic parameter groups. Chemical preservation was in accordance with requirements. For solid sample collection, 60-mL or larger, glass jars were used for VOC and SVOCs. For all other parameter groups, the laboratory provided appropriate glass containers. All sample containers were pre-cleaned in accordance with NLS cleaning requirements found in Appendix B of the project QAPP (URS, 2005b).

F.4.2 Conformance to Analytical Methodologies

Analyses were performed according to current versions of the laboratory SOPs provided in the project QAPP (URS, 2005b) and included the following parameter groups and test methods:

- VOCs – SW846² Method 8260B; Method 5030 (aqueous); Method 5035 (solids)
- SVOCs – SW846 Method 8270C (full scan for solids); Method 3510C (aqueous); and Method 3550B (solids)
- PAHs – SW846 Method 8270C (selective ion monitoring [SIM] mode for aqueous); Method 3510C (aqueous); and Method 3550B (solids)
- Polychlorinated Biphenyls (PCBs) – SW846 Method 8082; and Method 3550B (solids)
- Organochlorine (OC) Pesticides – SW846 Method 8081A; and Method 3550B (solids)
- Total Cyanide – EPA³ Method 335.4
- Turbidity – EPA Method 180.1
- Total Suspended Solids – EPA Method 160.2
- Oils/Grease – 40 CFR Part 136 Method 1664
- Total Metals – SW846 Method 6010B; Method 7041 (antimony); Method 7060 (arsenic); Method 7740 (selenium); Method 7841 (thallium); and Method 3005 or 3050M (aqueous digestion)
- Mercury – 40 CFR Part 136 Method 1631
- Hexavalent Chromium – SW846 Method 7196A
- Dissolved Organic Carbon (DOC) – SW846 Method 9060
- Percent Solids – EPA Method 160.3 (modified)

No deviations in the specified procedures were described in the laboratory analysis reports or in the DVRs for the aforementioned methods.

F.4.3 Sample Handling and Chain-of-Custody

Sample handling and COC procedures followed the procedures outlined in the project QAPP (URS, 2005b). The field team placed all samples on ice in a cooler until packaging for shipment to NLS. The exterior of the bottles were cleaned, checked for adequate labeling, wrapped to prevent breakage, and then packed on ice in a cooler for shipment. A COC form was completed for each cooler. Shipment was conducted using either private (e.g., URS) or commercial couriers. Copies of the completed forms (after receipt at NLS) are provided in Appendix E of the RI Report. Once received, NLS assigned a unique laboratory identification number to each sample and a Sample Delivery Group (SDG). The SDG is the means by which the laboratory identifies and groups samples for reporting purposes.

F.4.4 Adherence to Sample Holding Times

Holding times were met for most project sample analyses with the exception of several sets of re-analyses for SVOCs for some soil samples, several VOCs for some aqueous samples (e.g. NSP-SW-ER3-1105-NB-FI), and several original analyses for hexavalent chromium for the groundwater samples. Affected results were qualified as estimated (“J” flag for positive results and “UJ” for non-detect results) on the basis of the holding time exceedances. Results associated with analyses performed past recommended hold times may be negatively biased since the chemical of concern may be lost during extended storage. For the soil/sediment analyses, no results were rejected based on holding time issues. For the groundwater

² *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, (SW846) USEPA, Final Update IIIB, November 2004.

³ *Methods for Chemical Analysis of Water and Waste*, (EPA600/4-79-020) USEPA, Third Edition, March 1983.

analyses, several non-detect SVOC results (e.g., SDG 364754) were rejected due to missed holding times. These results were not usable for quantitative purposes per the DQOs established for this project.

F.4.5 Results of Field and Laboratory QC Samples

NLS analyzed field and laboratory QC samples in accordance with QAPP requirements. The following paragraphs explain the general findings of the QC sample analyses performed.

Laboratory Control Samples and Matrix Spike/Matrix Spike Duplicates

The laboratory performed LCS analyses for all methods in accordance with laboratory-specific SOPs. Percent recoveries and statistical control limits were reported for each LCS spiked compound. Percent recovery results outside of statistical control limits were noted by NLS and further qualified in the DVR as estimated (“J” flag for positive results and “UJ” for non-detect results). In general, the accuracy of the analyses was acceptable for most analytical groups. It should be noted that the DVRs described several SDGs where the LCS results for VOC analyses were not included in the laboratory analysis reports. No results were qualified as estimated by the validation group⁴ due to the lack of LCS results since NLS indicated that they frequently use their continuing calibration verification (CCV) as an LCS analysis for the VOC method (i.e., the laboratory evaluated the percent recoveries of each compound to assess accuracy). The NLS CCV is similar to an LCS except that the CCV is not prepared from a second source. NLS stated that they do perform LCS analyses for the majority of batch analyses but not all. Additionally, they stated that if the MS/MSD analysis showed acceptable recoveries and RPDs, then the CCV and/or LCS results would not be provided. This reporting practice is outlined in the laboratory-specific SOP for VOCs in the approved QAPP (URS, 2005b) and was followed for all samples analyzed by NLS.

NLS also performed MS/MSD analyses on multiple project samples for all parameter groups. This included 32 spikes for VOCs, 31 spikes for SVOCs, 36 spikes for metals/cyanide, and 1 for OC Pesticides for the soil/sediment matrices, and 45 spikes for VOCs, 35 spikes for SVOCs, and 64 spikes for metals/cyanide for the groundwater/surface water matrices. (No aqueous samples were analyzed for OC Pesticides.) The project samples for VOC, SVOCs, OC Pesticides, and metals were spiked with the target chemical list and percent recoveries reported for each. The project samples for PCB analysis were spiked with two target chemicals, Aroclors 1016 and 1260, per the laboratory SOP. Similar to the LCS analyses, percent recoveries and statistical control limits were reported for each MS/MSD compound. Percent recoveries outside of statistical control limits were noted by the laboratory and further qualified in the DVR to show the impact on the analytical results. It should be noted that if the spike concentration was significantly less (four times) than the project sample concentration, the laboratory would not calculate the percent recovery. Qualification of data was not performed in these cases. The DVRs identified acceptable spike recoveries for most parameter groups and matrices. For the soil/sediment analyses, only a few VOCs (e.g., toluene in SDG 375103) and metals were rejected based on MS/MSD recoveries. For the groundwater analyses, only antimony was rejected in several SDGs (e.g., 365504) due to significantly low MS/MSD recoveries (less than 10 percent). These results are not usable for quantitative purposes per the DQOs established for this project. In general, the accuracy of all analytical methods performed by NLS was acceptable for all analytical groups.

Method Blanks, Equipment Blanks, Field Blanks, and Trip Blanks

For the groundwater and soil investigations, the field QC samples included trip blanks, equipment blanks, field blanks, and methanol blanks. These were collected and analyzed at a frequency of one or more per day for the quarterly groundwater sampling event and approximately once per day for the

⁴ EDS, Concord, New Hampshire

soil/sediment/surface water investigations. The trip blanks and methanol blanks were analyzed for VOCs. The field blanks and equipment blanks were analyzed for VOCs, SVOCs, and metals (26 inorganics including hexavalent chromium, trivalent chromium, total chromium, and cyanide). Field blanks for the sediment and water quality studies also included filtered and unfiltered samples and the analysis of dissolved organic carbon. Laboratory blanks included method blanks and instrument blanks where required. These were analyzed at least once per batch in accordance with the laboratory SOPs.

All field QC samples were sent to NLS for analysis. No SVOCs were detected in the field blanks, but one SVOC (1-methylnaphthalene) was detected in two of the equipment blanks. The equipment blanks also contained multiple metals, the most frequent of which were zinc, nickel, aluminum, sodium, manganese, and chromium. The impact to the associated project samples was to qualify affected results based on an action level of 5 times the blank result. Few metals were detected in the field blanks. One VOC (toluene) was frequently detected in nearly all trip blanks, and most equipment and field blanks. After a thorough evaluation of its laboratory water system and solvents, NLS identified that toluene was most likely introduced into the field QC samples by cross contamination during sample storage and shipment. (Methanol readily absorbs volatile compounds from the atmosphere.) To test this theory, NLS sent methanol blanks with the pre-cleaned bottle shipments to the site in April and June 2005. After field storage and shipment, the results of the methanol blank analysis showed the presence of toluene even though none was present in the solvent prior to shipment. The results indicate that the toluene in some samples does not accurately reflect the actual environmental concentrations at the site. Data validation and qualification of results was based on this information. If the equipment blank, field blank, or trip blank contained a positive concentration of a chemical, then the associated project sample result was evaluated for potential contamination. If the sample result was positive, the result was revised to non-detect ("U" flag) if it was less than five times the concentration in the blank (i.e., action level). This qualification practice avoids the use of analytical results that are affected by cross-contamination and that are not truly representative of site conditions.

Laboratory blanks included method blanks, preparation blanks, and calibration blanks (as appropriate for the method) and that were analyzed at the frequency specified in the laboratory's SOPs. The results of the laboratory blank analyses for VOCs, SVOCs, and metals revealed very little laboratory organic and inorganic contamination. Method blanks for the metals analyses seemed to show the most frequent presence of several elements (e.g., aluminum, chromium, etc.). No contamination was found in the method blanks associated with the other methods (e.g., OC Pesticides) performed by NLS. For the VOC, SVOC, and metal analyses, the presence of these chemicals in the blanks indicates the possibility of false positives in the associated project samples. Similar to the qualification for field blanks, sample results were compared to action levels (i.e., 10 times for common laboratory contaminants or five times for non-common contaminants) established for each blank contaminant and the data qualified as non-detect ("U" flag) when less than the action level.

Duplicate Samples

Field duplicates were collected at a frequency of approximately one sample for every ten project samples for both solid and liquid matrices. The required rate of collection was 10 percent per QAPP requirements. The results of the field duplicates generally demonstrated good agreement (RPD less than 50 percent) for the majority of parameters and samples. However, the RPDs of the SVOC and inorganic analyses seemed to show greater imprecision than the results for VOCs. Sample homogenization techniques used by field staff coupled with method extraction techniques are thought to have yielded divergent results. Selected chemicals were qualified as estimated ("J" for positive results and "UJ" for non-detected results) based on

RPDs that exceeded 30 percent for aqueous samples and 50 percent for solid samples. No results were rejected based on field duplicate results.

Laboratory duplicate samples were not required to be analyzed for this project with the exception of the MS/MSD duplicates. MS/MSD analyses were performed at a frequency of at least one per 20 samples for all parameter groups. Agreement between the two sample results, expressed as RPD, met the criteria of the analytical methods for the primary methods of analysis (VOC, SVOC, and inorganics) and for other analyses (PCBs, hexavalent chromium, etc.) in most cases. Where the RPDs were greater than the laboratory's corresponding upper control limits, the results were qualified as estimated ("J" flag for positive results and "UJ" for non-detect results).

F.4.6 Instrument Calibration and Tuning

Initial and continuing calibrations were reviewed for all NLS analysis reports for all methods. Instrument tuning was reviewed for all VOC and SVOC methods. In general, calibrations were performed at the designated frequency and met the laboratory acceptance limits/criteria specified in the SOP. A few chemicals did not meet initial or continuing calibration criteria. These chemicals, in the associated samples, were qualified as estimated ("J" for positive results and "UJ" for non-detect results). Instrument tunes for the VOC and SVOC analyses met the specified frequency in all cases. No data were qualified on this basis.

F.4.7 Analytical Performance Checks

Performance checks such as surrogate recovery and internal standard responses were also reviewed during data validation of the VOC and SVOC analyses. Surrogate percent recoveries were reviewed in the PCB and OC Pesticide analyses. Performance checks for inorganic analyses included Inductively Coupled Plasma (ICP) serial dilution analyses, ICP interference check sample results, and CRDL standard analysis.

Generally, surrogate recoveries were within acceptable limits for all OC Pesticides analyses and most VOC, SVOC, and PCB analyses. Surrogate recoveries were outside acceptance criteria (less than 10 percent) in a limited number of samples. The results for these samples were qualified based on USEPA CLP guidance (USEPA, 1999).

Internal standard performance, which is used to assess the condition of the analytical instrumentation for the VOCs and SVOC analyses, was acceptable for the majority of samples. If an internal standard was outside acceptable limits, the results for chemicals quantitated using the non-compliant internal standard were qualified as estimated ("J" for positive results and "UJ" for non-detect results) in the associated samples. The chemicals affected by poor internal standard responses are listed specifically in the DVRs in Appendix G.

ICP interference check samples were run at the required frequency. In a limited number of cases, positive or negative interference was noted. Sample results were qualified as estimated ("J" for positive results and "UJ" for non-detect results) as appropriate. ICP serial dilutions were performed periodically to assess potential bias due to interferences from the sample matrix. For selected samples, the results of the serial dilution exceeded the 10 percent acceptance limit. Positive and non-detect results for associated samples were qualified as estimated ("J" or "UJ"). CRDL samples were run frequently to assess the accuracy of the calibration near the reporting limit. Standard results were acceptable in all cases.

F.4.8 Analytical Sensitivity of Results

The quantitation limits conformed to the target quantitation limits in the project QAPP (URS, 2005b), which were established using risk-based DQLs. Analytical dilutions were performed on selected samples based on the concentrations of target compounds. Where dilutions were performed to quantify elevated levels of target compounds (concentrations above the upper calibration standard), the original results were removed from the database and replaced with the dilution results. (Note: the original results for all other chemicals were reported from the initial analysis.) This practice ensured that only quantitative results were available in the database for risk evaluations.

Dilutions were also required on the basis of sample matrix interference. Quantitation limits for diluted samples were raised by a factor equivalent to the dilution factor (i.e., results were reported with elevated reporting limits). These results are still usable for quantitative purposes; however, the elevated reporting limits may prevent conclusive confirmation that the target chemicals of concern are not present at a specific location if the reporting limit is above the associated risk screening level.

Additionally, some results were rejected as a result of the data validation process. Rejected data are not usable for quantitative purposes, but may have some value as qualitative information to the data user to guide further evaluations of the Site and/or ecological conditions.

Results for Tentatively Identified Compounds (TICs) were provided by the laboratory for one group of surface water samples (i.e., high energy sampling event) for both VOC and SVOC analyses. The TIC results, however, were not provided in the laboratory EDD. They were included in the raw data only. The TIC summary forms are provided at the end of the respective DVRs in Appendix G or in the NLS analysis reports in Appendix E. The applicable SDGs for the surface water samples are 94479, 94480, 94481, 94482, and 94483.

F.4.9 Representativeness, Completeness, and Comparability of Results

Representativeness expresses the degree to which data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, or an environmental condition. Representativeness is a qualitative parameter that is dependent upon the proper design of the sampling program and/or proper laboratory protocols. This sampling network was designed to provide data representative of site conditions. Representativeness was satisfied by ensuring that the QAPP was followed, proper sampling techniques were used, proper analytical procedures were followed, and holding times of the samples were not exceeded in the laboratory. The majority of these factors were addressed/reviewed during the data validation process. Evidence of proper sampling techniques was obtained by the review of field logbooks and assessment of field duplicate results. A summary of the logbook review is found in Section F.11. The precision associated with duplicate sample analysis is discussed in Section F.4.5. In general, documentation of appropriate field sampling procedures was complete and field precision was very good in most cases for aqueous and solid matrices.

Data completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount of data expected to be obtained under normal conditions. (See calculation in Section 4.2.4 of the project QAPP.) Valid data is defined as data that are considered usable (i.e., non-rejected results) after the validation process. The amount of expected data refers to the number of measurements planned. The completeness goal (specified in the project QAPP) required that each chemical have a completeness value greater than 95 percent. This goal was obtained for the NLS data sets for all chemicals measured in the groundwater, surface water, soil, and sediment matrices. (Note: Analytical results associated with sample re-analysis were not counted toward the completeness goal.) Some parameter groups had no rejected data

(e.g., OC Pesticides, PCBs); therefore, the amount of valid data available for use was 100 percent. The data generated by NLS for all chemicals of concern for the RI, therefore, meet the completeness criteria.

Data comparability expresses the confidence with which one data set can be compared to another; in this case, historical data compared to data collected during the 2005 investigation. Comparability is dependent upon the proper design of the sampling program and the use of proper sampling techniques. For the groundwater/aqueous matrices, analytical data collected during the two-year period of sample collection were considered comparable since one analytical laboratory performed the testing and, in general, the same contractor performed the actual field sampling. NLS followed the laboratory-specific SOPs for analysis of all groundwater samples during this time. Analytical data for the solid matrices (soils/sediments) were considered comparable based on the use of the same USEPA reference test methods (SW846) for the analysis of VOCs, SVOCs, metals, etc., and similar data validation protocols (USEPA, 1999; USEPA 2004). Although laboratory QC requirements may have differed slightly between analysis groups, the reference method procedures and validation practices are considered standard for all CLP work and most likely would ensure comparable data for the organic analyses. An in-depth verification of comparability to the historical data set was not conducted for this assessment summary.

F.4.10 Laboratory Flags and Codes

For the organic results, the laboratory used flags (e.g., “[]” to mean estimated data; ND to mean a non-detect result) that were not consistent with project requirements. During the data validation process and database management, these flags were updated to reflect the requirements in the project QAPP (URS, 2005b). Where the laboratory used brackets to indicate estimated data between the limit of quantitation (LOQ) and the limit of detection (LOD), the results were retained and a “J” qualifier was used. Where the laboratory used an “ND” code, the results were updated to show the LOQ and a “U” qualifier to indicate a non-detect value. (During the data validation process, where field and laboratory blank contamination was found, the results originally reported as positive values would have been revised to non-detect results when the project sample results were five times less than [or 10 times for common contaminants] the blank concentrations) Updates and adjustments to the laboratory qualifiers to meet project requirements are documented on the printouts of the analysis summary sheets found in each of the DVRs in Appendix G.

F.4.11 Summary of NLS Laboratory Performance

Nearly all the analytical data generated by NLS were valid and could be used with confidence for decision-making. Those data points indicated with a "J" qualifier should be considered estimated because of non-conformance with criteria established in the analytical methods or USEPA validation protocols. Those data points qualified with an “R” are rejected and should not be used for quantitative purposes. Only a few results were rejected because of laboratory QC performance (i.e., spike recoveries) but this did not impact the overall usability of the data set because re-analysis results were available, in general, to replace rejected data.

F.5 ENVIRONMENTAL CHEMISTRY CONSULTING SERVICES (ECCS)

Environmental Chemistry Consulting Service, Inc. (Madison, Wisconsin) performed analyses for the reconnaissance sediment investigations conducted in May and June 2005. Samples were analyzed for PAHs. Data validation was performed on two ECCS reports received for the investigations in accordance with the evaluation program described in Section F.3. The validation included a comprehensive review of the laboratory analysis reports for completeness, QC results, sample handling, hold time compliance,

accuracy, precision, and reported detection limits. A summary of the usability of the data generated by NLS is provided in Sections F.5.1 through F.5.12.

F.5.1 Conformance to the Sample Collection Procedures

Sample collection was conducted by URS and procedures were in accordance with collection protocols outlined in the project QAPP (URS, 2005b) Addendum No. 2. Samples were collected in one or two pre-cleaned glass containers (250-mL) for PAH and percent solids analysis. No deviations were noted in the collection practices. Aqueous samples were not required to be submitted to the mobile laboratory.

F.5.2 Conformance to Analytical Methodologies

Analyses were performed according to current versions of the mobile laboratory's SOP provided in the project QAPP (URS, 2005b), Addendum No. 2. The following parameter groups and test methods were used:

- PAHs by SW846 Method 8270C (modified)
- Percent Solids by Method 160.3 (modified)

The target chemical list is in Section 5.14 of the project QAPP (URS, 2005b) for the reconnaissance survey, but is shown here for clarity:

Acenaphthene	Fluoranthene
Acenaphthylene	Fluorene
Anthracene	Indeno(1,2,3-c,d)pyrene
Benzo(a)anthracene	1-Methylnaphthalene
Benzo(a)pyrene	2-Methylnaphthalene
Benzo(e)pyrene	Naphthalene
Benzo(k)fluoranthene	Phenanthrene
Benzo(ghi)perylene	Benzo(b)fluoranthene
Chrysene	Pyrene
Dibenzo(a,h)anthracene	

ECCS modified SW846 testing methodology for the PAH analysis to create a Performance-Based Measurement System (PBMS) method capable of meeting DQLs necessary to satisfy the DQOs for the investigation. The modified procedure, which provided quantitative results for the target list of chemicals, included minor changes to the extraction and analysis methods specified by SW846 for the parameter group. The specific differences are described as follows:

- Extraction - Five grams of soil is transferred to a tared glass 20-ml scintillation vial and 5-8 grams of anhydrous sodium sulfate is added. The soil is thoroughly mixed with the sodium sulfate and allowed to dry until the mixture pours like salt. 40 ul of a 2500(base/neutral) ug/ml surrogate standard solution is added to the dried soil. Ten mls of 90/10, dichloromethane/acetone solvent is added to the extraction vial. The vial is shaken for 2 minutes, allowed to sit for 10 minutes, shaken again for 2 minutes, and allowed to settle. 20 ul of a 1000 ug/ml internal standard solution is added to a 2 ml injection vial with a micro-syringe. 0.8 ml of the soil extract is transferred to the GC vial with an Eppendorf pipette and the sample is loaded onto an auto-sampler for GC/MSD analysis.

- GC/MSD Analysis - The analytical system consists of a Hewlett-Packard 5890 gas chromatograph with a 30m x 0.25mm, DB-5ms (or equivalent) capillary column interfaced to a Hewlett-Packard 5972 MSD with a Leap CTC A200S auto-sampler. One microliter of standard or extract is injected into the GC/MSD operated in the split-less mode. A Hewlett-Packard Enviroquant chromatography workstation is used for data acquisition and interpretation. Identification of target compounds is done by matching relative retention times and mass spectra of peaks found in samples to those found in a calibration standard using the internal standards as time reference peaks. The internal standards are 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, chrysene-d12, and perylene-d12. The surrogate standard reported was p-terphenyl-d14 for base-neutral compounds. Quantification is performed by the internal standard technique using an eight point standard curve generated from 0.25, 0.5, 1.0, 2.0, 3.0, 5.0, 10 and 25 ng/ml standards. These levels equate to 0.50, 1.0, 2.0, 4.0, 6.0, 10, 20 and 50 mg/kg for samples.

The PBMS approach was designed for this investigation to provide results with quicker turnaround time (within 24 hours of sampling) for rapid assessment of sediment conditions. This rapid analysis program allowed a thorough characterization of the type and level of potential impact in the Bay to assess the need for conducting further investigations for fish and sediment. Specific QC practices used for this method are listed in Section F.5.5.

F.5.3 Sample Handling and Chain-of-Custody

Sample handling and COC procedures followed the document procedures in the project QAPP (URS, 2005b). The field team chilled the samples on ice until delivery to the mobile laboratory. The samples were logged in to the laboratory using the mobile laboratory COC form. Copies of the completed forms are provided in Appendix E. The field and mobile laboratory analyst signed and dated these forms to affirm custody transfer. The trailer was secured at all times to maintain samples custody.

F.5.4 Adherence to Sample Holding Times

Holding times for all the project samples and analyses met the requirements of the analytical method (i.e., extraction within 14 days and analysis within 40 days). The samples for PAHs were delivered to the mobile laboratory within two to four hours of sample collection, then samples were either immediately extracted or were placed in a laboratory refrigerator (at 4°C±2°C) until sample preparation could take place. No qualification of mobile laboratory data was necessary on the basis of holding times. All samples were analyzed within 24 hours of sample collection.

F.5.5 Results of Field and Laboratory QC Samples

QC samples provide a measure of the accuracy and precision of the sample data. For this program, accuracy was measured using the results of spiked samples and blanks; and precision was measured by laboratory and field duplicate sample results. Spiked sample results are expressed as recoveries, which demonstrate the relationship between the amount spiked into the sample and the amount measured or "recovered." Spiked samples included MS/MSDs and/or LCSs, which were run at a frequency of one per batch for the PAH analyses.

Laboratory Control Samples and Matrix Spike/Matrix Spike Duplicates

The laboratory performed LCS analyses for each day of testing. The LCS samples for PAH analysis were spiked with most target chemicals with the exception of 1-methylnaphthalene, 2-methylnaphthalene, and benzo(e)pyrene. The percent recoveries for each LCS sample were within interim laboratory control limits of 75-125 percent. No qualification of results was required for the LCS evaluations. (Note: No

qualification of data was performed for the three unspiked chemicals [of a total of 19] since initial and continuing calibration standards met laboratory criteria.)

The laboratory performed MS/MSD analyses on a project sample for each day of testing. The project samples for PAHs were spiked similarly to the LCS and included 16 out of 19 target chemicals. The percent recoveries for each spiked sample were within interim laboratory control limits of 75 to 125 percent. No qualification of results was required for the MS/MSD evaluations.

Laboratory Method Blanks

Blank samples provide a measure of accuracy by monitoring chemicals potentially introduced in the field or in the laboratory. Laboratory blanks were analyzed daily by the mobile laboratory per SOP requirements. No equipment blanks, field blanks, or trip blanks were required to be submitted to the mobile laboratory for this investigation. Laboratory blanks for the organic analyses showed no contamination (all results were less than the reporting limit of 0.2 mg/Kg established for the project).

Duplicate Samples

No field duplicate samples were collected for the reconnaissance sampling event due to an oversight by field sampling personnel. Duplicates were required to be collected per the project QAPP (URS, 2005b). Field duplicates measure the variability associated with both the sampling and analytical processes. The evaluation of precision or variability of the sample results for the matrix could not be completely assessed. However, laboratory duplicates in the form of MS/MSD analyses were performed. MS/MSD analyses were performed at a frequency of at least one set of analyses per day. Agreement between the two sample results, expressed as relative percent difference (RPD), met the criteria (less than 30 percent) established by the QAPP (URS, 2005b). It should be noted that the mobile laboratory did not calculate or report RPD values for the MS/MSD analyses. The RPDs were generated and reviewed during the data validation process. The DVR for the mobile laboratory is presented in Appendix G.

F.5.6 Instrument Calibration and Tuning

Initial and continuing calibrations were reviewed during the data validation. In general, calibrations were performed at the designated frequency and met the acceptance limits. All PAH calibration results meet initial or continuing calibration criteria.

Instrument tunes for the PAH analyses also met the specified criteria in all cases. No data were qualified on this basis. The tuning process for mass selective detectors was performed at the time of project start-up and once a day (rather than at 12-hour intervals) as a check for the mass ratio criteria. The primary reason for mass selective detector tuning is to improve comparability of sample-generated mass spectra to computer library spectra created by the NIST when the analysis must identify “non-target compounds” commonly called TICs. This is not mandated when the objective of the analysis is a target chemical analysis that obtains an actual real-time mass spectra library built from initial calibration standards.

F.5.7 Analytical Performance Checks

Performance checks such as surrogate recovery and internal standard performance were also reviewed during data validation of the PAH analyses. Surrogate recoveries were within mobile laboratory interim control limits for all PAH samples.

Internal standard performance, which is used to assess the condition of the analytical instrumentation for the PAH analyses, was evaluated during data validation process. Internal standard values were not outside of laboratory criteria for the data set.

F.5.8 Analytical Sensitivity of Results

In general, the quantitation limits reported conformed to the target quantitation limits required by the project QAPP (URS, 2005b). In addition, ECCS provided method detection limits (MDLs) for all PAH compounds. All MDLs were less than the selected reporting limit indicating that the PBMS method was sensitive enough to measure the compound in a clean matrix. No analytical dilutions were required for the samples for either the May or June sampling events. No samples exceeded the calibration range of the instrument.

F.5.9 Representativeness, Completeness, and Comparability of Results

As previously stated (Section F.4.9), representativeness expresses the degree to which data accurately and precisely represents a parameter variation at a sampling point or an environmental condition and is a qualitative parameter dependent upon sampling program and proper laboratory protocol. The sampling network for the sediment reconnaissance survey was designed to provide data representative of sediment samples collected. Representativeness was satisfied by ensuring that the QAPP (Addendum 2) was followed, proper sampling techniques were used, approved analytical procedure were followed, and holding times of the samples are not exceeded in the laboratory. The majority of these factors were addressed/reviewed during the data validation process. Evidence of proper sampling techniques was obtained by discussions with the field crew and validation of results. The precision associated with MS/MSD sample analysis is discussed in Section F.5.5. In general, field sampling was conducted appropriately and field/laboratory precision was good, indicating that the results for the sediments collected were representative of site conditions.

Also previously discussed, data completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount of data expected to be obtained under normal conditions. (See calculation in Section 4.2.4 of the project QAPP.) Valid data is defined as data that are considered usable (i.e., non-rejected results) after the validation process. The amount of expected data refers to the number of measurements planned. The completeness goal (specified in the project QAPP) required that each chemical have a completeness value greater than 95 percent. This goal was obtained for the ECCS data sets for all chemicals measured in the sediment matrices. There were no rejected data; therefore, all data were valid and the completeness was 100 percent.

Data comparability expresses the confidence with which one data set can be compared to another. Comparability is dependent upon the proper design of the sampling program and that proper sampling techniques were used. Comparability was assessed for the mobile laboratory data set by submitting split samples to a second laboratory performing the conventional test method for PAH analysis (e.g., SW846 Method 8270C). It was not assessed by comparing ECCS sediment results to historical sediment data. Although historical sediment data are available, the goal of the reconnaissance survey was to finalize sample stations to assist in the implementation of a sediment triad study to evaluate sediment toxicity and benthic community structure. A rapid assessment using a modified reference test method helped select appropriate stations to meet schedule requirements.

A comparison was performed of split sample results from ECCS and NLS. This comparison evaluated the accuracy of ECCS results. This comparison indicates that the mobile laboratory results provided data of sufficient quality to screen the sediments for potential PAH impact. The mobile laboratory results for PAHs, therefore, were found to be usable for decision-making purposes and met DQOs. Specific details of the split sample evaluation are provided in Section F.5.11.

F.5.10 Laboratory Qualifiers

The mobile laboratory data did not use or apply flags for the reporting of sediment data. Flags were not required since calibration, surrogate, LCS and MS/MSD results were within laboratory criteria. Flags for non-detect results (e.g., “U”) were not used. Instead, the laboratory reported non-detect results with a dashed line (“—”). Also, revisions to results were not needed during the data validation process; therefore, no additional flags were applied by the validation group.

F.5.11 Evaluation of Split-Sample Results (ECCS vs. NLS)

Three sediment samples were split between the mobile laboratory and NLS to assess the accuracy of the PBMS method used for PAH analysis. These sediments include 9A, 13A, and 15C. (Complete sample identifications are shown below.) The on-site laboratory’s performance was acceptable for the total PAH analyses since results were similar. There was an overall positive bias for the ECCS results which was acceptable for the reconnaissance sampling since it reflects an over-estimation of the concentrations of target chemicals and is a conservative approach to determining the presence or absence of impact. It should be noted that although the ECCS result for sample “13A” was a non-detect result, the discrepancy is not significant because the mobile laboratory total PAH reporting limit was higher than the total concentration reported by NLS. The project DQOs did not require ECCS to achieve a DQL at the same level as NLS; hence the higher “total” reporting limit. The results of the split sample analyses are as follows (units in mg/Kg dry-weight as “total PAHs”):

Sample ID	ECCS	NLS
NSP-SE-1-13A-0505_20050512_(0-0_5)_NM	2290 U	957
NSP-SE-1-15C-0505_20050512_(0-0_5)_NM	84374	51900
<u>NSP-SE-1-9A-0505_20050510_(0-0_5)_NM</u>	<u>7928</u>	<u>4140</u>

*U=Non-detect result (at the calculated reporting limit for a “total” analysis).

ECCS and NLS also reported results for individual PAH compounds. The comparability of the results for the individual concentrations of PAHs are shown in Table F-3.

F.5.12 Summary of ECCS Laboratory Performance

All analytical data were valid and could be used for decision-making purposes in accordance with the DQOs outlined in the RI/FS Work Plan (URS, 2005a). No data were qualified during the validation process since there were no reported or identified non-conformances of sampling handling or method QC criteria. Although one data point exhibited a negative bias, the overall PAH data set was deemed usable based on the comparability data obtained from split-sample analyses and generated specifically for this investigation.

F.6 SEVERN TRENT LABORATORIES, BURLINGTON, VT

STL Burlington performed fish tissue and sediment chemical analyses for the RI. Data validation was performed on all STL Burlington analytical reports in accordance with the evaluation program described in Section F.3. This included a comprehensive review of the laboratory analysis reports for completeness, QC results, sample handling, hold time compliance, accuracy, precision, and reported detection limits. A summary of the usability of the data generated by STL is provided in Sections F.6.1 through F.6.11.

F.6.1 Conformance to the Sample Collection Procedures

Sample custody for the laboratory was in accordance with the FSP (URS, 2005a) and the project QAPP (URS, 2005b). Sample collection procedures for tissue were in accordance with SOPs provided in the project QAPP (URS, 2005a), Addendum 3.

F.6.2 Conformance to Analytical Methodologies

STL Burlington performed chemical analysis on fish tissue (e.g., SVITREUM-W-1-1) and sediment samples (e.g., NSP-SE-SQT5-0605-A) for the RI. All analyses were performed according to current versions of the laboratory SOPs provided in the project QAPP Addendum No. 3 (URS, 2005b) and included the following parameter groups and test methods:

- PAHs – SW846 Method 8270C (SIM analysis)⁵
- Lipids - Lipids in Tissue⁶
- Acid Volatile Sulfide and Simultaneously Extracted Metals (AVS/SEM)⁷
- Total Organic Carbon (TOC) – Lloyd Kahn Method⁸

- Grain Size – ASTM D422-63 Standard Test Method for Particle-Size Analysis of Soils and/or ASTM E112-96(2004) Standard Test Methods for Determining Average Grain Size⁹
- Percent Solids - ASTM D2216-98. Standard Test Method for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass or USEPA CLP OLM04.2/4.3¹⁰.

No deviations were described in the laboratory analysis reports or in the DVRs for the aforementioned methods. No validation was performed or required for the grain size methodology for this project.

F.6.3 Sample Handling and Chain-of-Custody

Sample handling and COC procedures followed the document procedures in the project QAPP (URS, 2005b). The field team placed all samples on ice in a cooler until packaging for shipment to STL Burlington. Containers were checked for adequate labeling, wrapped to prevent breakage, and then packed on ice in a cooler for shipment. Shipment was conducted using commercial couriers. A COC form was completed for each cooler. Copies of the completed forms (after receipt at the laboratory) are provided in Appendix E. Once received, STL Burlington assigned a unique laboratory identification

⁵ Additionally, the STL Burlington used the *SOP for the Determination of Organics in Fish*. SOP IK: ASBP100, Mod1.0 October 1990; ASB E100, Mod1.0, October 1990, USEPA Region 4 Science and Ecosystem Support Division, Analytical Support Branch.

²*Determination of Percent Lipid in Tissue*. Geochemical and Environmental Research Group, Texas A&M University College Station, TX. NOAA Technical Memorandum NOS ORCA 130 National Status and Trends Program for Marine Environmental Quality Sampling and Analytical Methods of the National Status and Trends Program Mussel Watch Project: 1993-1996 Update

⁷*Draft Analytical Method for Determination of Acid Volatile Sulfide in Sediment*. (H.E. Allen et al., EPA-821-R-91-100, August 1991).

⁸ *Methods of Soil Analysis*, Part 2. Chemical Methods. Science Society of America Book Series Number 5. American Society of Agronomy, Madison, WI. 1982.

⁹ *Annual Book of ASTM Standards, Section 4 Construction*. Volume 4.08 Soil and Rock (I).

¹⁰ *Multi-Media, Multi-Concentration SOW for Organic Analytical Service*, USEPA CLP OLM04.2/4.3.

number to each sample. Each sample is also associated with a laboratory SDG. The SDG is the means by which the laboratory identifies and groups samples for reporting purposes.

Several deviations with respect to sample handling and/or custody were noted in the DVRs. These are as follows (noting SDG ID and issue):

- SDG 108059: The laboratory performed AVS/SEM analyses on sample NSP-SE-SQT10-0605-B rather than on sample NSP-SE-SQT10-0605-D, which was requested on the COC. The alternate sample was analyzed because field personnel did not submit a sample for AVS/SEM analysis for sample NSP-SE-SQT10-0605-D.
- SDG 108055: One duplicate (SVITREUM-F-1-DUP1) was included and analyzed by the laboratory in the report that was not listed on the COCs. Samples SVITREUM-W-1-1 and SVITREUM-W-1-3 were included in the laboratory sample ID list; however, they were not analyzed and no results were reported for these two samples.
- SDG 108066: Two field duplicates were analyzed by the laboratory but were not listed on the COC. These samples were MMACROLEPIDOTUM-F11DUP & MMACROLEPIDOTUM-F31DUP).
- SDG 108176: One duplicate (MDOLOMIEUI-W-3-DUP1) was included and analyzed by the laboratory in the report that was not listed on the COC. The laboratory noted that no FedEx shipping tag was recovered for this sample shipment. The COC lists sample NS-TA-MDOLOMIVE-W-3-7 at 1150 that was not received by the laboratory. Instead the laboratory received sample NS-TA-MDOLOMIVE-W-1-8 at 1415. The laboratory was instructed to log the sample as NS-TA-MDOLOMIVE-W-3-7 at 1150.
- SDG 108177: Two duplicates (ANEBULOSIS-W-1-DUP1 and ANEBULOSIS-W-3-DUP1) were included and analyzed by the laboratory in the report but were not listed on the COCs. Samples SVITREUM-W-3-5 and SVITREUM-W-3-6 were included in the laboratory sample ID list; however, they were not analyzed and no results were reported for these two samples. Also, the laboratory incorrectly transcribed the sample ID as ANEBULOSUS-W-1-1 instead of ANEBULOSIS-W-1-1 throughout the report.

Greater detail can be found in the respective DVRs (Appendix G) of the RI report.

Also, to provide consistency for reporting, STL modified the sample identifications for this project as follows: the “NS-TA” and “ANEBU” prefixes were removed from the field name to facilitate the use of commercial data processing systems by the laboratory (i.e., the field ID character length was too long to use as a name).

F.6.4 Adherence to Sample Holding Times

Holding times were met for most project sample analyses with the exception of a set of sediment samples for TOC and tissue samples for PAHs. Affected results were qualified as estimated (“J” flag for positive results and “UJ” for non-detect results). Results associated with analyses performed past recommended hold times may be negatively biased since the chemical of concern may be lost during extended storage. For the AVS/SEM analyses, no results were rejected based on holding time issues. Note: Results for PAH analyses were qualified as estimated based on the hold time criteria for soils (14 days until extraction). The hold time for frozen fish tissue samples for organic analyses is actually “indefinite.” Qualification of fish tissue data based on hold times was not required.

F.6.5 Results of Field and Laboratory QC Samples

STL Burlington analyzed field and laboratory QC samples in accordance with QAPP requirements. The following paragraphs explain the general findings of the QC sample analyses they performed.

Laboratory Control Samples and Matrix Spike/Matrix Spike Duplicates

The laboratory performed LCS analyses for the PAH, AVS/SEM, and TOC methods in accordance with laboratory-specific SOPs. Percent recoveries and statistical control limits were reported for each LCS spiked compound. Percent recoveries results outside of statistical control limits were flagged by the laboratory and further qualified in the DVR as estimated (“J” flag for positive results and “UJ” for non-detect results), but in general, accuracy was acceptable for all analytical groups.

STL Burlington performed MS/MSD analyses on multiple project samples for PAH, AVS/SEM, and TOC analyses. MS/MSD analyses are not applicable to the lipids analysis. The project samples were spiked with the target chemical list (e.g., all PAH compounds) and the percent recoveries reported for each. Similar to the LCS analyses, percent recoveries and statistical control limits were reported for each MS/MSD compound. Percent recoveries outside of statistical control limits were noted by the laboratory and further qualified in the DVR to show the impact on the analytical results. It should be noted that if the spike concentration was significantly (four times) less than the project sample concentration, the laboratory would not calculate the percent recovery. Qualification of data was not performed in these cases. The DVRs identified acceptable recoveries for most parameter groups and matrices. For the tissue analyses, only a few PAHs (e.g., 2,6-methylnaphthalene in SDG 108177) and SEM results were qualified based on MS/MSD recoveries. For the sediment analyses, only one result (benzo(g,h,i)perylene) was rejected due to MS/MSD recoveries less than 10 percent (NS-TA-MMACROLEPIDOTUM-F-3-8 in SDG 108066). Rejected results are not usable for quantitative purposes per the DQOs established for this project. In general, the accuracy of all analytical methods performed by STL Burlington was acceptable for all analytical groups.

Method Blanks, Equipment Blanks, Field Blanks, and Trip Blanks

For the sediment and tissue samples, no field QC samples such as trip blanks, field or equipment blanks were required to be submitted to the laboratory. Laboratory method blanks were required for each analysis and were analyzed at least once per batch in accordance with laboratory SOPs.

Laboratory blanks included method blanks and “tissue equipment blanks” (used to assess any contamination in the equipment for fish tissue homogenization). The results of the laboratory blank analyses for PAHs and lipids showed no contamination. Method blanks for the SEM analyses showed the presence of nickel. TOC was also detected in one method blank for the sediments. When detected, contaminants in the blanks indicated the possibility of false positives of the chemical in the associated project samples. When method blank results were compared to action levels established for each blank contaminant, the result was qualified as non-detect (“U” flag) at the laboratory reporting limit when less than five times the action level.

Duplicate Samples

Field duplicates were collected at a frequency of approximately 1 sample for every ten project samples for sediment samples. No field duplicates were required for the fish tissue (the laboratory prepares method duplicates once received, see below). The results of the field duplicates generally demonstrated good agreement (RPD less than 50 percent) for the majority of parameters and samples. Selected chemicals were qualified as estimated (J/UJ for positive and non-detected results, respectively) based on RPDs that exceeded 50 percent for sediment samples. No results were rejected based on field duplicate results.

Laboratory duplicate samples were required to be analyzed for this project for the PAH analysis of fish tissue. The laboratory also performed MS/MSD analyses for SEM and TOC, which represents duplicates also. MS/MSD analyses were performed at a frequency of approximately one per 20 samples per parameter group. Agreement between the two sample results for fish tissue, expressed as RPD, met the criteria of the analytical methods. Agreement was also acceptable (less than the laboratory's upper control limit) for most analysis of SEM and TOC analyses. Where the RPDs were greater than the laboratory's corresponding upper control limits, the results were qualified as estimated ("J" flag for positive results and "UJ" for non-detect results). Results flagged "J" or "UJ" due to laboratory imprecision are listed in the DVRs shown in Appendix G.

F.6.6 Instrument Calibration and Tuning

Initial and continuing calibrations and instrument tuning were reviewed for applicable test methods. Instrument tuning was applicable to the PAH method only. In general, calibrations were performed at the designated frequency and met the laboratory acceptance limits/criteria specified in the laboratory SOPs. No chemicals were qualified on the bases of non-conforming initial or continuing calibration criteria. Instrument tunes for the VOC and SVOC analyses met the specified frequency in all cases. No data were qualified on this basis. It should be noted that several PAH results exceeded the concentration of the upper calibration standard and the sample had to be re-analyzed using a dilution. Where results were re-analyzed, the original result was removed from use within the database and the analysis associated with the secondary dilution was kept. The results that were removed were qualified with an "R." (Note: these data were not rejected, but simply "removed from use".)

F.6.7 Analytical Performance Checks

Performance checks such as surrogate recovery and internal standard values were also reviewed during data validation of the PAH fish tissue and sediment analyses. Performance checks for the SEM analyses included Inductively Coupled Plasma (ICP) serial dilution analyses, ICP interference check sample results, and CRDL analysis.

Generally, surrogate recoveries were within acceptable limits for the PAH analyses for both matrices. No results were rejected based on surrogate samples were qualified based on USEPA guidance (USEPA, 1999).

Internal standard performance, which is used to assess the condition of the analytical instrumentation for the PAH analyses, was acceptable for most samples for both matrices. It should be noted that the PAH-SIM analyses includes five surrogate compounds spiked at the time of extraction. Therefore, each surrogate compound must be labeled as an internal standard in the laboratories data processing software to allow utilization of the response of the surrogate compound in the calculation of each target compound. Since quantification for this procedure is based on the isotopic dilution technique, or surrogate corrected quantification, surrogates are internally identified as internal standards and appear on the Form 8. These qualifications can be ignored as the compounds were fortified at extraction rather than immediately prior to analysis leaving only two "true" internal standards (fluorene-d10 and benzo(a)pyrene-d12). The true internal standard values were within laboratory criteria; no qualification of the results was required except in a few cases. If an internal standard was outside acceptable limits (e.g., SVITREUM-F-1-2 in SDG 108055), the laboratory re-analyzed the sample to determine if the original analysis was correct. Typically, the secondary analysis proved similar to the first analysis for internal standard performance, but in some cases, the secondary analysis showed internal acceptable performance. During validation, the results for chemicals quantified using the non-compliant internal standard were qualified as estimated ("J" for positive results and "UJ" nor non-detect results). When the secondary analysis was acceptable, the

original analysis was removed from use from the database. This process ensured that only results associated with acceptable performance were being used in the data evaluation. The chemicals affected by poor internal standard responses are listed in the DVRs in Appendix G.

ICP interference check samples were run at the required frequency. In a limited number of cases, positive or negative interference was noted. Sample results were qualified as estimated (“J” for positive results and “UJ” nor non-detect results) as appropriate. ICP serial dilutions were performed periodically to assess potential bias due to interferences from the sample matrix.. For selected samples, the results of the serial dilution exceeded the 10 percent acceptance limits. Positive results for associated samples were qualified as estimated (“J”).

F.6.8 Analytical Sensitivity of Results

The quantitation limits conformed to the target quantitation limits in the project QAPP (URS, 2005b), which were established using risk-based DQLs. Analytical dilutions were performed on selected samples based on the concentrations of target compounds. Where dilutions were performed to quantify elevated levels of target compounds (concentrations above the upper calibration standard), the original results (flagged “E” by the laboratory) were removed from the database and replaced with the dilution results. (Note: The original results for all other chemicals were reported from the initial analysis.) This practice ensured that only quantitative results were available in the database for risk evaluations.

Dilutions were also required on the basis of sample matrix interference. Quantitation limits for diluted samples were raised by a factor equivalent to the dilution factor (i.e., results were reported with elevated reporting limits). These results are still usable for quantitative purposes; however, the elevated reporting limits may prevent conclusive confirmation that the target chemicals of concern are not present at a specific location if the reporting limit is above the associated risk screening level.

Additionally, some results were rejected as a result of the data validation process. Rejected data are not usable for quantitative purposes, but may have some value as qualitative information to the data user to guide further evaluations of the Site and/or ecological conditions.

STL reported tissue results as a wet-weight value along with the percent solids content so that an evaluation of both wet and dry-weight results could be performed.

During the TOC analysis for SDG 108036, sample NSP-SE-SQT5-0605-D yielded area counts in two replicates that exceeded the area count of the highest calibration standard. However, as the TOC analysis is not amenable to dilution, the result from the analysis is formally presented. The analysis is not amenable to dilution because of the small size of sample used (10 mg). When the area count in the sample is above the area of the high standard, there is no opportunity to weigh a lesser amount of sample. The result was qualified as estimated (“J” for positive results).

F.6.9 Representativeness, Completeness, and Comparability of Results

Representativeness expresses the degree to which data accurately and precisely represents a characteristic of an environmental condition. Representativeness is a qualitative parameter that is dependent upon the proper design of the sampling program and proper laboratory protocol. This sampling network was designed to provide data representative of sediment and site ecological conditions. Representativeness was satisfied by ensuring that the QAPP was followed, proper sampling techniques were used, proper analytical procedure were followed, and holding times of the samples were not exceeded in the laboratory. The majority of these factors were addressed/reviewed during the data validation process. Evidence of proper sampling techniques was made by review of field logbooks, discussions with field personnel, and assessment of field duplicate results. A summary of the logbook review is found in Section F.10. The precision associated with duplicate

sample analysis is discussed in Section F.6.5. In general, documentation of proper field sampling was complete and field precision was good for the sediment matrix.

Data completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount of data expected to be obtained under normal conditions. (See calculation in Section 4.2.4 of the project QAPP [URS, 2005b].) Valid data is defined as data that are considered usable (i.e., non-rejected results) after the validation process. The amount of expected data refers to the number of measurements planned. The completeness goal required that each chemical have a completeness value greater than 95 percent. This goal was obtained for STL Burlington for all chemicals measured in the tissue and sediment matrices. (Note: Analytical results associated with sample re-analysis were not counted toward the completeness goal.) Since there were no rejected data, the amount of valid data available for use was 100 percent. All results from STL for the RI, therefore, are acceptable for completeness.

Data comparability expresses the confidence with which one data set can be compared to another; in this case historical data (1998/99) is compared to data collected during the 2004/2005 investigation. Comparability is dependent upon the proper design of the sampling program, sampling techniques and the use of appropriate laboratory methods to measure the chemical of concern in the matrix.

An in-depth verification of comparability of the fish tissue data was not conducted for this assessment because historic (pre-2004) tissue collection procedures were not reviewed and there is limited information on analytical methodology and validation. It was presumed that sampling would be consistent with USEPA guidance for collection of fish for analysis. Additionally, the analytical method used for the 1998/99 fish analyses was SW846 Method 8310. The method used in 2005 was SW846 Method 8270. Since variations in methodology exist, there is a chance that PAH results may not be comparable barring environmental changes in site conditions. For geophysical analyses, no comparability is applicable since no samples for AVS/SEM and TOC were collected prior to the 2005 investigation.

F.6.10 Laboratory Flags and Codes

For the organic results, the laboratory used several qualifiers (i.e., D, E, J, and U) that were reviewed for applicability to the project reporting requirements. The laboratory “D” qualifier (indicating the result was from a diluted analysis) was acceptable and was retained in the database in all cases. Similarly, the laboratory “U” (non-detect result) and “J” flag (estimated result between the MDL and the laboratory reporting limits) was retained in most cases unless revised during the validation process due to non-conformances of QC criteria. The laboratory “E” flag was used to indicate that a result exceeded the concentration of the upper calibration standard. For corrective action, the laboratory re-analyzed the project sample using a dilution and reported both original and diluted analyses. In general, the result flagged “E” was removed from use within the database (“R” qualified) and the diluted result was retained. This process ensured that inaccurate data was not used in the risk evaluations.

For the inorganic results (e.g., SEM), the laboratory used one qualifier (i.e., B) that was not applicable to the reporting of data for this project. The laboratory reported positive concentrations below the reporting limit but above the MDL with a “B” flag. The “B” flag for inorganic analyses may be confused with its use for organic analyses, where it is used to indicate laboratory contamination. Therefore, since the accuracy of concentrations detected below the reporting limit is questionable, the “B” flag was changed to a “J” flag to indicate an estimated value and to be consistent with the organic data in the database. Additionally, the laboratory “U” qualifier was used; it was retained in the database since its use was consistent with data presentation. In all cases, when laboratory qualifiers were adjusted to meet project requirements; this information was noted in the DVRs (Appendix G).

F.6.11 Summary of STL Burlington Laboratory Performance

Overall, the analytical data generated by STL Burlington were valid and could be used with confidence for decision-making. Those data points indicated with a "J" qualifier should be considered estimated because of non-conformance with criteria established in the analytical methods or USEPA validation protocols. Those data points qualified with an "R" are rejected and should not be used for quantitative purposes. Only a few results were rejected because of laboratory QC performance (i.e., re-analyses) but this did not impact the overall usability of the data set.

F.7 SEVERN TRENT LABORATORIES, KNOXVILLE, TN

STL Knoxville performed the soil gas and vapor intrusion analyses for the RI. Data validation was performed on all STL analytical reports in accordance with the evaluation program described in Section F.3. This included a comprehensive review of the laboratory analysis reports for completeness, QC results, sample handling, hold time compliance, accuracy, precision, and reported detection limits. A summary of the usability of the data generated by STL is provided in Sections F.7.1 through F.7.11.

F.7.1 Conformance to the Sample Collection Procedures

Sample custody for the laboratory was in accordance with the FSP (URS, 2005a) and the project QAPP (URS, 2005b). No deviations were identified with respect to sample custody for the field or laboratory. However, one sample collection deviation was identified that was associated with the initial work performed in March 2005. Vapor probe, V-4, was damaged; therefore no sample could be collected until April 18, 2005. Along with this sample, an outdoor one-hour upwind sample and an indoor 24-hour background air sample were required to be collected since the original samples were not obtained due to field procedural error (i.e., the V-10 vapor probe was sampled using a 24-hour regulator instead of obtaining an instantaneous grab). The re-sampling has the potential to impact the data usability since the cluster of samples was not collected during the same timeframe, thus affecting comparability. A discussion of data usability for the air analyses is presented in the Section F.7.9 and the RI Report (Section 4.5).

F.7.2 Conformance to Analytical Methodologies

Analyses were performed according to current versions of the laboratory SOP provided in the project QAPP (URS, 2005b) and included the following parameter group and test method:

- VOCs – Compendium Methods TO-14 and TO-15, Determination of Volatile Organic Compounds (VOCs), in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS). USEPA Office of Research and Development, Cincinnati, OH. January 1999.

No deviations were described in the laboratory analysis reports or in the DVRs for the aforementioned methods.

E.7.3 Sample Handling and Chain-of-Custody

Sample handling and COC procedures followed the document procedures in the project QAPP (URS, 2005b). All samples were collected in Summa canisters and shipped to STL Knoxville for analysis. Summa canisters were shipped from the laboratory under negative pressure. Shipment was conducted using commercial couriers. A COC form was completed for each cooler. Copies of the completed forms (after receipt at the laboratory) are provided in Appendix E. Once received, STL assigned a unique

identification number to each sample and an SDG work order. The SDG is the means by which the laboratory identifies and groups samples for reporting purposes.

F.7.4 Adherence to Sample Holding Times

Holding times were met for all project sample analyses. No results were qualified based on holding time issues.

F.7.5 Results of Field and Laboratory QC Samples

STL analyzed field and laboratory QC samples in accordance with QAPP requirements. The following paragraphs explain the general findings of the QC sample analyses they performed.

Laboratory Control Samples and Matrix Spike/Matrix Spike Duplicates

The laboratory performed LCS analyses for the VOC method in accordance with the laboratory-specific SOP. Percent recoveries and statistical control limits were reported for each LCS spike compound. Percent recoveries results outside of statistical control limits were flagged by the laboratory and further qualified in the DVR as estimated (“J” flag for positive results and “UJ” for non-detect results), but in general, accuracy was acceptable.

MS/MSD are not applicable to the TO15 method; therefore, none were performed.

Method Blanks, Equipment Blanks, Field Blanks, and Trip Blanks

Field QC samples such as aqueous trip blanks, equipment blanks, and field blanks are not applicable to the air analysis method. Laboratory method blanks are applicable and were analyzed by STL at the frequency specified in the laboratory’s SOP. The results of the laboratory blank analyses for VOCs, revealed no laboratory contamination. No analytical data were required to be qualified based on laboratory method blank results.

Duplicate Samples

One set of field duplicates (e.g., VP-05_20050718_(2-2)_DUP) were collected for the air matrix. The results of the field duplicates generally demonstrated good agreement (RPD less than 50 percent) for the majority of parameters with the exception of toluene. Toluene was detected at concentrations of 1.9 ug/m³ in the parent sample and 0.26 U ug/m³ (non-detect value) in the field duplicate sample. Results were qualified as estimated (“J” for the positive value and “UJ” for the non-detect value). Other positive concentrations of target chemicals (e.g., trichlorofluoromethane) showed good agreement. No results were rejected based on field duplicate results.

Laboratory duplicate samples were not required to be analyzed for this project.

F.7.6 Instrument Calibration and Tuning

Initial and continuing calibrations were reviewed for all STL analysis reports. In general, calibrations were performed at the designated frequency and met the laboratory acceptance limits/criteria specified in the SOP. A few chemicals did not meet initial or continuing calibration criteria (e.g., the percent difference for the continuing calibration for toluene in SDG H5G210136). These chemicals, in the associated samples, were qualified as estimated (“J” for positive results and “UJ” for non-detect results). Instrument tunes for the VOC and SVOC analyses met the specified frequency in all cases. No data were qualified on this basis. No data were rejected based on instrument calibration or tuning.

F.7.7 Analytical Performance Checks

Performance checks such as surrogate recovery and internal standard values were also reviewed during data validation of the VOC analyses. Generally, surrogate recoveries were within acceptable limits for most analyses. Surrogate recoveries were outside acceptance criteria (less than 10 percent) in a limited number of samples (e.g., see SDG H5G210136). As corrective action, the laboratory re-analyzed the samples and reported both sets of data. The results for the original analyses were removed from use in the database, and the second analysis was used.

Internal standard response, which is used to assess the condition of the analytical instrumentation for the VOC analyses, was acceptable for the majority of samples. If an internal standard was outside acceptable limits, the results for chemicals quantitated using the non-compliant internal standard were qualified as estimated (“J” for positive results and “UJ” for non-detect results) in the associated samples. The chemicals affected by poor internal standard responses can be found in DVRs labeled SDG H5D200132 and H5G210136 in Appendix G.

F.7.8 Analytical Sensitivity of Results

The quantitation limits conformed to the target quantitation limits in the project QAPP (URS, 2005b). No analytical results exceeded the upper calibration standard or were required to be flagged for this issue.

F.7.9 Representativeness, Completeness, and Comparability of Results

As previously stated, representativeness expresses the degree to which data accurately and precisely represent a characteristic of an environmental condition. It is a qualitative parameter that is dependent upon the proper design of the sampling program and proper laboratory protocol. This sampling network was designed to provide data representative of site conditions. Representativeness was satisfied by ensuring that the QAPP was followed, proper sampling techniques were used, proper analytical procedures were followed, and holding times of the samples were not exceeded in the laboratory. The majority of these factors were addressed/reviewed during the data validation process. Evidence of proper sampling techniques was obtained by the review of field logbooks and assessment of field duplicate results. A summary of the logbook review is found in Section F.11. The precision associated with duplicate sample analysis is discussed in Section F.7.5. In general, documentation of proper field sampling was complete and field precision was very good in most cases for the air matrix.

Data completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount of data expected to be obtained under normal conditions. (See calculation in Section 4.2.4 of the project QAPP.) Valid data is defined as data that are considered usable (i.e., non-rejected results) after the validation process. The amount of expected data refers to the number of measurements planned. The completeness goal (specified in the project QAPP) required that each chemical have a completeness value greater than 95 percent. This goal was obtained for the STL data sets for most chemicals measured in the air matrix. (Note: Analytical results associated with sample re-analysis were not counted toward the completeness goal.) There were no rejected data for VOCs; therefore, the amount of valid data available for use was 100 percent. The results for all chemicals of concern for the RI, therefore, are acceptable for completeness.

Data comparability expresses the confidence with which one data set can be compared to another. Comparability is dependent upon the proper design of the sampling program and that proper sampling techniques were used. Since there are no historic data for soil gas and vapor intrusion, comparability is not required to be assessed at this time.

F.7.10 Laboratory Flags and Codes

For the air analyses, the laboratory used only a “U” flag to indicate a non-detect. This flag was consistent with project requirements; therefore, no revision to the database was required. Updates and adjustments to the laboratory qualifiers to meet project requirements are documented on the printouts of the analysis summary sheets found in each of the DVRs in Appendix G.

F.7.11 Summary of STL Knoxville Laboratory Performance

Overall, the analytical data generated by STL Knoxville were valid and could be used with confidence for decision-making. Those data points indicated with a "J" qualifier should be considered estimated because of non-conformance with criteria established in the analytical methods or USEPA validation protocols. Those data points qualified with an “R” are rejected and should not be used for quantitative purposes. Only a few results were rejected because of laboratory QC performance (i.e., re-analyses) but this did not impact the overall usability of the data set since re-analysis results were available for use.

F.8 PACE ANALYTICAL LABORATORIES (PACE), MINNEAPOLIS, MN AND ENCHEM, GREEN BAY, WI

Pace performed PAH analyses of aqueous samples (e.g., JDUP-8HR) and sediment samples (e.g., SQT 7) collected during toxicity testing conducted by LSRI. Pace was a subcontract laboratory to LSRI for this project. Pace also performed tissue analyses (e.g., SQT9-4) for the RI through their network laboratory, EnChem, Green Bay, Wisconsin¹¹. Data validation was performed on all analytical reports in accordance with the evaluation program described in Section F.3. Validation included a comprehensive review of the laboratory analysis reports for completeness, QC results, sample handling, hold time compliance, accuracy, precision, and reported detection limits. A summary of the usability of the data generated by Pace and EnChem is provided in Sections F.8.1 through F.8.11.

F.8.1 Conformance to the Sample Collection Procedures

Sample custody for the laboratory was in accordance with the FSP (URS, 2005a) and the project QAPP (URS, 2005b). No deviations with respect to sample collection and/or custody were noted in the DVRs. Sample collection procedures for tissue were in accordance with SOPs provided in the project QAPP (URS, 2005a), Addendum 3. An evaluation was not performed of the sample collection by LSRI for the aqueous samples from the toxicity testing.

F.8.2 Conformance to Analytical Methodologies

Analyses were performed according to current versions of the laboratory SOPs provided in the project QAPP (URS, 2005b) and included the following parameter groups and test methods:

- PAHs – SW846 Method 8270C (SIM mode for aqueous); Method 3510C (aqueous extraction); and Method 3550B (solid extraction)
- Percent Solids – Multi-Media, Multi-Concentration SOW for Organic Analytical Service, USEPA CLP OLM04.2/4.3.

No deviations were described in the laboratory analysis reports or in the DVRs for these methods.

¹¹ S-F Laboratories, Inc. was used for sulfide analyses for the bioassays. This laboratory was not included in the DAR evaluation.

F.8.3 Sample Handling and Chain-of-Custody

Sample handling and COC procedures followed the document procedures in the project QAPP (URS, 2005b). A COC form was completed for each sample shipment (e.g., LSRI to Pace). Copies of the completed forms, after receipt at the laboratory, are provided in the laboratory analysis reports in Appendix E. Once received, Pace and EnChem assigned a unique laboratory identification number to each sample. Each sample is also associated with a laboratory SDG. The SDG is the means by which the laboratory identifies and groups samples for reporting purposes. The SDGs for the Pace analysis reports are 860987, 862259, 862425, and 862803; the SDGs for the EnChem analysis reports are 864677A and 864677B.

F.8.4 Adherence to Sample Holding Times

Holding times were met for most project sample analyses for both laboratories with the exception of one set of re-analyses for SVOCs for the soil samples (e.g., SQT 10) due to surrogate recoveries outside of laboratory control limits. The re-extraction was performed past the hold time of 14 days. Since the initial analysis results were acceptable regardless of the surrogate exceedances, these results were retained in the database and the re-analysis results were qualified as estimated and removed from use. For the tissue analyses, no results were rejected based on holding time issues. All results, therefore, are usable for quantitative purposes per the DQOs established for this project.

F.6.5 Results of Field and Laboratory QC Samples

Pace/EnChem analyzed field and laboratory QC samples in accordance with QAPP requirements. The following paragraphs explain the general findings of the QC sample analyses they performed.

Laboratory Control Samples and Matrix Spike/Matrix Spike Duplicates

The laboratory performed LCS analyses for the PAH analyses in accordance with laboratory-specific SOPs. Percent recoveries and statistical control limits were reported for each LCS spike compound. Percent recovery results outside of statistical control limits were flagged by the laboratory and further qualified in the DVR as estimated (“J” flag for positive results and “UJ” for non-detect results). In general, accuracy was acceptable for all analytical groups.

Pace/EnChem performed MS/MSD analyses on multiple project samples for PAHs. The project samples were spiked with the PAH chemical list and percent recoveries reported for each. Percent recoveries outside of statistical control limits were flagged by the laboratory and further qualified in the DVR to show the impact on the analytical results (e.g., J-DAY 14 for Pace SDG862803). In a few cases, insufficient volume (e.g., Pace SDG 862425) was provided to perform MS/MSD analyses. In this situation, the laboratory analyzed an LCS and an LCS duplicate to assess precision. In general, the accuracy of all analytical methods performed by NLS was acceptable for all analytical groups.

Method Blanks, Equipment Blanks, Field Blanks, and Trip Blanks

Field QC samples such as trip blanks, equipment blanks, and field blanks were not applicable to the toxicity testing effluents and sediments submitted for analysis. Laboratory QC samples were applicable, and included method blanks and instrument blanks where required. These QC samples were analyzed at least once per batch in accordance with laboratory SOPs. The results of the blank analyses revealed very little laboratory contamination. If a contaminant was detected in the method blank, sample results were compared to action levels established for each blank contaminant (see previous discussions) and the data qualified as non-detect (“U” flag) when less than the action level.

Duplicate Samples

Field duplicates were collected at a frequency of approximately one sample for every ten project samples for both solid and liquid matrices. The results of the field duplicates generally demonstrated good agreement (RPD less than 30 percent for aqueous samples and less than 50 percent for soil/tissue samples) Selected chemicals were qualified as estimated (J/UJ for positive and non-detected results, respectively). No results were rejected based on field duplicate results.

Laboratory duplicate and MS/MSD samples were analyzed for tissue and aqueous samples, respectively. The laboratory duplicates were prepared by EnChem and showed good precision (RPD less than 50 percent). The precision associated with the MS/MSD analyses was acceptable in most cases. Where the RPDs were greater than the laboratory's corresponding upper control limits, the results were qualified as estimated ("J" flag for positive results and "UJ" for non-detect results).

F.8.6 Instrument Calibration and Tuning

Initial and continuing calibrations were reviewed for all Pace/EnChem analysis reports. In general, calibrations were performed at the designated frequency and met the laboratory acceptance limits/criteria specified in the SOP. A few chemicals (e.g., benzo(a)pyrene in SDG 862803) did not meet initial or continuing calibration criteria. These chemicals, in the associated samples, were qualified as estimated ("J" for positive results and "UJ" for non-detect results). Instrument tunes for the PAH analyses met the specified frequency in all cases. No data were qualified on this basis.

F.8.7 Analytical Performance Checks

Performance checks such as surrogate recovery and internal standard responses were reviewed during data validation of the PAH analyses. Surrogate recoveries were within acceptable limits for most PAH analyses. Surrogate recoveries were outside acceptance criteria (less than 10 percent) in a limited number of samples (e.g, sample SQT10-4 in SDG 864677). The results for these samples were qualified based on USEPA guidance (USEPA, 1999). The chemicals affected by surrogate recoveries outside of laboratory limits are listed in the DVRs in Appendix G.

Internal standard response, which is used to assess the condition of the analytical instrumentation for the PAH analyses, was acceptable in all tissue and sediment samples. No qualification of data was required.

F.8.8 Analytical Sensitivity of Results

The quantitation limits conformed to the target quantitation limits in the project QAPP (URS, 2005b), which were established using risk-based DQLs. Analytical dilutions were performed on selected samples due to concentrations of target compounds. Where re-analyses were performed to quantify elevated target compound concentrations (e.g., concentrations above the upper calibration standard), the original results were removed from the database and replaced with the re-analysis results. (Note: The original results for all other chemicals were reported from the initial analysis.) This practice ensured that only quantitative results were available in the database for risk evaluations.

F.8.9 Representativeness, Completeness, and Comparability of Results

Representativeness expresses the degree to which data accurately and precisely represents an environmental condition. It is a qualitative parameter that is dependent upon the proper design of the sampling program and proper laboratory protocol. The sampling program ensured that PAH data were representative for the sediments and fish tissue tested. Representativeness was also satisfied by ensuring that the QAPP was

followed, proper sampling techniques were used, proper analytical procedure were followed, and holding times of the samples were not exceeded in the laboratory. The majority of these factors were addressed/reviewed during the data validation process. The precision associated with duplicate sample analysis is discussed in Section F.8.5.

Data completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount of data expected to be obtained under normal conditions. (See calculation in Section 4.2.4 of the project QAPP.) Valid data is defined as data that are considered usable (i.e., non-rejected results) after the validation process. The amount of expected data refers to the number of measurements planned. The completeness goal (specified in the project QAPP) required that each chemical have a completeness value greater than 95 percent. This goal was obtained for the Pace/EnChem data sets for the PAH results for tissue. (Note: All non-detect PAH results except naphthalene were rejected for sample SQT10-4, but this still allowed completeness to be greater than 95 percent). A completeness goal was not established for the toxicity testing effluent waters. However, since no analytical results were rejected during the validation, the completeness value would be 100 percent.

An in-depth verification of comparability of the fish tissue data was not conducted for this assessment because historic tissue collection procedures (pre-2004) were not reviewed and there is limited information on analytical methodology and validation. It was presumed that sampling would be consistent with reference guidance for collection of fish tissue for analysis, as well as analytical methodology; however it was identified that PAHs were measured in fish tissue using SW846 Method 8310 using Wisconsin Industrial Hygiene. The method used by EnChem and STL Burlington for the 2004/2005 tissue was SW846 Method 8270. Since variations in methodology exist, there is a chance that PAH results may not be comparable barring environmental changes in site conditions. For the toxicity testing effluents, no comparability is applicable since bioassay effluent samples were not collected prior to the 2005 investigation.

F.8.10 Laboratory Flags and Codes

PACE and EnChem used various flags for the tissue analyses that were not consistent with project data reporting requirements. During the data validation process and database management, these flags were updated to reflect the requirements in the project QAPP (URS, 2005b). A “<” flag was used by the laboratory for non-detect results. The “<” qualifiers were replaced with a “U” for non-detect results. A “Q” flag was used by the laboratory to indicate results that were greater than the MDL but below the reporting limits (RL). For consistency, these were changed to a “J” to indicate estimated data. A “B” qualifier was used to indicate method blank contamination. The “B” qualifier was removed if the sample concentration was greater than five times the blank concentration, or it was revised to a “U” if sample concentration was less than five times the blank concentration. A “&” qualifier was used to indicate LCS percent recovery outside control limits. A “*” qualifier was used when the LCS relative percent difference (RPD) exceeded acceptance criteria. The “&” and “*” qualifiers were replaced with a “J” for positive results or “UJ” for non-detect results where applicable. Updates and adjustments to the laboratory qualifiers to meet project requirements are documented on the printouts of the analysis summary sheets found in each of the DVRs in Appendix G.

F.8.11 Summary of Pace/EnChem Laboratory Performance

Overall, the analytical data generated by Pace/EnChem were valid and could be used with confidence for decision-making. Those data points indicated with a "J" qualifier should be considered estimated because of non-conformance with criteria established in the analytical methods or USEPA validation protocols. Those data points qualified with an “R” are rejected and should not be used for quantitative purposes.

Only a few results were rejected because of laboratory QC performance issues (i.e., surrogate recovery) but this did not impact the overall usability of the data set.

F.9 TELEDYNE BROWN ENGINEERING (TBE), KNOXVILLE, TN

TBE performed the radio-chemistry analyses for ^{210}Pb and ^{136}Cs age-dating of sediment cores for the RI. Data validation was performed on all TBE analytical reports in accordance with the evaluation program described in Section F.3. This included a comprehensive review of the laboratory analysis reports for completeness, QC results, sample handling, hold time compliance, accuracy, precision, and reported detection limits. A summary of the usability of the data generated by TBE is provided in Sections F.9.1 through F.9.11.

F.9.1 Conformance to the Sample Collection Procedures

Sample custody and collection procedures were in accordance with the FSP (URS, 2005a) and the project QAPP (URS, 2005b), Addendum No. 3. No deviations with respect to sample collection and/or custody were noted in the analysis reports or DVRs (Appendix G).

F.9.2 Conformance to Analytical Methodologies

Analyses were performed according to current versions of the laboratory SOPs provided in the project QAPP Addendum No. 3 (URS, 2005b), and included the following parameter groups and test methods:

- PB-210 and CS-137 – TBE SOP. “Gamma-Emitting Radioisotope Analysis, No. TBE 2007” and *Prescribed Procedures for Measurement of Radioactivity in Drinking Water*, EPA 600/4-80-032, August 1980
- Percent Solids – EPA Method 160.3 (modified)

No deviations were described in the laboratory analysis reports or in the DVRs for the aforementioned methods. The data validation was conducted using the following USEPA guidance: “Routine Validation of Gamma Spectroscopy Data,” ER-SOP15.06, 7/20/00. Results were reported in dry-weight per QAPP requirements.

F.9.3 Sample Handling and Chain-of-Custody

Sample handling and COC procedures followed the document procedures in the project QAPP Addendum No. 3 (URS, 2005b). The field team placed all sample on ice in a cooler until packaging for shipment to TBE. Containers were checked for adequate labeling, wrapped to prevent breakage, and then packed on ice in a cooler for shipment. Shipment was conducted using commercial couriers. A COC form was completed for each cooler. Copies of the completed forms (after receipt at TBE) are provided in Appendix E. Once received, TBE assigned a unique laboratory identification number to each sample (i.e., SDG). The SDGs for TBE analysis reports are L25881, L25935, and L25936.

F.9.4 Adherence to Sample Holding Times

Holding times were met for all project sample analyses. Holding time for gamma spectroscopy is 6 months. No data were required to be qualified due to missed holding times.

F.9.5 Results of Field and Laboratory QC Samples

TBE analyzed laboratory QC samples in accordance with QAPP requirements. However, field QC samples were not collected (or designated on the COC). The following paragraphs explain the general findings from review of QC sample analyses.

Laboratory Control Samples and Matrix Spike/Matrix Spike Duplicates

The laboratory performed LCS analyses for all methods in accordance with its laboratory-specific SOP. Percent recoveries and statistical control limits were reported for each compound. No results were flagged by the laboratory based on LCS results, indicating that the accuracy of the measurements was acceptable. For MS/MSD analyses, field personnel did not designate a sample for MS/MSD analysis on the COC, nor did the laboratory select a project sample for internal MS/MSD analyses. Due to this oversight, no MS/MSD analyses were performed. Assessment of the accuracy of the spectroscopy measurement on the sediment matrix could not be determined.

Method Blanks, Equipment Blanks, Field Blanks, and Trip Blanks

No field QC samples such as equipment blanks, field blanks, etc. were required to be collected for the sediment age-dating work. Laboratory method blanks were required to be analyzed. Laboratory blanks included method blanks and calibration blanks that were analyzed at the frequency specified in the laboratory's SOPs. The results of the laboratory blank analyses showed no contamination. The presence of a radionuclide in a blank would indicate the possibility of false positives in the associated project samples.

Duplicate Samples

Field duplicates were required to be collected at a frequency of approximately one sample for every ten project samples per QAPP requirements (URS, 2005b). None were collected due to a field sampling oversight. Assessment of sampling precision could not be determined for the age-dating of the core samples.

Per project requirements MS/MSD analyses were required to be performed to assess matrix and method precision. Similar to the situation for field duplicates, no MS/MSD analyses were performed; however, laboratory duplicate analyses (analysis of two sub-samples from the sample container) were performed. (These analyses were not required for this project but were performed by TBE for internal precision information.) Per summaries in the DVRs, laboratory duplicates showed agreement between sample results, and met laboratory criteria for RPDs for the analytical method. No results were required to be qualified based on laboratory duplicate results.

F.9.6 Instrument Calibration and Tuning

Instrument performance and background checks were reviewed for all TBE analysis reports. In general, verification of activity for instruments was performed at the designated frequency and met the laboratory acceptance limits/criteria specified in the SOP. No data were qualified on this basis.

F.9.7 Analytical Performance Checks

Performance checks such as LCS recovery and method duplicates were also reviewed during data validation. LCS recoveries were acceptable for all project sample analyses and the precision of method duplicate runs were below the applicable laboratory control limits. No data were qualified on this basis.

F.9.8 Analytical Sensitivity of Results

All activity concentrations reported below the minimum detectable concentration (MDC), including negative results, were qualified as "U by the laboratory. Activity concentrations above the MDC were reported unqualified (i.e., no flag). Diluted analyses were not performed and are typically not applicable to spectroscopy analysis. No re-analyses were required for the sediment samples due to instrument performance or sample matrix issues. Results were corrected for moisture content and reported in units of

pCi/g. The sensitivity was acceptable for the analyses based on information presented in the DVRs (Appendix G).

F.9.9 Representativeness, Completeness, and Comparability of Results

Representativeness expresses the degree to which data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness is a qualitative parameter that is dependent upon the proper design of the sampling program and proper laboratory protocol. This sampling network was designed to provide data representative of site conditions. Representativeness was satisfied by ensuring that the QAPP was followed, proper sampling techniques were used, proper analytical procedures were followed, and holding times of the samples are not exceeded in the laboratory. No deviations were documented by the field personnel for sampling and proper analytical procedures were followed during analysis. Therefore, the results are expected to be representative of the Site conditions for the measurements performed.

Data completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount of data expected to be obtained under normal conditions. (See calculation in Section 4.2.4 of the project QAPP.) Valid data is defined as data that are considered usable (i.e., non-rejected results) after the validation process. The amount of expected data refers to the number of measurements planned. The completeness goal (specified in the project QAPP) required that each chemical have a completeness value greater than 95 percent. This goal was obtained for the TBE data sets for both ^{210}Pb and ^{136}Cs as measured in the sediment. No results were rejected. All results for the RI, therefore, are acceptable for completeness. The completeness was 100 percent.

Data comparability expresses the confidence with which one data set can be compared to another. Comparability is dependent upon the proper design of the sampling program and that proper sampling techniques were used. Comparability was not assessed for the TBE results since historical sediment data was not generated.

F.9.10 Laboratory Flags and Codes

The laboratory used flags (e.g., “U,” “+,” etc.) to indicate to the data user information regarding the accuracy of the reported data. A “U” flag indicated non-detect data and was consistent with project reporting requirements. A “+” flag indicated an activity concentration that exceeded the MDC and 3 sigma for peak identification for gamma measurement only. A “Yes” or “No” flag indicated the presence or absence of the peak, respectively, for the gamma measurement. The “+” and “Yes or No” flags were not present in the electronic database; therefore, no revision of flags were required during the data validation process and database management.

F.9.11 Summary of TBE Laboratory Performance

The analytical data generated by TBE were valid and could be used with confidence for decision-making. No analytical data were required to be qualified as estimated (“J” qualifier) or rejected (“R” qualified) as a result of the data validation. Laboratory QC performance (blank, standard, and LCS) analyses were acceptable for all project samples. Assessment of sampling precision could not be determined for the age-dating of the core samples since field duplicates were not collected; however, laboratory precision, measured by the analysis of laboratory duplicates, was acceptable.

F.10 FIELD AND LABORATORY AUDITS

On-site field and laboratory audits were not conducted during the RI; however, a technical system review of the RI field logbooks was performed to assess completeness and accuracy of documentation supporting

the RI investigations. The technical review was also performed to determine whether information documented in the field logbooks was compliant with the record-keeping requirements specified in the field SOPs (Numbers 100 through 300) found in the FSP (URS, 2005a). The project SOPs require recording field observations, technical data, and health & safety practices that directly affect the field task being conducted. Field log books also are intended to provide sufficient data and observations to enable reconstruction of events that occur while performing field activities. As such, all entries are to be as factual, detailed, and descriptive as appropriate so that a particular situation can be reconstructed without reliance on the author's memory. Field log books are also used to compare and check other field documents.

Eight logbooks were provided for the technical review. The following three logbooks and sections (dated entries) were evaluated: 1) Kreher Park Test Pit/Surface Soil (6/6-6/16/05); 2) Round 2 Vapor Probes/Diffusion Bag Sampling (7/18-7/19); and 3) Supplemental Test Pit (10/31-11/04).

The technical review was accomplished using a checklist. The checklist itemizes the criteria used for the review, and lists associated examples of acceptable practices or alternatively, examples of missing information and records. Generally, deficiencies were minor (i.e., those that do not impact the final evidence file for the project significantly) but several deficiencies were considered major (i.e., those that impact the final evidence file since critical data are not present that may be needed to support the findings of the investigation). An example of a minor deficiency was the lack of records to state that field equipment were being decontaminated. An example of a major deficiency was the lack of records for calibration of field meters. The completed checklist and findings are provided in Attachment F-1.

F.11 OVERALL SUMMARY OF ANALYTICAL LABORATORIES' DATA USABILITY

Nearly all analytical results are considered usable based on the QC information provided by the laboratories and the results of the data validation. The laboratories followed industry-accepted test methods and performed standard QC analyses as required by the laboratory-specific SOPs provided in the approved QAPP (URS, 2005b). Some sample results were reported with elevated reporting limits due to the presence of high concentrations of target compounds or matrix interference. These results are still usable for quantitative purposes; however, the elevated reporting limits may prevent conclusive confirmation that the target chemicals of concern are not present at a specific location if the reporting limit is above the associated risk screening level. Additionally, some results were rejected as a result of the data validation process. Rejected data are not usable for quantitative purposes, but may have some value to the data user as qualitative information to guide further evaluations of the Site and/or ecological conditions. Issues related to blank contamination for the groundwater monitoring program are considered adequately resolved but will continue to be assessed during FS work at the Site.

F.12 REFERENCES

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- URS, 2005b. Quality Assurance Project Plan RI/FS Tasks, Revision 03, Ashland/NSP Lakefront Superfund Site, Ashland, Wisconsin. Remedial Investigation/Feasibility Study (February 2005) plus Addendums 1, 2, and 3.
- URS 2005c. Remedial Investigation/Feasibility Study (RI/FS) Work Plan Revision 02 Ashland/NSP Lakefront Superfund Site, Ashland, Wisconsin. (February 2005).

- USEPA. 1983. Methods for Chemical Analysis of Water and Wastes. USEPA/600/4/79/020. U.S. Environmental Protection Agency. March 1983.
- USEPA. 1991. Draft Analytical Method for Determination of Acid Volatile Sulfide in Sediment. USEPA (Document Number 821R91100), December 1991. (This method describes procedures for the determination of acid volatile sulfide and for selected metals that solubilized during the acidification step [simultaneously extracted metals].)
- USEPA. 1999. Contract Laboratory Program National Functional Guidelines for Organic Data Review. U.S. Environmental Protection Agency. October 1999.
- USEPA. 2000. Routine Validation of Gamma Spectroscopy Data. ER-SOP15.06, July 20, 2000.
- USEPA. 2004a. Contract Laboratory Program National Functional Guidelines for Inorganic Data Review. U.S. Environmental Protection Agency. March 2004.
- USEPA. 2004b. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (Final Update IIIB). SW-846. U.S. Environmental Protection Agency. November 1986, April 1998, and November 2004.

Table F-1
Sampling Periods and Matrices Collected for the RI

Event Date	Sample Matrix	Event Description
June 2004 to March 2005	Groundwater	Quarterly groundwater sampling
March 2005	Air	Vapor probe & indoor air – winter event
April 2005	Soil	Surface & subsurface soil sampling
April 2005	Tissue	Rainbow smelt sampling
May 2005	Sediment	Sediment reconnaissance sampling
May 2005	Surface water	Physical hydraulic parameters
June 2005	Soil	Test pit, surface soil & forensic sampling
June 2005	Tissue	Various fish species sampling
June 2005	Surface water	Surface water sampling – low energy event
June 2005	Sediment	Sediment & forensic sampling
June 2005	Tissue	Benthic invertebrate sampling
July 2005	Groundwater	Passive diffusion bag sampling
July 2005	Air	Vapor probe & indoor air – summer event
September 2005	Sediment	Sediment chemistry & forensic sampling
November 2005	Soil	Test pits – forensic sampling
November 2005	Surface water	Surface water sampling – high energy event

**Table F-2
 Field QC Samples Collected Per Matrix for Each Laboratory**

Lab	Method	Matrix	Total No. of Spikes ¹	Total No. of Samples	Total No. Required	Accuracy Requirement Met? (5%) ²	Total No. of Field Duplicates	Total No. of Samples	Total No. Required	Precision Requirement Met? (10%) ³
NLS	Metals	Soil/Sed.	36	278	14	Yes	25	278	28	No
NLS	VOCs	Soil/Sed.	32	268	14	Yes	25	268	27	No
NLS	SVOCs	Soil/Sed.	31	418	21	Yes	34	418	42	No
NLS	Metals	Water	64	684	35	Yes	58	684	69	No
NLS	VOCs	Water	45	589	30	Yes	42	589	59	No
NLS	SVOCs	Water	35	522	27	Yes	41	522	53	No
ECCS	PAHs	Sediment	4	18	1	Yes	0	18	2	No ⁴
STL VT	PAHs	Tissue	8	142	8	Yes	11	142	15	No ⁵
TBE	²¹⁰ Pb and ¹³⁶ Cs	Sediment	N/A ⁷	---	N/A	N/A	0	16	2	No
STL TN	TO15	Air	N/A ⁶	33	N/A	N/A	1	33	4	No

ECCS-Environmental Chemistry Consulting Services

NLS-Northern Lake Services

PAHs-Polycyclic Aromatic Hydrocarbons

STL-Severn Trent Laboratory (Vermont and Tennessee)

SVOCs-Semi-Volatile Organic Compounds

TBE-Teledyne Brown Engineering

TO15-See Section F.7.2 for method reference.

VOCs-Volatile Organic Compounds

N/A-Not applicable

1. One spike sample was collected for OC pesticide analyses.
2. One matrix spike/matrix spike duplicate (MS/MSD) was required to be collected for every 20 project samples.
3. One field duplicate was required to be collected for every 10 project samples.
4. The laboratory performed MS/MSD analyses to assess method/matrix precision.
5. The tissue samples were not actual field duplicates, but laboratory replicates created by the laboratory after homogenization.
6. Spike analyses are not applicable to air analyses.
7. Spike analyses are not applicable to radiochemistry analyses.

Table F-3
Comparison of Split-Sample Results for PAHs
ECCS versus NLS

Analyte	Units	NSP-SE-1-13A-DUP	NSP-SE-1-13A-NM		NSP-SE-1-15C-DUP	NSP-SE-1-15C-NM		NSP-SE-1-9A-DUP	NSP-SE-1-9A-NM
	LAB:	NLS	ECCS		NLS	ECCS		NLS	ECCS
1-Methylnaphthalene	ug/kg	32 U	241 U		450	1338		32 U	634 U
2-Methylnaphthalene	ug/kg	31 U	241 U		740	754		31 U	634 U
Acenaphthene	ug/kg	32 U	241 U		710	1143		32 U	634 U
Acenaphthylene	ug/kg	31 U	241 U		2400	3673		31 U	634 U
Anthracene	ug/kg	38 U	241 U		950	2335		38 U	634 U
Benzo(a)anthracene	ug/kg	83 J	241 U		4500	7467		340	698
Benzo(a)pyrene	ug/kg	120	241 U		7300	10823		440	1142
Benzo(b)fluoranthene	ug/kg	110 J	241 U		6400	5570		500	729
Benzo(e)pyrene	ug/kg	73 J	241 U		4400	6372		310	634 U
Benzo(g,h,i)perylene	ug/kg	33 U	241 U		2300	6299		200	634
Benzo(k)fluoranthene	ug/kg	38 U	241 U		2400	6470		160	698
Chrysene	ug/kg	87 J	241 U		4700	7005		390	793
Dibenzo(a,h)anthracene	ug/kg	34 U	241 U		34 U	2554		34 U	634 U
Fluoranthene	ug/kg	100 J	241 U		3600	4962		570	1078
Fluorene	ug/kg	32 U	241 U		570	559		32 U	634 U
Indeno(1,2,3-cd)pyrene	ug/kg	31 U	241 U		1400	4840		140	634 U
Naphthalene	ug/kg	31 U	241 U		880	1435		31 U	634 U
Phenanthrene	ug/kg	63 J	241 U		1800	2554		470	951
Pyrene	ug/kg	140	241 U		6400	8221		620	1205

U=Non-detect result at the laboratory reporting limit.

J=Estimated result below the laboratory reporting limit but above the laboratory MDL.

Laboratory results are corrected for moisture content.

ECCS-Environmental Chemistry Consulting Services

NLS-Northern Lake Services

ATTACHMENT F-1
Field Logbook Documentation Review

Criteria	Yes	No	N/A	Comment (Major deficiencies are bolded)
A. Set-Up				
1. Is the log book hard bound, water-resistant with water-proof pages?	√			
2. Does the inside of the log book contain the following information: book number, project name and number, contract/project number, site name, start date, end date, person to whom the log book belongs, company name, address, and phone number	√			All log books contained some elements that identified the project; however, complete records were not present in a few cases. Sequential book numbers were not used.
3. At a minimum, do log book entries include the following information at the beginning of each day, as appropriate to the activities: <ul style="list-style-type: none"> • Date • Time • Weather • H&S meetings/notes • Field personnel present • Equipment used • Procedures to be used 		√		All log books contained some elements that identified current conditions; however, complete records were not present. First names of personnel were used frequently, but no surnames. Field sampling equipment was not specified in all cases. H&S topics not recorded in all cases.
4. Does the log book contain a description of the exclusion zone and decontamination methods employed?		√		Described infrequently. No decontamination methods were listed in any of the log books reviewed.
5. Does the log book contain a description of the personnel protection equipment being used?		√		No reference to Level D was found.
6. Does the log book contain a description of the procedures for containerization of investigation-derived wastes?		√		Only occasionally described.
B. Log Book Entries				
7. Are entries made in indelible blank or blue ink?	√			One log book entry was made in pencil.
8. Are incorrect entries crossed out with a single line and initialed and dated by the originator?			√	No incorrect entries were observed.

Criteria	Yes	No	N/A	Comment (Major deficiencies are bolded)
9. Are entries signed and dated at the bottom of the last page of the log books completed for the day?	√			Some log book entries did not contain dates; some log books were signed with initials only.
10. Are the time records in the log book made using either chronological sequence or 24-clock?	√			There was some mixing of time record formats on a given day.
11. Is a diagonal line used to mark any un-used pages or sections of a page when not used during the day?		√		A diagonal strike line is seldom used to define the end of page and/or completed daily activities.
12. If more than one person enters information for a given day, do both individuals sign and date the entry at the end of the date?			√	None observed.
13. Does the log book define when a photograph is taken?		√		References to photographs were not discussed in any of the logbooks.
14. Is specific information that is compiled in another logbook (e.g., health and safety meeting log, borehole log, sample tracking sheets, field equipment calibration, etc.) referenced in the log book for cross-reference?		√		H&S meeting topics were not listed; field equipment calibration (e.g., PID) was not listed in a few cases.
15. Were hand-sketched maps of the site and/or field conditions prepared?	√			Very good use of hand sketches.
16. Did hand sketches provide direction arrows (north), discernible gradients, wind direction, vegetation, man-made structures, surrounding facilities, a scale and/or legend, water flow, or anything else considered important?	√			Good.
17. When collected, does the log book define the field sample identification associated with any environmental, forensics or geophysical samples?	√			Several entries did not provide an accurate name for the field sample collected (e.g., test pit sampling).
18. Where required, does the log book define the date of shipment of samples, courier method, and time released?		√		Many entries did not provide an accurate listing.
C. Log Book Review				
19. Does the Project Manager review the log book entry and sign and date his/her review?		√		No project manager reviews were observed in any of the log books.
20. Are acronyms defined?		√		Abundant use of acronyms, but no definitions provided.
21. Other items: <u>Work summaries</u>	√			Several log books created an “end of day” summary of all the tasks completed. This is an excellent practice. Some information not recorded during the day was later documented in the “daily summary.”

