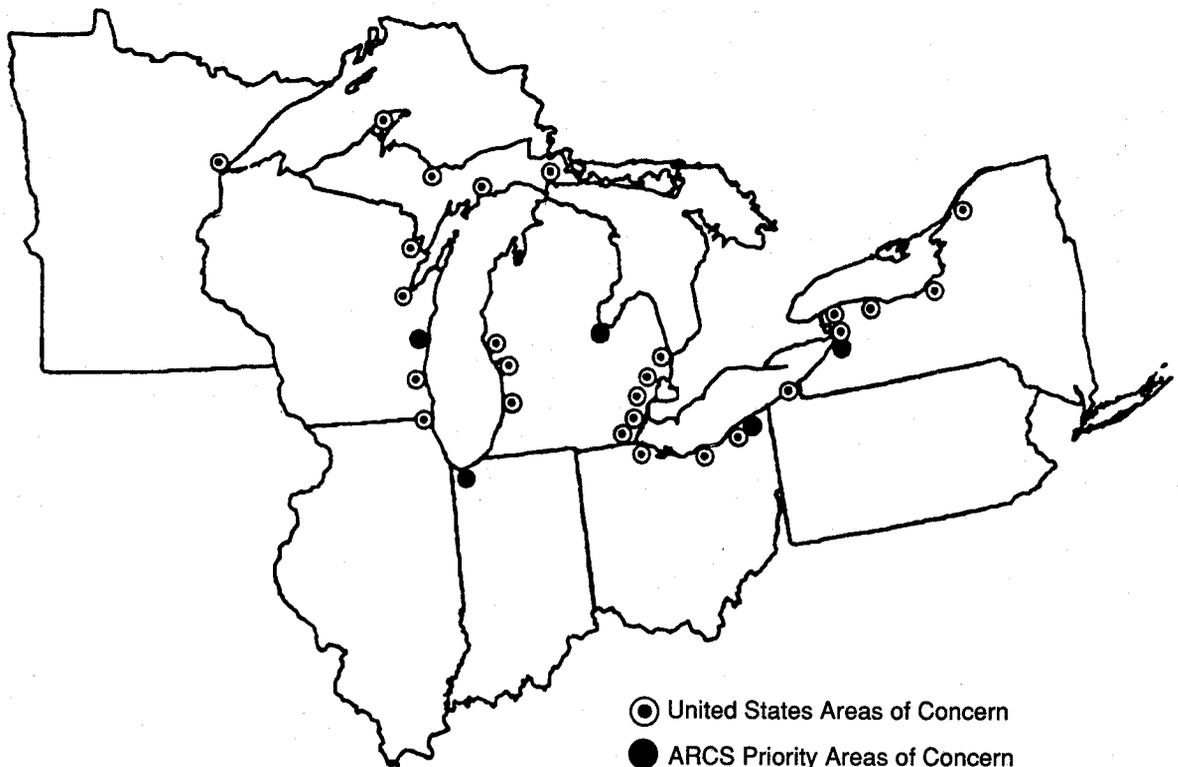




# Assessment and Remediation Of Contaminated Sediments (ARCS) Program



## BENCH-SCALE EVALUATION OF RCC'S BASIC EXTRACTIVE SLUDGE TREATMENT (B.E.S.T.)<sup>®</sup> PROCESS ON CONTAMINATED SEDIMENTS FROM THE BUFFALO, SAGINAW, AND GRAND CALUMET RIVERS



**Bench-Scale Evaluation of RCC's Basic Extractive Sludge  
Treatment (B.E.S.T.®) Process on Contaminated Sediments  
from the Buffalo, Saginaw, and Grand Calumet Rivers**

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## **DISCLAIMER**

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## ABSTRACT

The Great Lakes National Program Office (GLNPO) leads efforts to carry out the provisions of Section 118 of the Clean Water Act (CWA) and to fulfill U.S. obligations under the Great Lakes Water Quality Agreement (GLWQA) with Canada. Under Section 118(c)(3) of the CWA, GLNPO is responsible for undertaking a 5-year study and demonstration program for the remediation of contaminated sediments. GLNPO has initiated an Assessment and Remediation of Contaminated Sediments (ARCS) Program to carry out this responsibility. In order to develop a knowledge base from which informed decisions may be made, demonstrations of sediment treatment technologies are being conducted as part of the ARCS Program. Bench-scale studies on the B.E.S.T.® Solvent Extraction Process, which is the subject of this report, took place at Resources Conservation Company (RCC) in Bellevue, WA on August 5 to 9, 1991. The specific objectives for this effort were to determine process extraction efficiencies for polychlorinated biphenyls (PCBs) and polynuclear aromatic hydrocarbons (PAHs); to conduct a mass balance for solids, water, oil, PCBs and PAHs; and to examine process effects on metals, oil and grease, and several other parameters.

The B.E.S.T.® Solvent Extraction Process was tested using sediment samples obtained from the Buffalo River, Saginaw River, and Grand Calumet River. The concentration of the contaminants of concern in the sediment were 0.3 to 22 mg/kg PCBs and 3 to 220 mg/kg PAHs. The PCB and PAH concentrations of 0.2 to 0.4 and 0.4 to 37 mg/kg, respectively, were found in the treated solids. This corresponds to PCB and PAH removals of >95 to 99 percent and 65 to 96 percent, respectively. Metals analyses were performed on the treated solids and untreated sediments. The data demonstrate that the treatment process, as expected, had little affect on metal removal from the sediments. The feed sediments and treated solids were analyzed for percent moisture, oil and grease, total organic carbon (TOC), total volatile solids, and pH. Reductions in oil and grease concentrations (ranging from 80 to 99 percent) correspond to sediment PCB and PAH removal. A mass balance was also carried out as part of this study for the different constituents: solids, oil, water, PCBs, and PAHs.

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## 1.0 EXECUTIVE SUMMARY

The B.E.S.T.® Solvent Extraction Process was tested using sediments obtained from the Buffalo River, Saginaw River, and Grand Calumet River. The contaminants of concern in the sediments for these tests were PCBs and PAHs. Samples of the feed material and the treated solids produced using the B.E.S.T.® Solvent Extraction Process were analyzed by Battelle Marine Sciences Laboratory and RCC for residual PCB contamination. The data from these analyses are presented in Table 1.

**Table 1. Battelle and RCC Data - PCB Summary**

Sample	<u>Feed</u> (mg/kg, dry basis)		<u>Treated Solids</u> (mg/kg, dry basis)		<u>Removal Efficiency</u> %	
	Battelle	RCC	Battelle	RCC	Battelle	RCC
	Buffalo River	0.32	0.60	<0.3	<0.03	>6
Saginaw River	21.9	21	0.24	0.18	99	99
Grand Calumet River	15.0	22	0.44	0.23	97	99

As these data obtained by RCC and Battelle demonstrate, PCB removal efficiencies for the Grand Calumet River and Saginaw River sediments complement each other. However, the removal efficiencies determined by RCC and Battelle for the Buffalo River sediment are substantially different. This can be attributed to the fact that the contaminant concentration in the raw Buffalo River sediment was close to the analytical detection limit achievable by Battelle. The potential errors associated with these data undermine the relevance of the removal efficiency obtained by Battelle for the Buffalo River sediment.

Feed material and treated solids were also analyzed for residual PAH concentrations. Table 2 outlines the analytical results obtained by Battelle.

**Table 2. Battelle Data - Summary of Total PAHs**

Sample	<u>Feed</u> (mg/kg dry basis)	<u>Treated Solids</u> (mg/kg dry basis)	<u>Removal Efficiency</u> %
	Buffalo River	9.90	0.37
Saginaw River	2.70	0.95	65
Grand Calumet River	230	37.1	84

During the RCC analyses of the Buffalo River and Saginaw River sediments, residual PAH concentrations of <0.2 mg/kg per compound were found in the treated solids. Treated solids with PAH concentrations ranging from <1 to <3 mg/kg per compound were obtained for the Grand Calumet River sediment. Because RCC was unable to report lower detection limits, comparisons between RCC and Battelle data are not conclusive.

Metal analyses were performed on the treated solids and untreated sediments (see Table 11). The Battelle data demonstrate that the treatment process, as expected, had little affect on metal removal from the sediments. The RCC data cannot be compared to the Battelle data because these data were obtained using different analytical methods than those employed by Battelle. Because of the ashing of the sediment feed sample (potentially causing metals to be lost by volatilization) and because different methods were used to analyze the feed sediments and product solids, a reliable comparison of the RCC and Battelle data is not possible.

The feed sediments and treated solids were analyzed for percent moisture, oil and grease, Total Organic Carbon (TOC), volatile solids, and pH (see Table 12). As the data in Table 12 shows, the reductions in oil and grease concentrations (ranging from 80 to 99 percent) correspond to sediment PCB and PAH removal.

A mass balance was also carried out as part of this study. Table 3 summarizes the results obtained for the different constituents: solids, oil, water, PCBs, and PAHs.

**Table 3. Mass Balance Summary (percent recovered)**

Sample	<u>Solids</u>		<u>Oil</u>		<u>Water</u>		<u>PCBs</u>		<u>PAHs</u>
	Battelle	RCC	Battelle	RCC	Battelle	RCC	Battelle	RCC	Battelle
Buffalo River	98	97	163	112	68	70	129	70	90
Saginaw River	99	98	192	137	74	82	80	280	240
Grand Calumet River	92	86	69	97	78	75	94	64	11

Assuming that a full-scale application of this technology occurred and a volume of 500,000 tons of sediment required treatment, RCC estimated that it would cost approximately \$150 to \$250/ton to treat the material. The cost is dependent on the quantity of material processed, the cleanup target and the settling

characteristics of the waste. The waste would be treated at a rate of 200 to 300 tons per day using the B.E.S.T.® Model 615 Unit operated on a 24-hour-per-day basis. This estimate includes mobilization/demobilization costs but does not account for costs associated with site excavation, civil work, applicable taxes, pre-screening needs, and overall site management and disposition of the product oil.

Small vials of the residuals from the treatability test were retained and given to the EPA Technical Project Manager for the GLNPO for "show" purposes. All quantities of the test products (water, solids, and oil residuals) from each treatability test were sent to the analytical laboratory, Battelle Marine Sciences Laboratory, for analysis. Due to the quantities generated from the tests, none were retained and shipped to EPA for possible further treatability studies.

## **2.0 INTRODUCTION**

The Great Lakes National Program Office (GLNPO) leads efforts to carry out the provisions of Section 118 of the Clean Water Act (CWA) and to fulfill U.S. obligations under the Great Lakes Water Quality Agreement (GLWQA) with Canada. Under Section 118(c)(3) of the CWA, GLNPO was responsible for undertaking a 5-year study and demonstration program for the remediation of contaminated sediments. Five areas were specified for priority consideration in locating and conducting demonstration projects: Saginaw River and Bay, Michigan; Sheboygan River, Wisconsin; Grand Calumet River/Indiana Harbor Canal, Indiana; Ashtabula River, Ohio; and Buffalo River, New York. In response, GLNPO initiated the Assessment and Remediation of Contaminated Sediments (ARCS) Program.

In order to develop a knowledge base from which informed decisions may be made, bench- and pilot-scale demonstrations of sediment treatment technologies were conducted as part of the ARCS Program. Information from remedial activities supervised by the U.S Army Corps of Engineers and the Superfund program were also utilized. The Engineering/Technology (ET) Work Group was charged with overseeing the development and application of the bench- and pilot-scale tests.

Science Applications International Corporation (SAIC) was contracted to provide technical support to the ET Work Group. The effort consisted of conducting bench-scale treatability studies on designated sediments to evaluate the removal of specific organic contaminants. The bench-scale studies of the B.E.S.T.® Solvent Extraction Process, which are the subject of this report, took place at Resources Conservation Company (RCC) in Bellevue, WA on August 5 to 9, 1991. The specific objectives for this effort were: to determine process extraction efficiencies for polychlorinated biphenyls (PCBs) and

polynuclear aromatic hydrocarbons (PAHs); to conduct a mass balance for solids, water, oil, PCBs and PAHs; and to examine process effects on metals, oil and grease, and several other parameters.

## **2.1 Background**

SAIC and its subcontractors have conducted seven bench-scale tests for the ARCS Program on four different sediments using four treatment technologies: B.E.S.T.® Solvent Extraction Process (RCC), Low Temperature Thermal Desorption Process (ReTeC), Wet Air Oxidation (Zimpro Passavant), and Anaerobic Thermal Process Technology (SoilTech). This report summarizes the approach used and results obtained during treatability testing of the B.E.S.T.® Solvent Extraction Process. The sediments used during this technology evaluation were obtained from the Buffalo River, Grand Calumet River/Indiana Harbor Canal, and Saginaw River.

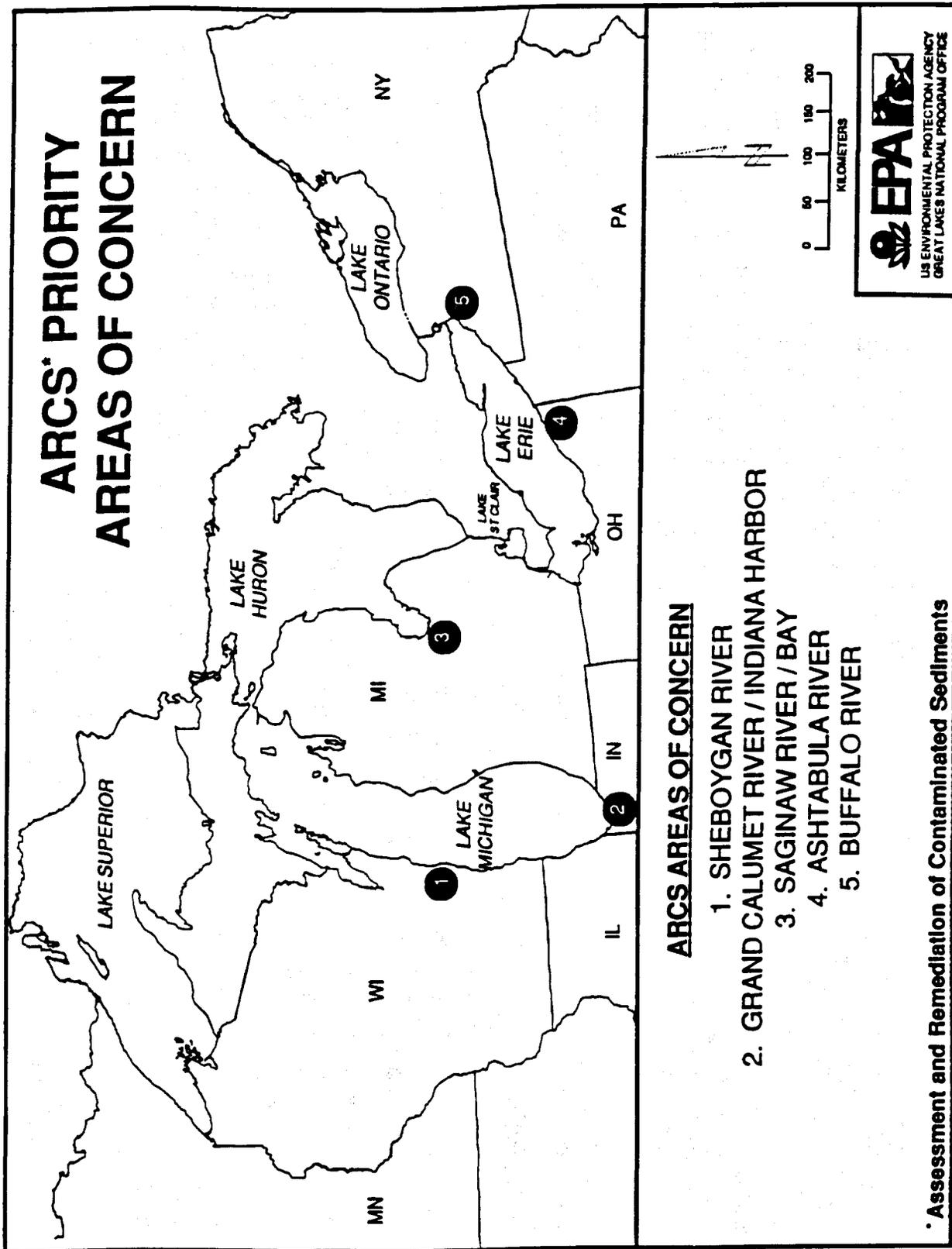
The primary objective of this portion of the study was to determine the feasibility and cost-effectiveness of the B.E.S.T.® Solvent Extraction Process for treating and removing PCBs and PAHs from the three sediments. Based upon previous tests performed by RCC, it is their experience that the data obtained from the bench tests simulate full-scale operation. Thus, data generated by these tests may be used to estimate treatment costs for full-scale operation and to evaluate process feasibility. The ability to evaluate process feasibility from these tests was also reported by the U.S. Environmental Protection Agency (EPA) in their report entitled, "Evaluation of the B.E.S.T.® Solvent Extraction Sludge Treatment Technology - Twenty-Four Hour Test."

## **2.2 Sediment Descriptions**

The sediments used for these tests are typical of sediments in the Great Lakes and their tributaries. They are representative of locations where future field demonstration projects may be conducted. For the purpose of these tests, the primary contaminants in these sediments were PCBs and PAHs.

### **2.2.1 Site Names and Locations for Each Sediment**

GLNPO collected sediments for study from the following areas around the Great Lakes: Saginaw River, Michigan; Sheboygan River, Wisconsin; Grand Calumet River/Indiana Harbor Canal, Indiana; Ashtabula River, Ohio; and Buffalo River, New York. SAIC was contracted to treat four of the sediments (from the Grand Calumet River/Indiana Harbor Canal, Buffalo River, Ashtabula River, and Saginaw River) using four different technologies. Samples from Grand Calumet River/Indiana Harbor Canal, Buffalo River, and Saginaw River were treated using the B.E.S.T.® Extraction Process. A map is provided in Figure 1 which shows the ARCS Priority Areas of Concern. Specifics of the sample location for the Buffalo River, Saginaw River and Grand Calumet River are shown in Figures 2, 3, and 4, respectively.



Assessment and Remediation of Contaminated Sediments

Figure 1. ARCS Priority Areas of Concern

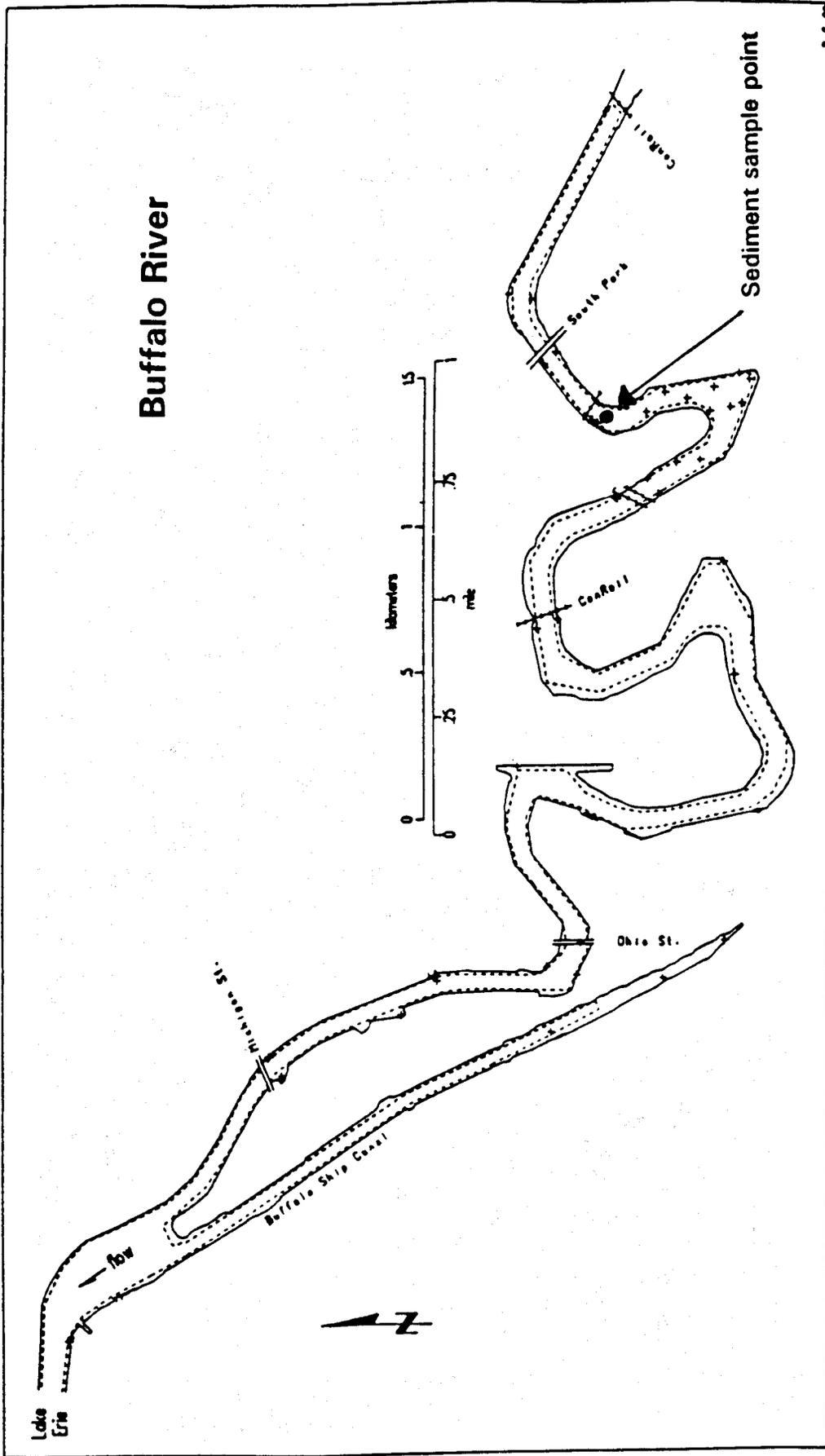


Figure 2. Buffalo River Sample Location

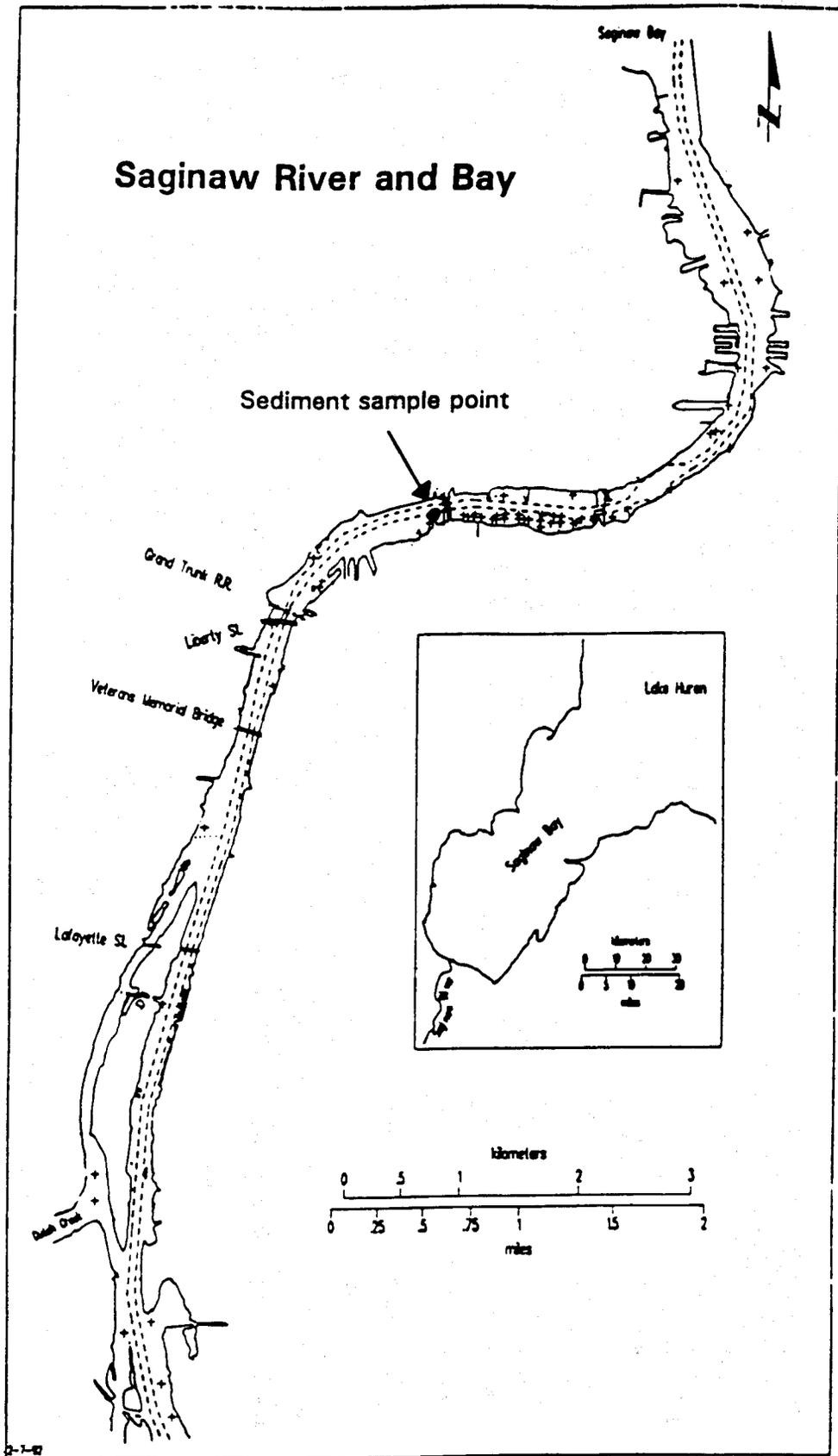


Figure 3. Saginaw River Sample Location

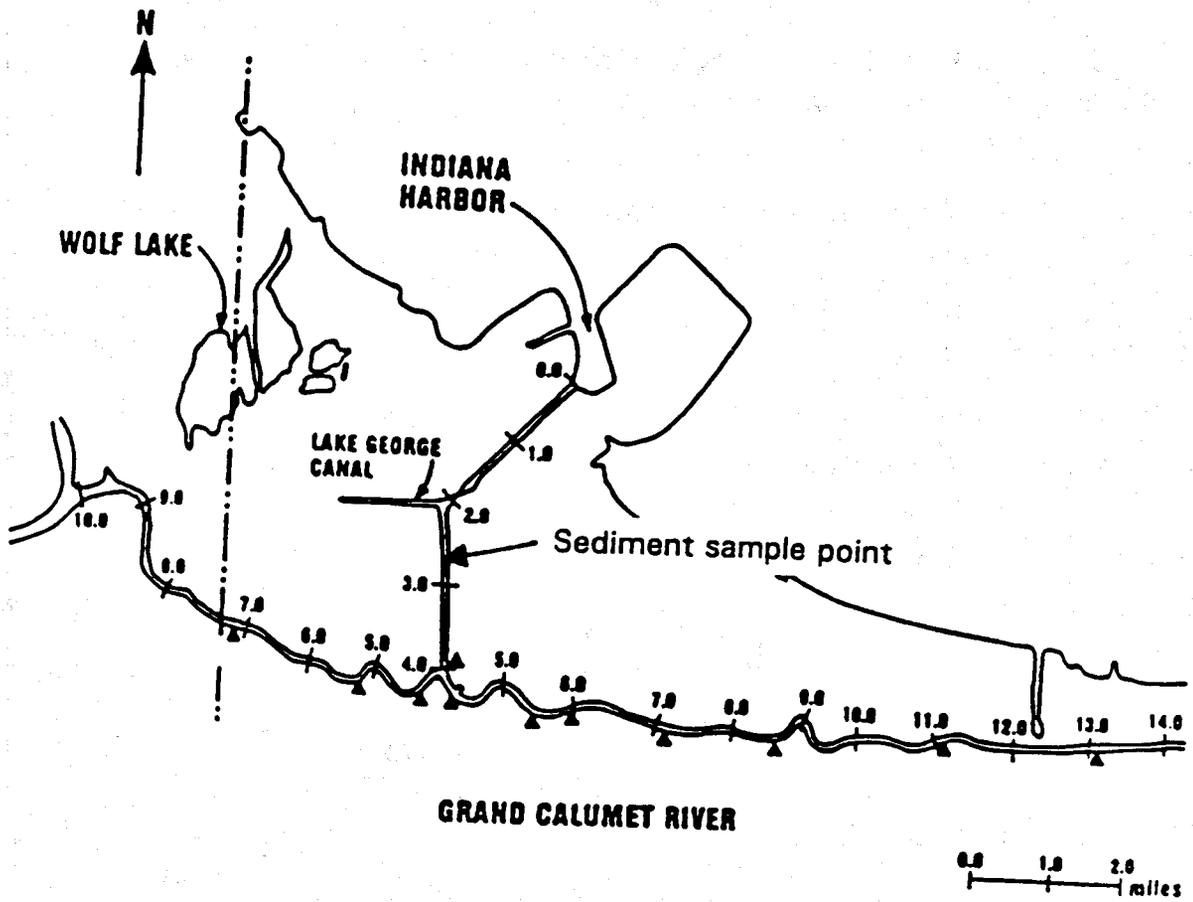


Figure 4. Grand Calumet River Sample Location

## 2.2.2 Sediment Acquisition and Homogenization

Prior to conducting the bench-scale treatability study using the B.E.S.T.® technology, the GLNPO samples were homogenized and stored under refrigeration by the U.S. EPA Environmental Research Laboratory in Duluth, Minnesota.

The homogenized sediments were sent to SAIC by the Duluth laboratory. Eighty ounces of each sediment sample were then transferred by SAIC to RCC. RCC used these samples to perform a series of standard tests to determine if the waste samples were compatible with their process and to determine optimum testing conditions and procedures for the treatability study (Phase I). The sediments used during the treatability studies also originated from this stock and were forwarded to RCC by SAIC.

## 2.3 Sediment Characterization

SAIC was responsible for the physical and chemical characterization of the raw sediment samples used during the tests. Table 4 provides characterization data of the sediments. In order to limit inter-laboratory variation, the different sediments and their residuals were analyzed by Battelle Marine Sciences Laboratory in Sequim, Washington. Raw sediment analyses conducted by RCC are also included in this report and can be found in Appendix A. The raw sediment samples analyzed by RCC and Battelle were collected simultaneously.

**Table 4. Battelle Data - Characterization of Feed Sediments  
(mg/kg, dry basis, unless specified)**

	Buffalo River	Saginaw River	Grand Calumet River
Total PCBs	0.32	21.9	15.0
Total PAHs	9.90	2.70	230
Moisture, % (as received)	42.0	24.0	57.0
Oil & Grease	2420	1350	32200
TOC, % weight	1.98	0.83	17.03
Total Volatile Solid, %	4.03	2.09	14.2
pH, S.U. (as received)	7.29	7.30	7.35

## 2.4 Technology Description

The B.E.S.T.® process is a patented solvent extraction technology developed by RCC. This process employs triethylamine (a solvent) to extract contaminants from wastes. Triethylamine is an aliphatic amine produced by reacting ethyl alcohol and ammonia. This solvent is distinguished from other solvents because it is inversely miscible. At temperatures below 65° F, triethylamine is completely miscible with water, while at temperatures above 65° F, triethylamine and water are only partially miscible. Since oil and water are similarly soluble in cold triethylamine, it can be utilized to treat wastes containing both contaminated oil and water.

The B.E.S.T.® process produces a single-phase extraction solution. If water and oil are present in the feed material, a homogeneous mixture of triethylamine, water, and oil is produced. Any organic contaminants contaminating the feed material, such as PCBs, PAHs, and volatile organic compounds (VOCs) are trapped within the water and oil portion of the extraction solution. Since triethylamine achieves intimate contact with the waste at nearly ambient temperatures and pressures, emulsions (oil containing the organic contaminants) are not expected to occlude the solute. Thus the extraction efficiency of the B.E.S.T.® process will not be compromised by feed mixtures with high water content.

RCC utilizes triethylamine because it exhibits several characteristics that enhance its use in a solvent extraction system. These characteristics, as reported by RCC, include: 1) a high vapor pressure (therefore, the triethylamine can be recovered from the extract via simple steam stripping); 2) formation of a low-boiling azeotrope with water (therefore, the solvent can be recovered from the treated solids by heat with a low energy input); 3) triethylamine has an alkaline pH=10 (therefore, some heavy metals are converted to the hydroxide form, which precipitate and exit the process with the treated solids); and 4) triethylamine is only moderately toxic and readily biodegrades (data available in EPA document EPA-600/2-82-001a show that a level of 200 ppm triethylamine in water was degraded completely in 11 hours by *Aerobacter*, a common soil bacteria).

A block diagram for the B.E.S.T.® Process is presented in Figure 5. Since triethylamine is soluble in water at temperatures below 65° F, the first extraction of the contaminated material is conducted at temperatures near 40° F. Therefore, the first extract solution will contain most of the water initially present in the feed material. If the first stage extract contains sufficient water to allow a phase separation of the triethylamine and water, the extract is heated to a temperature above the miscibility limit (130° F). At this temperature, the extract separates into two distinct phases; a triethylamine/oil phase and a water phase. The two phases are separated by gravity and decanted.

# Solvent Recovery

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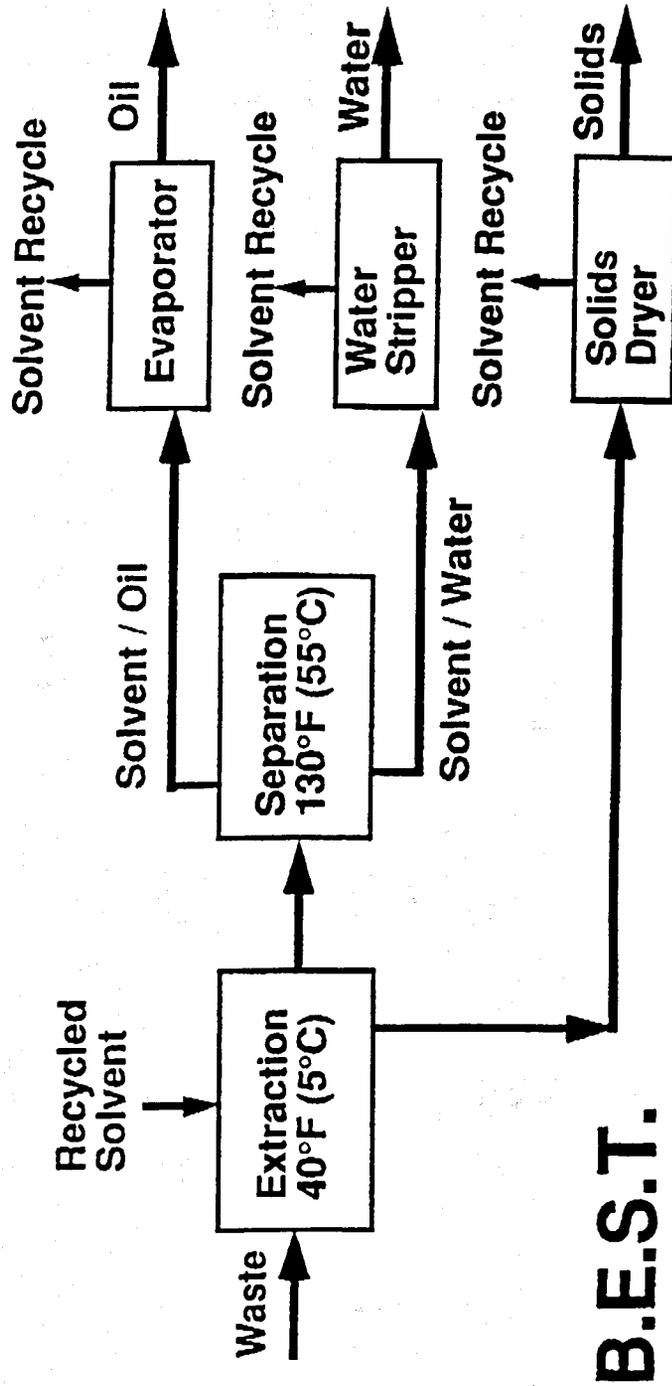


Figure 5. Flow Diagram of the B.E.S.T.® Process (Source: RCC, Inc.)

At 130° F the solubility of oil (organic contaminants) in triethylamine increases. Since this enhances the removal of oil from the contaminated solids, subsequent extractions are conducted at temperatures above 130° F. Because these extracts contain mostly oil and very little water, they are combined with the decanted triethylamine/oil phase from the first extraction stage. In the full-scale unit, the solvent is recovered from the two phases by way of steam stripping and is recycled directly to the extraction vessels for the solvent recovery portion of the process. Residual triethylamine in the water and oil products is usually low.

Triethylamine is removed from the treated solids by indirect heating with steam. A small amount of steam may be added directly to the dryer vessel to provide the water required to form the low boiling azeotrope. Typically the residual triethylamine remaining with the treated solids biodegrades readily. Thus, unless restricted by a contaminant not treated by the process, the dry treated solids may be used on site as backfill.

The B.E.S.T.® Process operates near ambient pressure and temperature and at a mildly alkaline pH. Liquid temperatures vary from about 40 to 170° F and high pressures are not required. A low-pressure nitrogen blanket creates a small positive pressure in tanks and vessels. Since the process operates in a closed loop with one small vent for removal of non-condensing gases, air emissions are minimal. RCC typically uses a water scrubber and activated carbon on this vent to minimize triethylamine releases.

### **3.0 TREATABILITY STUDY APPROACH**

#### **3.1 Test Objectives and Rationale**

SAIC was contracted by the ARCS Program to test four technologies for removing organic contaminants (PCBs and PAHs) from sediments typical of locations around the Great Lakes. This treatability study was performed to determine the feasibility and cost-effectiveness of the B.E.S.T.® Solvent Extraction Process for treating and removing PCBs and PAHs from three different sediments. In order to accomplish this, this bench-scale test had the following objectives:

- To record observations and data to predict full-scale performance of the B.E.S.T.® process
- To take samples during the extraction tests and conduct analyses sufficient to allow for calculation of mass balances for oil, water, solids and other compounds of interest
- To calculate the extraction efficiency of target compounds
- To obtain treated solids (300 g dry basis), water, and oil for independent analysis

Based upon previous tests performed by RCC, it is their experience that the data obtained from the bench test simulate full-scale operation. Ultimately, this data may be used to estimate both the feasibility and treatment costs associated with a full-scale application of the technology. The ability to evaluate process feasibility from these tests was also reported by EPA in their report entitled, " Evaluation of the B.E.S.T.® Solvent Extraction Sludge Treatment Technology - Twenty-Four Hour Test."

A two-phase approach was used during this study. During Phase I, SAIC sent a sample of the untreated sediments to RCC. These samples underwent a series of initial tests in order to determine the optimum conditions to be used during the actual treatability tests (Phase II). During Phase II, wet sediment from each of the three locations (Buffalo River, Grand Calumet River, and Saginaw River) was sent to RCC. Samples of raw (untreated) sediments and the various end products generated during the treatability tests (Phase II) were obtained and analyzed by both SAIC and RCC. The data generated by SAIC were primarily used to determine treatment extraction efficiencies and mass balances. Vendor- or subcontractor-generated data are reported and commented on when available.

This study is only one part of a much larger program and is not intended to evaluate the treatment of the sediments completely. In order to ensure that the data obtained from this study can be objectively compared with data generated from the other studies performed in support of the ARCS Program, Battelle Marine Sciences Laboratory was subcontracted to perform all analyses for the different treatability studies performed by SAIC (seven treatability studies utilizing four technologies on four sediments). The same set of analyses listed in Table 5 was applied during the characterization of each raw sediment and end products from the different treatability tests. In addition, representatives from SAIC observed how all Phase II treatability tests were conducted.

**Table 5. Parameters for Analysis of ARCS Technologies**

Parameters	
TOC/TIC	Arsenic
Total Solids	Barium
Volatile Solids	Cadmium
Oil & Grease	Chromium
Total Cyanide	Copper
Total Phosphorus	Iron (total)
PCBs (total & Aroclors)	Lead
PAHs (16)	Manganese
pH	Mercury
BOD	Nickel
Total Suspended Solids	Selenium
Conductivity	Silver
	Zinc

### 3.2 Experimental Design and Procedures

#### 3.2.1 Phase I

Phase I was designed to allow RCC to explore a range of variables in order to set test parameters which would optimize the performance of the B.E.S.T.® technology for the bench-scale tests (Phase II). In order to accomplish this, samples of the different sediments were sent to RCC by SAIC prior to bench-scale testing. The amount of material sent was approximately 1 kg of each sediment, as specified by RCC.

RCC analyzed the three raw sediment samples to determine whether the sediments were compatible with triethylamine. During these compatibility tests, the feed samples were individually mixed with cold triethylamine and then monitored to observe the amount of heat generated as well as any visual signs that adverse reactions (such as an extremely exothermic chemical reaction) were occurring. All samples satisfied the compatibility criteria.

Since triethylamine can be ionized at a low pH into unrecoverable triethylammonium salts, the pH of the sample needed to be adjusted to approximately 11 during Phase II in order to enable RCC to efficiently recover the triethylamine from the separated phase fraction products. To determine the amount of caustic needed to increase the pH of the raw feed to the operating pH of the B.E.S.T.® process (pH = 11), RCC slurried 5 g portions of the feed samples with deionized water. Incremental portions of the caustic (50% sodium hydroxide) were added to bring the pH to 11. The amount of caustic required to adjust the pH was recorded.

After observing these simple mixing tests previously described, RCC applied its past experience in selecting operating parameters for the technology to determine the number of extraction stages, solvent to feed ratios, and the proposed temperature and mixing time for each stage for the bench-scale tests.

### 3.2.2 Phase II

Phase II of the treatability program is referred to as the "B.E.S.T.® Bench-scale Treatability Test Workup" in RCC's B.E.S.T.® Bench-Scale Treatability Test Plan. This section outlines five major operations including: 1) Pre-treatment and first wash; 2) Second wash; 3) Third wash and solids drying; 4) Decantation; and 5) Distillation. These procedures and associated equipment used are described in Appendix B.

#### 3.2.2.1 *Procedures*

##### Test Sample Preparation--

The contaminated samples from the Buffalo River, Saginaw River, and Grand Calumet River sites were gray-colored sediments with very little debris present. Each of the three samples contained free-standing water. As it was very difficult to homogenize the samples with the free-standing water present, this water was decanted prior to conducting the bench-scale tests and proportionally recombined with the portion used for the bench testing. This was done by weighing the entire decanted sediment and the portion used for the test. The percentage of the portion used to the whole determined the percentage of the decanted water to recombine with the test sample.

Bench-scale testing requires material greater than 1/4 inch be removed. Full-scale processing requires that the feeds be screened to remove only material greater than 1 inch in diameter. There was no material greater than 1/4 inch in any of the three samples received. Therefore, the samples were not screened.

The following are summaries of the five major operations. With slight variations, the same procedure was used to treat each of the sediments.

##### Pre-treatment and First Wash--

During the treatability tests of the different sediments, each sample was placed in a 4-L resin kettle immersed in a temperature-controlled water bath set at 33° F. Each sample's pH was adjusted using sodium hydroxide (NaOH) and 2.7 L of chilled triethylamine (2 percent H<sub>2</sub>O). The 2 percent water was

added to offset the amount of water remaining in the triethylamine after the extraction step. The amount of sodium hydroxide needed to adjust the pH of each sample to 11 was determined during Phase I and is listed in Table 6, as is the amount of sediment treated during each trial.

**Table 6. Sodium Hydroxide Addition**

Sediment	Sample Weight (g)	Caustic Added per kg of Sediment (ml 50% NaOH)
Buffalo River	700	6.0
Saginaw River	700	6.0
Grand Calumet River	1400	6.0

While immersed in the cooling bath, the sample was mixed with the NaOH and the chilled triethylamine using a air-driven prop mixer. Mixing occurred for approximately 20 minutes for the Saginaw River and Buffalo River sediments with the pneumatic mixer in the chiller bath. Because of the high water content of the Grand Calumet River sediment, a larger sample size was needed to yield the quantity of treated solids required. For this sediment, the first extraction was conducted in two steps, using 700 g of feed for each step. Each sample was mixed for 10 minutes.

At the end of the first mixing stage, the sample was allowed to separate by gravity. The particulates were then separated from the liquid by centrifuging the extract at 2,100 rpm for 10 minutes. The solvent/oil/water/centrate were set aside for later decantation. The solids from the centrifuge were placed back into the resin kettle for additional wash stages.

#### Second Wash--

Solids recovered from the first extraction were mixed with 2.7 L of fresh triethylamine. Part of the triethylamine was used to transfer solids from the centrifuge bottles into the mixing container. For the second extraction, the samples were heated to 127 to 140° F. The mixture was kept heated while mixing was in progress. Mixing was conducted with a pneumatic mixer for approximately 20 minutes. This sample settled very quickly. The solvent/oil was poured off and held for later combination with the solvent/oil portion from the decantation procedure.

### Third Wash and Solids Drying--

For the third wash, the same procedure that was used in the second wash was repeated. Mixing for this wash was for 30 minutes.

The treated solids resulting from the third wash were then dried at 220° F in a forced-draft oven. Occasional mixing in the oven to facilitate triethylamine volatilization was conducted. This mixing was accomplished by turning the sample in the oven with a clean spatula. After the initial drying, a portion of de-ionized water was added to wet the solids thoroughly. The solids were then redried in order to reduce residual triethylamine concentrations further. To ensure that the triethylamine residual in the dried solids was low, the solids were treated with caustic soda (applied with the de-ionized water) when the pH of these solids was less than 10. Sufficient caustic soda was added to raise the pH to approximately 10.5. The required amount of caustic soda added was determined on a small portion of the solids.

### Decantation--

The first stage extracts trap nearly all of the water present in the feed sample. Because of this, only the water from the first stage extracts is recovered. There are two methods to decant water. The decantation method is chosen depending on the water content of the feed. The following methods were employed to recover the water from the test series samples.

**Method 1:** During the decantation of the Buffalo River and Grand Calumet River extracts, a 4-L separatory funnel immersed in a temperature-controlled water tank was employed. The tank was kept at 140° F by circulating water between the tank and a temperature-controlled water bath set at 140° F.

Supernatant/centrate from the first wash (chilled to this point at 40° F) was heated to above 130° F with continuous mixing on a hotplate and poured into the separatory funnel. Since above 130° F water is no longer miscible with the triethylamine, the water settled to the bottom of the mixture. Forty minutes quiescent residence time in the separatory funnel for the Buffalo River sample and 15 and 90 minutes for the Grand Calumet River sample were required. The length of time required depended on how long it took for near-separation of the layers. A sample from the rag layer, which is an emulsion where any solids present tend to collect and create a region where the triethylamine/oil/water separation is not distinct, was taken by RCC for later possible analyses. Generally, the smaller the rag layer is in comparison to the triethylamine/oil and water phases, the better the separation. The rag layers for all three sediments were relatively small and within expected volumes. SAIC did not collect samples from this rag layer.

Method 2: Because of its low water content (<25%), the water present in the Saginaw River extract was separated from the oil by evaporation instead of decantation. When the first extract was evaporated, the water in the triethylamine/oil/water mixture formed an azeotrope with the distilled triethylamine, leaving the oil behind. After the triethylamine/water isotope had condensed, the water was decanted by heating the triethylamine/water mixture above 130° F and pouring the mixture into the 4-L separatory funnel. Since no temperature control system was required, separation occurred immediately. This method produces a much purer water stream and is preferable for low-water-content feeds where the extra energy cost to evaporate the water is small.

#### Distillation--

Water Layer: After recording the water's pH, which should have been >10, and its volume, the water was stripped by steam at 110° C in a Buchi Rotovapor apparatus to ensure that the triethylamine left the water. Periodically, the water volume and water pH were checked. When the water pH was <10, the pH was adjusted to >12 and stripping continued until the pH of the water remained above 10. The pH was periodically checked, and if found <10, the previous steps were repeated until the water pH remained above 10. At this point, distilling continued for 15 minutes longer before being terminated. The elevated pH is necessary to ensure that the majority of the triethylamine remains in the volatile molecular form.

Oil/Triethylamine Layer: The bulk of triethylamine was removed from this layer by boiling the triethylamine/oil mixture at 110° C in the Rotovapor (no steam necessary). The triethylamine condensed as it evaporated and was collected separately. Normally the oil remaining in the flask would then be steam-stripped of any residual triethylamine by adding a known quantity of water (typically 5 ml) to the hot oil in the boiling flask of the rotovapor and then measuring the volume of distillate recovered. When all triethylamine was removed, the amount of the distillate recovered would equal the amount of water added. However, because of the low oil content of the feed in this test, the amount of oil recovered was too small to enable the oil to be effectively stripped. Triethylamine was allowed to remain in the Rotovapor, thereby allowing the oil from the sample to remain in solution. The homogeneous oil was able to be poured out of the Rotovapor flask. The final product oil/triethylamine weight was recorded. This procedure is an artifact of this test due to the small quantities of sediment.

### 3.3 Sampling and Analysis

The Quality Assurance Project Plan is presented in Appendix C.

### 3.3.1 Sampling

At the beginning of the Phase II treatability test, SAIC personnel observing Phase II packed and shipped a sample of the untreated Buffalo River, Grand Calumet River and Saginaw River sediments to SAIC's subcontract laboratory, Battelle, in accordance with written detailed instructions supplied to the SAIC on-site representative. Each sample contained free-standing water which was decanted prior to conducting feed analyses and was proportionally recombined prior to any analysis and bench testing. This was done because it is very difficult to homogenize material when free-standing water is present. These samples were obtained from separate unopened containers of the sediments sent for Phase II.

Although the samples would normally be screened to remove any material greater than 1/4 inch, Phase I results indicated that no material of that size or greater was present in the three samples. Thus the samples were not screened.

After the extractions were complete, samples of the final water, oil, and solids residuals were distributed to SAIC and RCC. As specified in the Quality Assurance Project Plan (QAPP) a minimum of 300 g (dry basis) of solid material was required in order for Battelle to be able to complete the necessary analyses of that material. Since the quantity of oil and water was dependent on the sediment and the technology employed, it was not possible to obtain enough water and oil to perform the full scope of analyses specified in Table 7.

### 3.3.2 Analysis

Two separate sets of analyses were conducted by SAIC's subcontracted laboratory, Battelle, and RCC on the three raw sediments and the process products during Phase II. Battelle's data was used for the results presented in this report. RCC's data is discussed and commented upon, where possible, to facilitate interpretation of the results of the treatability test.

#### 3.3.2.1 *Battelle Analyses*

Following the Phase II treatability test, Battelle conducted analyses on the three raw sediments and the end products. The number of analyses conducted on these sediments and their residuals are listed in Table 6. Descriptions of the analytical methods employed can be found in the QA Section of this report.

Since the actual quantities of oil and water produced by the technology during the bench-scale treatability tests were not sufficient to perform all the analyses in Table 6, only PCB and PAH analyses were performed on the water and oil.

Table 7. SAIC's Analysis Schedule for the Phase II Solvent Extraction Evaluation of Buffalo River, Grand Calumet River, and Saginaw River Sediments

Parameters	QC Sample ( ) Method Blank	Untreated Sediment	MS	Tripli- cate	Treated Solids	MS	MSD	Tripli- cate	Water	MS	MSD	Tripli- cate	Oil	MS	Tripli- cate
Total Solids (Moisture)	(1) YES	(3) B,G,S		(2) S	(3) B,G,S			(2) S							
Volatile Solids	(1) YES	(3) B,G,S		(2) S	(3) B,G,S			(2) S	NA*						NA
O & G	(1) YES	(3) B,G,S		(2) S	(3) B,G,S			(2) S	NA						NA
Metals	(0) YES	(3) B,G,S	(1) S	(2) S	(3) B,G,S	(1) S		(2) S	NA	NA					NA
PCBs	(1) YES**	(3) B,G,S	(1) S	(2) S	(3) B,G,S	(1) S	(1) S	(2) S	(3) B,G,S	NA	NA		(3) B,G,S	(1) S	(2) S
PAHs	(1) YES**	(3) B,G,S	(1) S	(2) S	(3) B,G,S	(1) S	(1) S	(2) S	(3) B,G,S	NA	NA		(3) B,G,S	(1) S	(2) S
TOC	(0) YES	(3) B,G,S	NA	NA	(3) B,G,S	NA	NA	NA	NA	NA	NA				
Total Cyanide	(0) YES	(3) B,G,S	NA	NA	(3) B,G,S	NA	NA	NA	NA	NA	NA				
Total Phosphorus	(0) YES	(3) B,G,S	NA	NA	(3) B,G,S	NA	NA	NA	NA	NA	NA				
pH	(0) YES	(3) B,G,S		NA	(3) B,G,S			NA	NA						NA
BOD	NA														NA
Total Suspended Solids	NA								NA						NA
Conductivity	NA								NA						NA

\* Not Analyzed  
 \*\* A laboratory pure water spike is required for recovery determination  
 (3) = Number of Analyses  
 B = Buffalo River  
 G = Grand Calumet River  
 S = Saginaw Bay  
 MS = Matrix Spike  
 MSD = Matrix Spike Duplicate

### 3.3.2.2 RCC Analyses

RCC analyzed the sediment samples for moisture content, oil and grease, ash content, metals, PAHs, PCBs, and particulate solids content. RCC also conducted their own analyses on the products for Phase II. Table 8 shows the analyses performed by RCC. Details on the analytical methods used by RCC are presented in Appendix D. The following section is a summary of the RCC analyses conducted.

**Table 8. RCC Analyses**

Matrix Sample	PAHs	PCBs	Total Metals	Oil & Grease	Solvent	Water	Particulate Content	TCLP	pH
Raw Sediment	yes	yes	yes	yes	no	yes	yes	no	yes
Treated Solids	yes	yes	yes	yes	yes	no	yes	metals	yes
Residual Oil	no	no	no	no	yes	no	no	no	no
Residual Water	no	yes	yes	no	no	no	no	no	no

TCLP = Toxicity Characteristic Leaching Procedure

## 4.0 RESULTS AND DISCUSSION

### 4.1 Summary of Phase I Results

RCC analyzed the three raw sediment samples to determine whether the sediments were compatible with triethylamine. Since there were no visible signs indicating that adverse reactions were occurring and the heat of the solution did not exceed normal expectations, the B.E.S.T.® bench-scale treatability tests proceeded.

The amount of caustic (NaOH) needed to increase the pH of the raw sediments to the operating pH of the B.E.S.T.® process (pH = 11) was determined. This information, as well as the original pH of the sample, is summarized in Table 9.

**Table 9. pH Adjustments**

Sediment	Initial pH	Caustic Added per kg of Sediment (ml 50% NaOH)
Buffalo River	7.6	6.0
Saginaw River	8.1	6.0
Grand Calumet River	7.5	6.0

A sieve analysis of the raw sediment was conducted to determine the screening and size reduction requirements. Since there was no material greater than 1/4-inch in diameter, sample screening was determined unnecessary.

#### **4.2 Summary of Phase II Results**

As stated previously, the concentrations of PAHs, PCBs, metals, total solids, volatile solids, and oil and grease present in the untreated sediments and treated solids are the critical measurements associated with this study. Oil and water residuals were analyzed to determine the fate of the contaminants of concern from the process. Since insufficient water and oil was produced from the quantity of untreated sediments used with the B.E.S.T.® process to perform all the analyses listed in Table 7, only PCB and PAH analyses were performed on the water and oil residuals. The following sections briefly address the analytical results obtained for contaminant concentrations present in the raw sediments and the process residuals (i.e., treated solids, water, and oil), as well as applicable extraction efficiencies. The discussion of Phase II results concludes with an analysis of the mass balance of the media and contaminants. The analytical data received from Battelle can be found in Appendix E.

Individual PAH compounds, PCB Aroclors, and metals were quantitated during sample analyses. In order to determine overall removal efficiencies for each class, it was necessary to sum these individual results. In instances where all reported results were less than the analytical detection limits, total concentrations are reported as less than the sum of the individual detection limits. Where one or more individual components are above detection limits, total concentrations are reported as the sum of these detected values.

#### 4.2.1 Sediments/Treated Solids

##### 4.2.1.1 PCBs

Samples of the feed material and the treated solids produced using the B.E.S.T.® Solvent Extraction Process were analyzed for PCB contamination. The data from these analyses are presented in Table 10.

**Table 10. Battelle Data - Total PCBs**

Sample	Feed (mg/kg, dry basis)	Treated Solids (mg/kg, dry basis)	Removal Efficiency %
Buffalo River <sup>1</sup>	0.32	<0.3	>6
Saginaw River <sup>2</sup>	21.9	0.24	99
Grand Calumet River <sup>3</sup>	15.0	0.44	97

<sup>1</sup> Identified primarily as Aroclor 1248

<sup>2</sup> Identified primarily as Aroclor 1242

<sup>3</sup> Identified primarily as Aroclor 1248

As demonstrated by these data, PCB concentrations of 0.24 mg/kg and 0.44 mg/kg were found in the treated solids generated from the Saginaw River and Grand Calumet River sediments, respectively. This corresponds to PCB removal efficiencies of 99 and 97 percent. At first glance, the Buffalo River solids achieved a much lower removal efficiency (i.e., >6 percent). This is attributed to the low PCB concentrations initially present in the untreated Buffalo River sediment and the high analytical detection limits achieved. As the concentration of a contaminant approaches analytical detection limits, the error associated with the analytical readings obtained increases. Thus, the relevance of the removal efficiency achieved for the Buffalo River sediment is undermined by: 1) error associated with the measurement of the contaminant concentrations near detection limits and 2) the high detection limits obtained for the samples.

##### 4.2.1.2 PAHs

Feed material and treated solids were also analyzed for PAHs. As shown in Table 11, total PAH concentrations of 0.37 mg/kg, 0.95 mg/kg, and 37.1 mg/kg were found in the treated solids produced by treating the Buffalo River, Saginaw River, and Grand Calumet River sediments. These values correspond to removal efficiencies of 96, 65, and 84 percent, respectively.

**Table 11. Battelle Data - Feed and Treated Solid PAH Concentrations (mg/kg, dry basis)**

Contaminant	Buffalo River			Saginaw River			Grand Calumet River		
	Feed	Treated	% Removal	Feed	Treated	% Removal	Feed	Treated	% Removal
Naphthalene	0.107	0.037	64	0.026	0.024	08	4.40	2.25	49
Acenaphthylene	<0.080	<0.020	NC	<0.020	<0.020	NC	2.32	0.121	95
Acenaphthene	<0.200	<0.030	NC	<0.030	<0.020	NC	4.40	0.726	84
Fluorene	0.160	<0.020	>88	0.033	<0.020	>39	4.62	1.08	77
Phenanthrene	1.020	0.068	93	0.267	0.099	62	15.2	5.64	63
Anthracene	0.547	0.030	95	0.066	0.017	70	5.63	1.47	74
Fluoranthene	1.20	0.045	96	0.397	0.138	65	32.0	3.11	90
Pyrene	1.16	0.039	97	0.439	0.120	73	32.0	3.55	89
Benzo(a)anthracene	0.861	0.022	98	0.186	0.057	68	18.3	3.13	83
Chrysene	1.04	0.039	96	0.269	0.088	67	24.4	3.99	84
Benzo(b)fluoranthene	0.876	0.027	96	0.242	0.097	60	19.2	1.89	90
Benzo(k)fluoranthene	0.733	0.004	100	0.179	0.066	63	13.4	1.32	90
Benzo(a)pyrene	0.887	0.018	98	0.225	0.076	65	20.6	3.22	84
Ideno(1,2,3-cd)pyrene	0.607	0.018	97	0.207	0.090	56	14.7	1.36	91
Dibenzo(a,h)anthracene	0.205	0.006	95	0.043	0.016	68	5.22	1.93	63
Benzo(g,h,i)perylene	0.495	0.014	96	0.117	0.060	48	13.8	2.35	83
Total PAH	9.90	0.37	96	2.70	0.95	65	230	37.1	84

NC = Not Calculated

Generally, the low removal efficiencies obtained for the PAHs in the Saginaw River sediment can be attributed to the low concentration of PAHs initially present in the sediment and errors associated with evaluating contaminant concentrations close to analytical detection limits.

The removal efficiency of 84 percent for the total PAHs in the Grand Calumet River sediment resulted in a final concentration of 37.1 mg/kg of PAHs in the treated solids. Additional extractions would likely reduce PAH concentrations in the treated solids even further.

#### 4.2.1.3 Total Metals

The data in Table 12 highlight the removal achieved for the metal contaminants present in the untreated feed and the treated solids. As demonstrated by the low or negative removal percentages, in general, the B.E.S.T.® Solvent Extraction Process does not effectively remove metals.

**Table 12. Battelle Data - Metals Concentration in the Feed and Treated Solids (mg/kg, dry basis)**

Contaminant	Buffalo River			Saginaw River			Grand Calumet River		
	Feed	Treated	% Removal	Feed	Treated	% Removal	Feed	Treated	% Removal
Arsenic	12.7	14.6	-15	2.21	2.85	-29	22.8	29.0	-27
Barium	413	396	4	322	319	1	317	290	9
Cadmium	2.10	2.11	-0	4.14	4.26	-3	8.56	6.97	19
Chromium	109	113	-4	107	118	-10	2270	1710	25
Copper	70.2	61.2	13	58.8	64.1	-9	188	223	-19
Iron	42900	44200	-3	7870	8260	-5	188000	82500	56
Lead	102	102	0	45.5	46.6	-2	582	656	-13
Manganese	667	684	-3	165	177	-7	3230	2540	21
Mercury	0.551	0.627	-14	0.167	0.335	-99	1.53	1.46	4
Nickel	43.1	42.1	2	58.3	64.3	-10	12.9	<10	>22
Selenium	0.74	0.87	-18	<0.3	<0.3	NC	<0.3	4.94	NC
Silver	0.31	0.24	23	0.84	0.82	2	4.84	4.34	10
Zinc	180	190	-6	140	169	-21	2380	2810	-18

NC = Not Calculated

#### 4.2.1.4 Other Analyses

The feed sediments and treated solids were analyzed for percent moisture, oil and grease, TOC, total volatile solids, and pH as shown in Table 13. As shown by comparing data in Tables 10 and 11 with data in Table 13, reductions in oil and grease concentrations correspond to PCB and PAH removal. This demonstrates that oil and grease analysis could possibly be used as a low-cost indicator for technology effectiveness for a given sediment.

**Table 13. Battelle Data - Removal Efficiencies for Other Parameters  
(mg/kg, dry basis, unless specified)**

Contaminant	Buffalo River			Saginaw River			Grand Calumet River		
	Feed	Treated	% Removal	Feed	Treated	% Removal	Feed	Treated	% Removal
Total PCBs	0.32	<0.3	>6	21.9	0.24	99	15.0	0.44	97
Total PAHs	9.90	0.37	96	2.70	0.95	65	230	37.1	84
Moisture, % (as received)	42.0	3.72		24.0	0.16		57.0	0.50	
Oil & Grease	2420	238	90	1350	265	80	32200	470	99
TOC, % weight	1.98	1.21	39	0.83	0.58	30	17.0	13.4	21
Total Volatile Solids, %	4.03	3.91	3	2.09	1.73	17	14.2	9.06	36
pH, S.U. (as received)	7.29	10.30		7.30	10.73		7.35	10.25	

#### 4.2.2 Oil

The concentrations of PAHs and PCBs in the oil extracted from the three sediments can be found in Tables 14 and 15. Final concentrations in the process solids and water have been included as a comparative measure of performance. Using values for percent oil determined in the samples received by Battelle (i.e., 9.3 percent for Saginaw River extract, 6.3 percent for the Buffalo River extract, and 60.0 percent for the Grand Calumet River extract), these concentrations have been adjusted to account for the triethylamine diluent found in the different oil samples received for analysis. The triethylamine was left in these samples because of the low oil content and small sample size used for these tests.

Please note that the possibility for introducing error to these corrected oil concentrations does exist. Analytically determined values are adjusted for the amount of oil determined to be present in the oil/triethylamine solution. When the PAH and PCB concentrations are adjusted, any error in this oil analysis may be conveyed to the new concentrations. Samples with less oil (i.e., Saginaw River and Buffalo River) are more likely to be affected.

**Table 14. Battelle Data - PAH Concentrations in the Treated Solids, Water, and Oil**

Contaminant	Buffalo River			Saginaw River			Grand Calumet River		
	Solid (ug/kg, dry)	Water (ug/L)	Oil <sup>a</sup> (ug/kg)	Solid (ug/kg, dry)	Water (ug/L)	Oil <sup>a</sup> (ug/kg)	Solid (ug/kg, dry)	Water (ug/L)	Oil <sup>a</sup> (ug/kg)
Naphthalene	37	0.998	<9000	24	1.44	<9000	2250	0.301	<30000
Acenaphthylene	<20	<0.4	<10000	<20	<0.6	<20000	121	0.495	48000
Acenaphthene	<30	<0.5	<20000	<20	<0.7	<20000	726	0.165	<60000
Fluorene	<20	<0.5	24500	<20	<0.7	18000	1080	0.278	51800
Phenanthrene	68	<0.3	160000	99	<0.5	170000	5640	2.72	213000
Anthracene	30	<0.4	126000	17	<0.6	104000	1470	0.997	110000
Fluoranthene	45	<0.3	201000	138	0.402	280000	3110	17.1	636000
Pyrene	39	<0.3	183000	120	<0.5	257000	3550	18.0	608000
Benzo(a)anthracene	22	<0.3	89400	57	<0.5	116000	3130	8.42	349000
Chrysene	39	0.242	120000	88	<0.4	143000	3990	10.9	484000
Benzo(b)fluoranthene	27	<0.3	86900	97	<0.4	125000	1890	6.80	432000
Benzo(k)fluoranthene	4	<0.2	68400	66	<0.3	92400	1320	3.97	289000
Benzo(a)pyrene	18	<0.3	84900	76	<0.4	114000	3220	6.18	433000
Indeno(1,2,3-cd)pyrene	18	<0.3	67500	90	<0.4	90500	1360	3.24	362000
Dibenzo(a,h)anthracene	6	<0.3	13300	16	<0.4	31000	1930	0.762	75400
Benzo(g,h,i)perylene	14	0.184	39400	60	0.289	69500	2350	2.84	217000
Total PAH	370	1.41	1260000	950	2.13	1610000	37100	83.2	4310000

<sup>a</sup> Corrected for actual volumes of oil present in oil/triethylamine samples analyzed.  
(9.3 percent for Saginaw River, 6.3 percent for Buffalo River, 60.0 percent for Grand Calumet River)

**Table 15. Battelle Data - PCB Concentrations in the Treated Solids, Water, and Oil**

Contami- nant	<u>Buffalo River</u>			<u>Saginaw River</u>			<u>Grand Calumet River</u>		
	Solids (ug/kg, dry)	Water (ug/L)	Oil <sup>a</sup> (ug/kg)	Solids (ug/kg, dry)	Water (ug/L)	Oil <sup>a</sup> (ug/kg)	Solids (ug/kg, dry)	Water (ug/L)	Oil <sup>a</sup> (ug/kg)
Total PCBs	<300	<0.6	62300	235	<0.6	5012000	440	4.8	268000

<sup>a</sup> Corrected for actual volumes of oil present in oil/triethylamine samples analyzed.  
(9.3 percent for Saginaw River, 6.3 percent for Buffalo River, 60.0 percent for Grand Calumet River)

#### 4.2.3 Water

The concentrations of PAHs and PCBs in the water extracted from the three sediments can also be found in Tables 14 and 15. As the data demonstrate, individual PAH and PCB concentrations for the Buffalo River and Saginaw River residual waters were mainly below the detection limits. Please note that, like the PAH and PCB concentrations associated with the treated solids and untreated sediments, the PAH and PCB concentrations found in the Grand Calumet River residual waters were substantially higher than the concentrations found in the Buffalo River and Saginaw River residual waters. Possibly additional extractions could reduce these concentrations to levels comparable to Buffalo River and Saginaw River concentrations.

#### 4.2.4 Mass Balance

For the B.E.S.T.® bench-scale treatability tests, good mass balance closures were obtained for the solids, water, oil, PCBs, and PAHs. The following sections address the different mass balances and expand on those factors that influenced their closure. Tables are included in these sections which provide the data used to calculate the mass balance.

During the mass balance discussions, terms are introduced which require definition. These definitions are provided as follows:

- Input solids include the solids initially present in the sample plus those solids introduced by caustic addition.
- Output solids are the final product solids and the RCC samples taken during the tests.
- Input water includes the water initially present in the sample and the water contributed by the addition of caustic.
- Output water consists of the volume of product water obtained after the cold wash extraction.

#### 4.2.4.1 Solids

Closure of the solids was very good, ranging from 92 to 99 percent (Table 16). Since the values used to determine closure were simple weights rather than analytical results, the mass balance was not compromised by errors associated with analytical methods. Small quantities of solids deposited in the vessels and containers used during the treatability study and found in the rag layer resulting from the cold wash were not accounted for in the mass balance.

**Table 16. Battelle Data - Solid Mass Balance**

	Buffalo River	Saginaw River	Grand Calumet River
<u>Input</u>			
Total Feed, g	900	899.7	1399.5
H <sub>2</sub> O, %	41.96	23.98	57.04
Total Feed Solids, g (dry)	522.4	682.6	601.2
Caustic (d=1.53)	4.1	4.1	12.6
Total Input, g (dry)	526.5	686.7	610.8
<u>Output</u>			
RCC Sample Cold Wash, g	11.2	30.6	6.9
RCC Sample 1st Hot Wash, g	16.3	23.4	7.1
RCC Sample 2nd Hot Wash, g	29.2	21.3	12.3
Final Dry Solids, g	461	606	485
Total Final Solids Output, g (dry)	517.7	681.3	511.1
Recovery, %	98.3	99.2	91.9

#### 4.2.4.2 Water

Closure of the water mass balance ranged from 67.5 to 77.9 percent (Table 17). Water lost to evaporation and water remaining in the rag layer and in the solids after the product water was decanted from the reactor vessel following the cold wash (i.e., beyond that directly associated with the triethylamine) did not contribute to the output water recovered. Because the majority of the water was removed from the raw sediments during the cold wash, closure calculations are based on data obtained during the cold wash only. The closed loop of the full-scale system would probably improve closure results.

**Table 17. Battelle Data - Water Mass Balance**

	Buffalo River	Saginaw River	Grand Calumet River
Total Feed, g	900	900	1399.5
Water, %	41.96	23.98	57.04
Total Feed Water, g	377.6	215.8	798.3
Caustic Water, g	4.1	4.1	9.6
Total Input Water, g	381.7	219.9	807.9
Water Recovered, g	262.8	174.9	609.8
Net minus 2% TEA, g	257.5	171.4	597.6
Recovery, %	67.5	77.9	74.0

**4.2.4.3 Oil**

The amount of oil initially present in the raw sediment and the amount of oil produced by the tests were used to determine the oil mass balance. The amount of oil present in either the treated solids or water is known from past tests to be insignificant and, therefore, was not accounted for. In order to retrieve the residual oil from the distillation flask, triethylamine was added to the oil so it could be poured. Battelle later determined the percentage of oil present in the resulting solutions (6.3 percent for the Buffalo River solution, 9.2 percent for the Saginaw River solution, and 60.0 percent for the Grand Calumet River solution) and compensated for these percentages when reporting final data. The need to compensate for these oil determinations possibly introduced error into the calculation of the oil mass balance. The calculated closures ranged from 69 to 192 percent as shown in Table 18.

**Table 18. Battelle Data - Oil Mass Balance**

	Buffalo River	Saginaw River	Grand Calumet River
Feed Input, g	900	899.7	1399.5
Oil and Grease, %	0.24	0.14	3.22
Input Oil, g	2.16	1.22	45.1
Final Oil & TEA, g	55.9	24.4	51.6
Oil, %	6.3	9.2	60.0
Final Oil, g	3.52	2.34	31.0
Recovery, %	163	192	68.7

#### 4.2.4.4 PCBs

The PCB mass balance was calculated using the amount of PCBs found in the feed, product oil (with triethylamine), and product solids. The contribution of the PCBs found in the product water was negligible and therefore not included in the mass balance calculations.

The closures of the PCBs were good, ranging from 80 to 129 percent (Table 19). When calculating these closures, it was assumed that the concentrations of the PCBs in the samples taken by RCC during the treatability study were the same as those found in the final products. In reality, these concentrations should be higher, since they were removed during earlier extraction stages. The need to compensate for the excess triethylamine present in the oil extract solutions may have introduced errors to the determination of the PCB concentrations found in the product oils. These errors could have subsequently been conveyed to the PCB mass balance closures.

**Table 19. Battelle Data - PCB Mass Balance**

	Buffalo River	Saginaw River	Grand Calumet River
Feed Input, g	900	900	1399.5
Solids, %	58.0	76.0	43.0
Dry Solids, %	522.4	684.2	601.2
PCBs, ug/kg	325	21865	15003
PCBs Input, mg	0.17	15.0	9.02
<b>OUTPUT</b>			
<u>Oil</u>			
TEA & Oil wt., gm	55.9	25.4	51.6
PCBs Conc., ug/L	2702	349,109	117,156
d of TEA & Oil, gm/L	690	752	730
PCBs Output wt., mg	0.22	11.8	8.28
<u>Solids</u>			
Dry Solids wt., gm	522.7	681.3	511.1
PCBs in Solids, ug/kg	ND	235	440
PCBs wt., mg	0.0	0.16	0.23
Total Output PCBs, mg	0.22	12.0	8.51
Recovery, %	129	80	94

ND = Not Detected

#### 4.2.4.5 PAHs

The closures of the PAHs were calculated using the amount of PAHs found in the feed, product oils (with triethylamine), and product solids. The contribution of the PAHs found in the product water was negligible and therefore not included in the mass balance calculations.

The closures of the PAHs were good (Table 20), ranging from 90 to 111 percent for Buffalo River and Grand Calumet River sediments. Saginaw River sediments, however, realized a closure of 240 percent. This may be attributed to the low concentrations of PAHs initially present in the sediment and the large errors associated with contaminant concentrations close to analytical detection limits.

**Table 20. Battelle Data - PAH Mass Balance**

	Buffalo River	Saginaw River	Grand Calumet River
Feed Input, g	900	900	1399.5
Solids, %	58.0	76.0	43.0
Dry Solids, %	522.4	684.2	601.2
PAHs, ug/kg	9,900	2,700	230,000
PAHs Input, mg	5.17	1.85	138
<b>OUTPUT</b>			
<u>Oil</u>			
TEA & Oil wt., g	55.9	25.4	51.6
PAHs Conc., ug/L	54,864	112,070	1,886,715
d of TEA & Oil, g/L	690	752	730
PAHs Output wt., mg	4.44	3.79	134
<u>Solids</u>			
Dry Solids wt., g	522.7	681.3	511.1
PAHs in Solids, ug/kg	370	950	37,100
PAHs wt., mg	0.19	0.65	19
Total Output PAHs, mg	4.63	4.44	153
Recovery, %	90	240	111

### 4.3 Summary of Vendor Results

The analytical results and the extraction efficiencies and mass balances performed by RCC match well with the analytical results provided by Battelle and the extraction efficiencies and mass balance calculated by SAIC. The RCC data were used to make comparisons with the results obtained and to help interpret data. Samples of the feed material and the treated solids produced using the B.E.S.T.® Solvent Extraction Process were analyzed by RCC for residual PCB contamination. The data from these analyses are presented in Table 21.

**Table 21. RCC Data - PCB Summary**

Sample (dry Basis)	Feed PCB Content, mg/kg (dry basis)	Treated Solids PCB Content, mg/kg (dry basis)	PCB Removal Efficiency (%)
Buffalo River	0.60	<0.03	>95
Saginaw River	21	0.18	99
Grand Calumet River	22	0.23	99

Feed material and treated solids were also analyzed for residual PAH and for metals concentrations. Residual PAH concentrations of <0.2 mg/kg per compound were found in the treated solids produced by treating the Buffalo River and Saginaw River sediments. Treated solids with PAH concentrations ranging from <1 to <3 mg/kg per compound were obtained for the Grand Calumet River sediments. In tests done by RCC, all of the treated solids passed the TCLP Toxicity Test for the leaching of metals.

RCC performed a mass balance of solids, water, oil, and PCBs using their own data. Table 22 summarizes the results of this mass balance.

**Table 22. RCC Data - Mass Balance Summary (Recovery %)**

Sample	Solids	Oil	Water	PCBs
Buffalo River	97	112	70	70
Saginaw River	98	137	82	280
Grand Calumet River	86	97	75	64

#### **4.4 Quality Assurance/Quality Control**

The conclusions and the limitations of data obtained during the evaluation of RCC's B.E.S.T.® Process are summarized in the following paragraphs.

Upon review of all sample data and associated QC results, the data generated for the B.E.S.T.® treatability study have been determined to be of acceptable quality. In general, QC results for accuracy and precision were good and can be used to support technology removal efficiency results.

In some cases, the demonstration of removal efficiency for PAHs and PCBs may be limited if relatively small amounts of these compounds are present in the untreated sediments. If minimal amounts are present, then detection limits become a factor. Removal efficiency demonstration may be limited by the sensitivity of the analytical methods.

Refer to Appendix F for the complete analysis related to Quality Assurance/Quality Control.

## APPENDIX A

### B.E.S.T.<sup>®</sup> BENCH-SCALE TREATABILITY TEST REPORT

Great Lakes National Program Office  
Buffalo River, Saginaw Bay and Indiana Harbor Sites

#### I. INTRODUCTION

##### SUMMARY

A bench-scale treatability test of the B.E.S.T. solvent extraction process was conducted on three polychlorinated biphenyl (PCB) contaminated sediment samples. One sample was received from each of the sites, Buffalo River, Saginaw Bay and Indiana Harbor. A summary of the bench-scale treatability test results follows:

#### BENCH SCALE TREATABILITY TESTS RESULTS

<u>Sample</u> (dry basis)	<u>Feed PCB</u> <u>Content, mg/kg</u> (dry basis)	<u>Product Solids</u> <u>PCB Content, mg/kg</u>	<u>PCB Removal</u> <u>Efficiency, %</u>
Buffalo River	0.60	< 0.03	> 95
Saginaw Bay	21	0.18	99
Indiana Harbor	22	0.23	99

As can be seen from the data above, the PCB residuals of the treated solids (Product Solids) varied from < 0.03 mg/kg to 0.23 mg/kg, yielding PCB removal efficiencies of > 95 to 99%. Individual, residual PAH concentrations in the treated solids were < 0.2 mg/kg for the Buffalo River and Saginaw Bay samples and ranged from < 1 to < 3 mg/kg for the Indiana Harbor sample.

All of the treated solids readily passed the TCLP Toxicity Test for the leaching of metals.

## THE B.E.S.T. SOLVENT EXTRACTION PROCESS

The B.E.S.T. process is a patented solvent extraction technology using triethylamine as the solvent. Triethylamine is an aliphatic amine that is produced by reacting ethyl alcohol and ammonia.

Triethylamine is an excellent solvent for treating hazardous wastes because it exhibits several characteristics that enhance its use in the solvent extraction system. These characteristics include:

- A high vapor pressure; therefore, the solvent can be easily recovered from the extract solution (oil, water, and solvent) via steam stripping.
- Formation of a low boiling temperature azeotrope with water, allowing the solvent to be recovered from the oil to very low residual levels (typically less than 100 ppm).
- A low heat of vaporization (1/7 of water), allowing solvent to be recovered from the treated solids with very low energy input.
- Triethylamine is alkaline (pH=10); therefore, some heavy metals are converted to the hydroxide form, precipitate and exit the system with the treated solids.
- Triethylamine readily biodegrades. Data available in EPA document EPA Data ORD USEPA Washington, D.C. 20460, Feb. 1983 (reprint) Manual, Volume 1 600/2-82-001a. shows that a level of 200 ppm triethylamine in water was degraded completely within 11 hours by the common soil bacteria *aerobacter*.

A block diagram of the B.E.S.T. process is presented in Figure 1. The first extraction of the contaminated feed is conducted at low temperatures (about 40 degrees F). At this temperature, triethylamine is soluble with water. Therefore, the extract solution contains most of the water in the feed sample. If the first extract solution contains sufficient water to allow a phase separation of the solvent and water, the extract is heated to a temperature above the miscibility limit (130 degrees F). At this temperature, the extract solution separates into two distinct phases, a solvent/oil phase and a water phase. The two phases are separated by gravity and decanted. The extract solution from the subsequent stages is combined with the decanted solvent/oil phase from the first extraction stage. The solvent is recovered by steam stripping and evaporation.

Triethylamine is removed from the treated solids by indirect steam heating. A small amount of steam may be added directly to the dryer vessel to provide the water required to form the low boiling temperature azeotrope. Residual solvent biodegrades readily, allowing the treated solids to be used as backfill at the site in some cases.

The B.E.S.T. process operates near ambient pressure and temperature and at a alkaline pH. Temperatures of the liquid streams within the the unit vary from about 40 to 170 degrees F, and elevated pressures are not required. This gives the B.E.S.T. process the advantage that it can use standard off-the-shelf processing equipment.

**B.E.S.T. PROCESS CONCEPT**

**Extraction**

**Solvent Recovery**

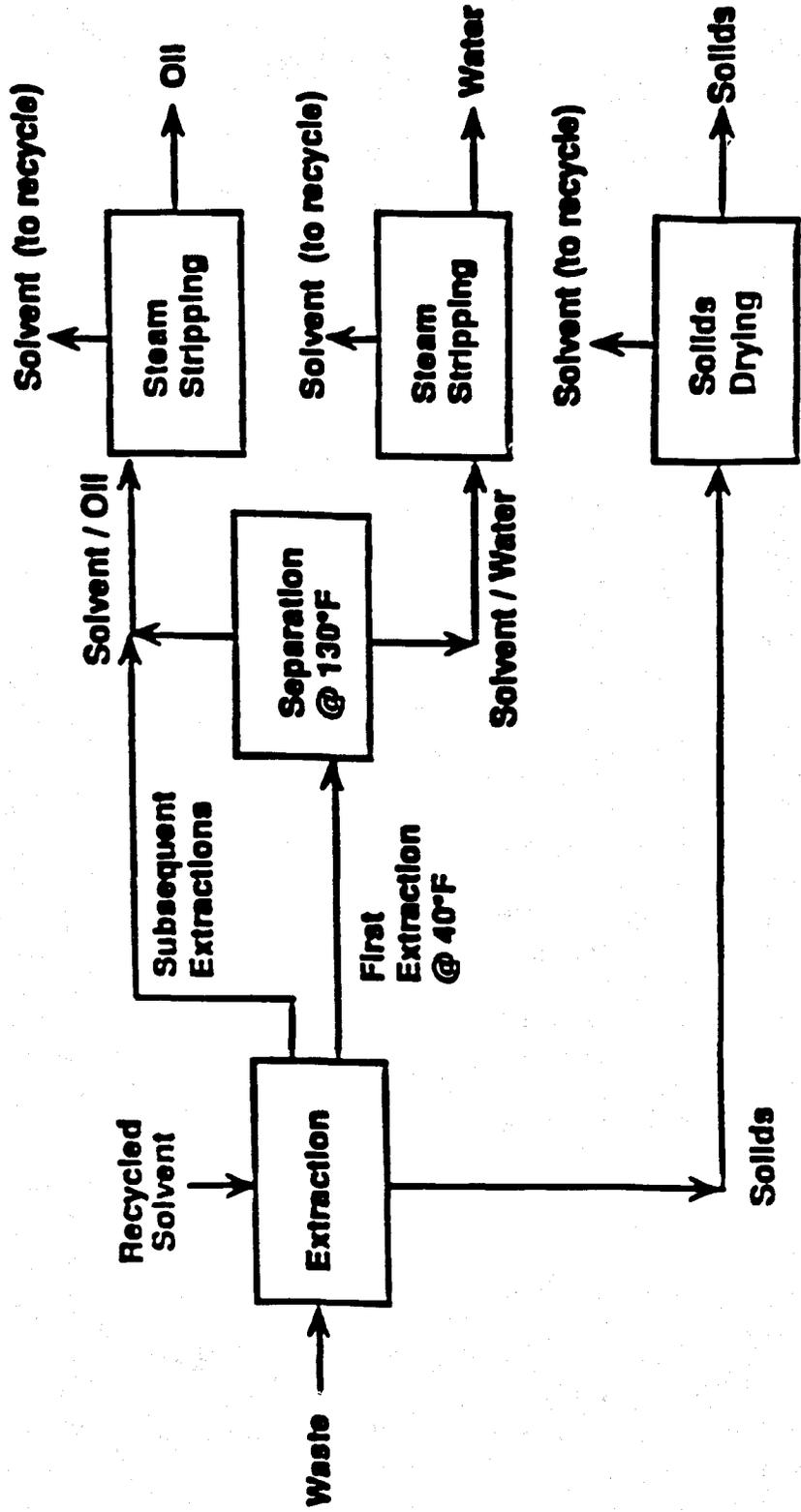


Figure 1

## AIR EMISSIONS AND ABATEMENT

The B.E.S.T. process uses one vent to the atmosphere. The vent provides pressure equalization for the nitrogen blanketing system and a purge for noncondensable gases from process condensers. RCC uses a refrigerated condenser and an auxiliary water scrubber system to reduce solvent emissions from the vent.

During a performance test in February 1987 at the General Refining Superfund Site cleanup, a third party reported the following emissions from the B.E.S.T. process vent at a time when the auxiliary water scrubber was not in operation:

	<u>Emission Rate, lb/hr</u>
Benzene	0.00114
Mercury	< 0.00000043
Toluene	0.000614
Triethylamine	0.0954
Xylene	0.000884

RCC expects air emissions from future operations to be similar to these results. The use of the auxiliary water scrubber will lower the triethylamine release rate even further. RCC now utilizes activated carbon filters on the single vent line to achieve zero emissions of triethylamine.

## EQUIPMENT DESCRIPTION

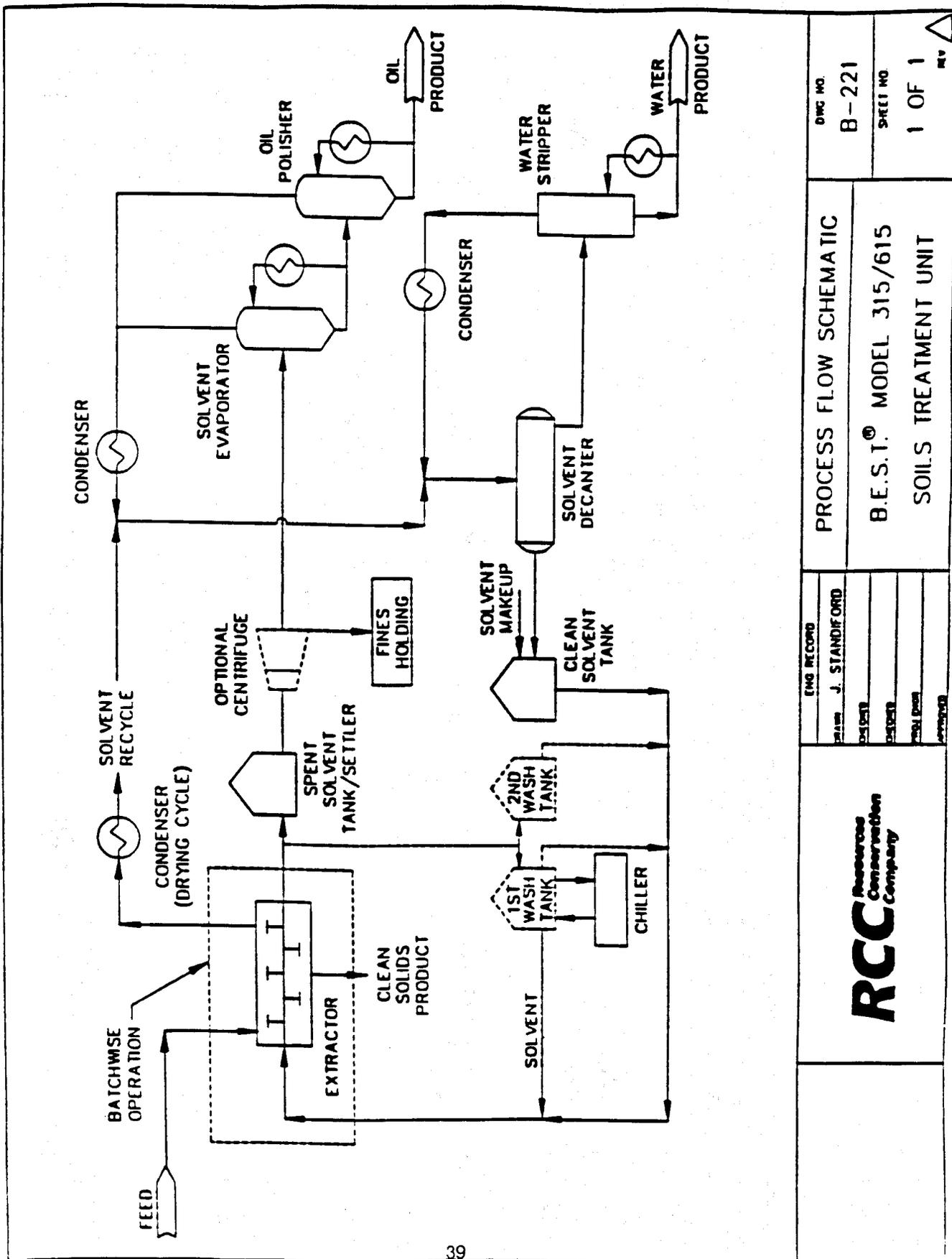
RCC proposes using a B.E.S.T. Model 615 unit to treat the PCB-contaminated material at this site. The B.E.S.T. Model 615 unit has a design capacity of approximately 200 - 300 tons of feed per day. A flow schematic for the B.E.S.T. Model is presented in Figure 2.

The B.E.S.T. Model 615 uses an extractor/dryer vessel to extract and dry the PCB-contaminated materials. The extractor/dryer is a horizontal, steam-jacketed vessel that allows for solvent contacting, mixing, solids/solvent separation, solids drying, and solids conditioning in one vessel. The extractor/dryer vessel is an off-the-shelf assembly that has a long history of reliable performance in a wide range of process industry applications.

Contaminated materials are excavated from the site and screened to one inch maximum dimension. The screened material is then loaded into top-loading, bottom-discharge hoppers. An overhead crane facilitates the positioning and lowering of the loaded hopper onto the loading port of the extractor/dryer unit. The flow of material through the extractor/dryer system is shown in Figure 3. Treated solids are discharged into hoppers and transported to a holding area.

Figure 4 provides the standard Site Plan for RCC's B.E.S.T. Model 615.

Figure 2



	ENG RECORD DRAWN BY J. STANDIFORD DESIGNED BY CHECKED BY APPROVED BY	DWG NO. B-221
	PROCESS FLOW SCHEMATIC B.E.S.T.® MODEL 315/615 SOILS TREATMENT UNIT	SHEET NO. 1 OF 1

# B.E.S.T.<sup>®</sup> PROCESS STEPS BATTERY LIMITS OPERATION

**RCC** Resources  
Conservation  
Company

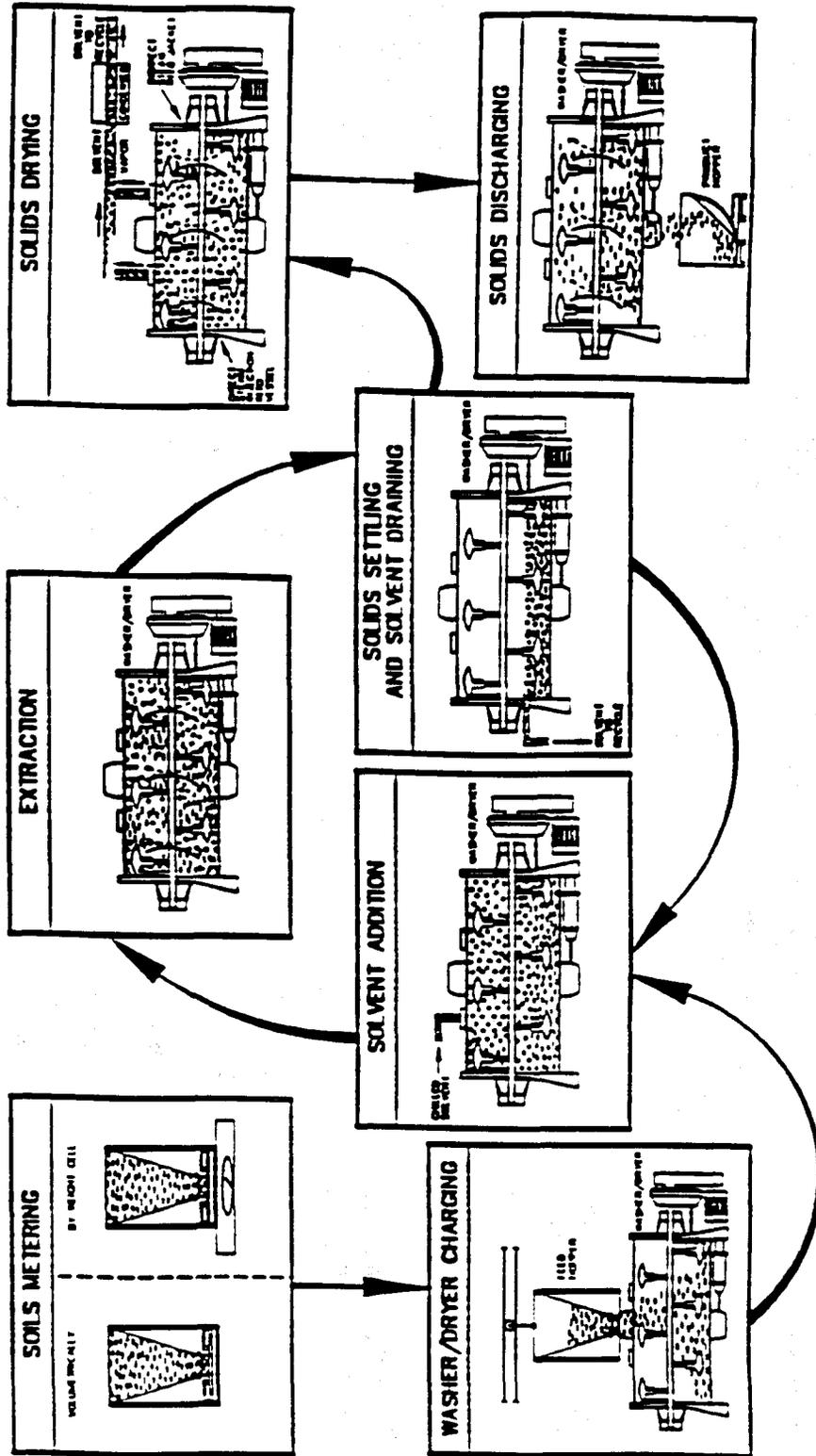


Figure 3



## **BENCH-SCALE TREATABILITY TEST DATA CORRELATION TO FULL-SCALE PERFORMANCE**

In order to evaluate each potential application for the B.E.S.T. process, RCC has developed a low cost bench-scale treatability test protocol that provides data that closely simulates full-scale system performance. The bench-scale treatability test data allows RCC to evaluate the feasibility of the process on a particular sample and to estimate treatment costs.

The reliability of the bench-scale treatability tests to predict full-scale performance has been verified by the US EPA report Evaluation of the B.E.S.T. Solvent Extraction Sludge Treatment Technology - Twenty-Four Hour Test, by Enviresponse, Inc., under EPA Contract 68-03-3255. A quote from this report evaluating the B.E.S.T. process states:

"Resources Conservation Company has conducted many laboratory tests and developed correlations to which data from full-scale operations, such as the General Refining site, can be compared."

Figures 5 and 6 present data from two separate bench-scale treatability tests and full-scale operating performance data at the General Refining, Inc., Superfund site, as collected by an EPA contractor. This data demonstrates a close correlation between bench-scale treatability test data and full-scale operating data.

Bench-scale treatability testing provides valuable information about the use of the B.E.S.T. process at full-scale including:

- The PCB removal efficiency from the sample.
- Solids separation requirements for full-scale operation.
- The separation efficiency of water from the water/solvent/oil solution by decantation.
- General information on the partitioning of metals and organic compounds in the oil, water, and solids products.
- Full-scale operating parameters to develop treatment costs.

GENERAL REFINING SITE  
PCB CONCENTRATIONS IN RAW SLUDGE & PRODUCT FRACTIONS  
(ppm)

LAB SCALE TESTING (1986) FULL SCALE PROCESSING  
TEST "A" TEST "B" FEB. 26-27, 1987

RAW SLUDGE (DRY BASIS) mg/kg	14.	12.	13.5
PRODUCT SOLIDS mg/kg	0.02	0.14	<0.13
PRODUCT WATER mg/L	<0.01	<0.01	<0.005
%EXTRACTION EFFICIENCY	99.9	98.8	>99.0

Figure 5

**COMPARISON OF BENCH SCALE TO FULL SCALE  
PHASE SEPARATION PERFORMANCE  
FOR  
GENERAL REFINING SITE SLUDGE**

	Bench Scale			Full Scale		
	Raw Sludge	Separated Phase Fractions	Raw Sludge	Separated Phase Fractions	Raw Sludge	Separated Phase Fractions
	Oil	Water	Oil	Water	Oil	Water
	Solids	Solids	Solids	Solids	Solids	Solids
Oil %	36	.017	27	0.0033	99.	0.81
Water %	56	N/A	66	>99.	0.88	<0.5
Solids %	8	N/A	7	0.81	>98.	>98.

N/A Not Available  
BS&W = 2.8%

Figure 6

## **BENCH-SCALE TREATABILITY TEST DOCUMENTATION**

The documentation of the testing can be separated into three distinct categories. The following summarizes the procedures used for each step of the treatability process:

1. When the samples were received in the laboratory, the shipment was checked for correctness of accompanying paper work, including Chain of Custody. The information was recorded both in a hardbound sample logbook and on a computer system that has been specifically designed by RCC for use in tracking samples. The samples were issued a discrete laboratory sample number, and a test request form was completed. The samples were kept in a refrigerator under controlled and documented temperature prior to any lab analysis or the treatability study. Chain of Custody records and other information received with the samples are kept as part of the project file.
2. The bench-scale treatability testing was conducted in accordance with the test plan, and all records and observations taken during the simulation of the process were recorded in laboratory notebooks. The laboratory notebooks are the property of RCC, and each analyst and engineer has been issued a notebook. The notebooks are retained by RCC as permanent record of raw data collection.
3. Samples that were collected during the bench-scale test, including samples internal to the process, were submitted to the RCC analytical chemistry laboratory for further analysis. Each sample collected was issued a discrete laboratory number. An analysis request form was completed. The samples were analyzed in accordance with RCC's Laboratory Quality Management Plan and reviewed for correctness prior to issuance. A file is maintained to permanently store the accumulated test results from completion of the analytical testing.

The bench-scale treatability test plan is provided in Attachment 1. PCB chromatograms are given in Attachment 2. PAH chromatograms are given in Attachment 3.

## **II. BENCH-SCALE TREATABILITY TESTING**

### **SAMPLE PREPARATION**

The contaminated samples from the Buffalo River, Saginaw Bay and Indiana Harbor Sites arrived at RCC's laboratory in July, 1991. The samples were labeled B-US-RCC, S-US-RCC and I-US-RCC, respectively. All samples were gray-colored sediments with very little debris present. Each of the three samples contained free standing water. This water was decanted prior to conducting feed analyses and was proportionally recombined prior to any analysis and bench testing. This effort was taken since it was very difficult to homogenize the samples with the free standing water present.

Bench-scale testing requires material greater than 1/4 inch be removed. There was no material greater than 1/4 inch in any of the three samples received. Therefore, the samples were not screened.

## FEED COMPOSITIONAL ANALYSIS

The feed was analyzed for percent oil, water, solids and metals per the following methods:

- The oil & grease content was determined as per Standard Methods for the Examination of Water and Wastewater, 16th Edition, Method 503D, with two exceptions: the extraction time was extended from 4 to 16 hours, and methylene chloride ( $\text{MeCl}_2$ ) was substituted for Freon based on RCC experience that  $\text{MeCl}_2$  is a better solvent for oils and greases.
- The water content was determined by weight loss at 70 degrees C.
- The particulate solids content was determined by rinsing a known quantity of feed through a Whatman GF/C filter under vacuum with acetone followed by  $\text{MeCl}_2$ . The residue was then dried and weighed. Since the particulate solids content was by far the largest component of all of the feeds, the percent solids values below are by difference.
- The PCB concentration was determined per EPA Publication SW846 Test Methods for Evaluating Solid Waste, Method 8080. The sample extraction method was by Soxhlet extraction with 1:1 acetone:hexane for 16 hours. The PCBs were quantitated as Aroclor 1242.
- The Polynuclear Aromatic Hydrocarbon (PAH) concentrations were determined per EPA Publication SW846, Method 8100 (1:1 Acetone:Hexane extraction solvent).
- The metals composition (except for Mercury) was determined by nitric acid digestion after ashing at 550 degrees C, followed by ICP analysis (EPA SW846, Method 6010).
- Mercury concentration was determined by the Cold Vapor Technique, Method 303F, of Standard Methods for the Examination of Water and Wastewater.
- Loss on ignition was determined by heating a sample from 105 to 550 degrees C which is a measure of the total organic content.

The results of these analyses on a wet basis were as follows:

**Feed Compositional Analysis**

(wet basis unless noted)

<u>Analyte</u>	<u>Sample Results</u>		
	<u>Buffalo River</u>	<u>Saginaw Bay</u>	<u>Indiana Harbor</u>
Oil & Grease (by MeCl <sub>2</sub> ), %	0.48	0.36	2.4
Water, %	41.	23.	56.
Solids, %	59.	77.	42.
Loss on Ignition, %	4.8	2.5	26.
PCBs, mg/kg, dry basis	0.6	21	22

Individual feed PAH concentrations for the Buffalo River sample ranged from <0.5 to 6 mg/kg, for the Saginaw Bay sample they were <0.4 to <3 mg/kg and for the Indiana Harbor sample they were <8 to <60 mg/kg. These results are presented on page 17 and 18.

The heavy metals composition of each feed was as follows:

**Feed Metals Composition, mg/kg**

(As received basis)

<u>Analyte</u>	<u>Buffalo River</u>	<u>Saginaw Bay</u>	<u>Indiana Harbor</u>
Antimony	< 12.	< 15.	< 15.
Arsenic	< 30.	< 40.	< 35.
Barium	34.	32.	76.
Cadmium	< 2.	< 3.	6.4
Chromium	14.	62.	156.
Copper	16.	37.	96.
Lead	28.	37.	290.
Mercury	0.38	0.10	0.72
Nickel	9.7	36.	29.
Selenium	< 20.	< 25.	< 20.
Silver	< 1.	< 1.	< 0.7
Sodium	115.	150.	300.
Zinc	52.	120.	1,340.

## TRIETHYLAMINE COMPATIBILITY TEST

Triethylamine is a compound with a unique chemical structure. The geometry of the structure is tetrahedral, meaning that the nitrogen atom is at the center of a three-sided pyramid. The four points of the pyramid structure are occupied by three ethyl functional groups and one electron cloud. This structure gives triethylamine dual polarity characteristics. The ethyl groups are essentially nonpolar, the electron cloud is polar. Although triethylamine is a very stable solvent, there is a remote possibility that the electron pair can react with certain types of materials. In order to determine if this will occur with a sample, a compatibility test is performed. This involves mixing of the sample with triethylamine and making observations as to the heat of solution and any other visual signs of reaction.

When each feed sample was mixed with cold triethylamine, no visible sign of adverse reaction was observed, and the heat of solution was in a normal range. The triethylamine was observed to darken upon mixing, indicating that extraction of the organic compounds was occurring.

Based on the favorable results of this preliminary test, it was decided that the B.E.S.T. bench-scale treatability test should proceed.

## FEED pH ADJUSTMENT

Triethylamine can be ionized at low pH to triethylammonium salts that cannot be removed from the products. The alkaline nature of triethylamine will buffer the pH of the sample to a pH of around 9. The solvent spent in the pH buffering will be lost. In order to efficiently recover the triethylamine from the separated phase fraction products, the pH of the sample is adjusted to about 11 with caustic soda.

A 5-gram portion of each feed sample was slurried with deionized water. The pH of this mixture indicated that caustic would need to be added to each sample. Incremental portions of caustic soda (NaOH) were added to bring the pH to 11. The amount of caustic that was required to perform this pH adjustment and the original sample pH is summarized below:

### Sample pH and Caustic Dose

<u>Sample</u>	<u>pH</u>	<u>Caustic Dose</u> <u>(mls 50% NaOH per kg)</u>
Buffalo River	7.6	6.0
Saginaw Bay	8.1	6.0
Indiana Harbor	7.5	9.0

## **SAMPLE EXTRACTION/PRODUCT SOLIDS**

A portion of the Buffalo River, Saginaw Bay and Indiana Harbor samples was prechilled by placing each in a 4-liter resin kettle, immersed in a temperature controlled water bath set at 0.5 degree C. Each sample pH was adjusted by adding caustic soda at the same time that chilled triethylamine was added. Mixing was performed by an air-driven prop mixer in the same 4-liter resin kettle immersed in the cooling bath.

As expected, the solvent became colored for all three samples, indicating extraction of organic compounds was occurring. After mixing, the solvent/oil/water liquid extract was separated from the solids by centrifugation. The liquid extract was temporarily set aside for testing as discussed later under DECANTATION OF WATER.

Two more extraction stages were performed on the solids, for a total of three extraction stages. No additional caustic was added for the subsequent extraction stages. A sample of the Product Solids was collected for analysis as follows:

- The oil & grease content was determined as per Standard Methods for the Examination of Water and Wastewater, 16th Edition, Method 503D, with two exceptions: the extraction time was extended from 4 to 16 hours, and methylene chloride ( $\text{MeCl}_2$ ) was substituted for Freon based on RCC experience that  $\text{MeCl}_2$  is a better solvent for oils and greases.
- Loss on ignition was determined by heating a sample to 550 degrees C for 3 hours.
- The PCB concentration was determined per EPA Publication SW846 Test Methods for Evaluating Solid Waste, Method 8080. The sample extraction method was by Soxhlet extraction with 1:1 Acetone:Hexane for 16 hours. The PCBs were quantitated as Aroclor 1242.
- The metals composition was determined by aqua regia digestion, followed by ICP analysis (EPA SW846, Method 6010).
- The triethylamine content was determined by shaker bath water extraction and packed column gas chromatography with a flame ionization detector.
- The pH was determined by measuring the pH of a slurry of 5 grams of sample and 50 mls of deionized water. The slurry was tested by pH probe after mixing overnight.
- The Polynuclear Aromatic Hydrocarbon (PAH) concentrations were determined per EPA Publication SW846, Method 8100 (1:1 Acetone:Hexane extraction solvent).
- Mercury concentration was determined by the Cold Vapor Technique, Method 303F, of Standard Methods for the Examination of Waste and Wastewater.

PCB analytical results of all of the solid samples were as follows:

**PCB Analysis Summary, mg/kg**  
(all data dry basis)

	<b><u>Buffalo River</u></b>	<b><u>Saginaw Bay</u></b>	<b><u>Indiana Harbor</u></b>
Feed	0.60	21	22
Product Solids	< 0.03	0.18	0.23

Total polynuclear hydrocarbon (PAH) analytical results of the feeds and product solids from the three samples are given on the following two pages. Due to a large number of interfering compounds eluting at or near the retention times of the analytes of interest, it was not possible to report lower detection limits.

**PAH Summary for Buffalo River**  
(mg/kg)

	<u>Feed</u>	<u>Product Solids</u>
Naphthalene	< 0.5	< 0.2
2-Methylnaphthalene	< 0.5	< 0.2
Acenaphthylene	< 0.5	< 0.2
Acenaphthene	< 0.5	< 0.2
Dibenzofuran	< 1.	< 0.2
Fluorene	< 0.5	< 0.2
Phenanthrene	< 2.	< 0.2
Anthracene	< 2.	< 0.2
Fluoranthene	< 5.	< 0.2
Pyrene	< 5.	< 0.2
Benzo(a)anthracene	< 6.	< 0.2
Chrysene	< 4.	< 0.2
Benzo(b)fluoranthene	< 6.	< 0.2
Benzo(k)fluoranthene	< 3.	< 0.2
Benzo(a)pyrene	< 6.	< 0.2
Indeno(1,2,3-cd)pyrene	< 0.5	< 0.2
Dibenzo(a,h)anthracene	< 0.5	< 0.2
Benzo(g,h,i)perylene	< 0.5	< 0.2

**PAH Summary for Saginaw Bay**  
(mg/kg)

	<u>Feed</u>	<u>Product Solids</u>
Naphthalene	< 0.4	< 0.2
2-Methylnaphthalene	< 0.4	< 0.2
Acenaphthylene	< 0.4	< 0.2
Acenaphthene	< 0.4	< 0.2
Dibenzofuran	< 0.4	< 0.2
Fluorene	< 0.4	< 0.2
Phenanthrene	< 0.8	< 0.2
Anthracene	< 1.	< 0.2
Fluoranthene	< 2.	< 0.2
Pyrene	< 1.	< 0.2
Benzo(a)anthracene	< 2.	< 0.2
Chrysene	< 3.	< 0.2
Benzo(b)fluoranthene	< 0.4	< 0.2
Benzo(k)fluoranthene	< 0.4	< 0.2
Benzo(a)pyrene	< 0.4	< 0.2
Indeno(1,2,3-cd)pyrene	< 0.4	< 0.2
Dibenzo(a,h)anthracene	< 0.4	< 0.2
Benzo(g,h,i)perylene	< 0.4	< 0.2

**PAH Summary for Indiana Harbor**

(mg/kg)

	<u>Feed</u>	<u>Product Solids</u>
Naphthalene	< 6.	< 3.
2-Methylnaphthalene	< 6.	< 3.
Acenaphthylene	< 4.	< 1.
Acenaphthene	< 7.	< 1.
Dibenzofuran	< 15.	< 3.
Fluorene	< 12.	< 1.
Phenanthrene	< 20.	< 3.
Anthracene	< 12.	< 1.
Fluoranthene	< 45.	< 2.
Pyrene	< 48.	< 2.
Benzo(a)anthracene	< 40.	< 2.
Chrysene	< 38.	< 3.
Benzo(b)fluoranthene	< 25.	< 2.
Benzo(k)fluoranthene	< 28.	< 1.
Benzo(a)pyrene	< 33.	< 1.
Indeno(1,2,3-cd)pyrene	< 13.	< 1.
Dibenzo(a,h)anthracene	< 10.	< 1.
Benzo(g,h,i)perylene	< 13.	< 1.

Additional product solids analyses, all on a dry basis since the product solids were dried in-process, follows:

**Product Solids Analysis**

(three extraction stages)

<u>Analyte</u>	<u>Sample Results</u>		
	<u>Buffalo River</u>	<u>Saginaw Bay</u>	<u>Indiana Harbor</u>
PCBs, mg/kg	< 0.03	0.18	0.23
Oil & Grease (by MeCl <sub>2</sub> ), %	0.22	0.15	0.52
Triethylamine, mg/kg	37.	20.	28.
Loss on Ignition, %	4.0	2.1	20.

PCB removal efficiency is determined by comparing the amount of PCBs in the feed to the amount remaining in the environment after treatment. The fraction of PCBs remaining in the environment is calculated by dividing the PCB content of the product solids by the PCB content of the feed, on a dry basis. An example of the calculation, the Saginaw Bay sample, follows:

**Saginaw Bay Sample  
PCB Removal Efficiency Calculation**

.....

$$\begin{aligned} \text{Fraction of PCBs remaining in environment} &= \frac{\text{Product solids PCB Content (dry basis)}}{\text{Feed PCB Content (dry basis)}} \\ &= \frac{0.18 \text{ mg/kg}}{20.5 \text{ mg/kg}} = 0.00878 \end{aligned}$$

.....

$$\begin{aligned} \% \text{ Removal from environment} &= 100 \cdot (1 - \text{fraction of PCBs remaining in environment}) \\ &= 100 \cdot (1 - 0.00878) \\ &= 99.1 \% \end{aligned}$$

The reduction in the PCB content and the corresponding removal efficiency of PCBs from the environment is summarized below for all of the samples:

**Total PCB Removal Summary**

<u>Sample</u>	<u>PCBs in Feed, mg/kg</u> (dry basis)	<u>PCBs in Product Solids, mg/kg</u> (dry basis)	<u>Removal Efficiency, %</u>
Buffalo River	0.60	< 0.03	> 95
Saginaw Bay	21	0.18	99
Indiana Harbor	22	0.23	99

Total heavy metal analysis of the product solids was as follows:

**Product Solids**  
**Total Metals Analysis, (mg/kg)**

<b><u>Analyte</u></b>	<b><u>Buffalo River</u></b>	<b><u>Saginaw Bay</u></b>	<b><u>Indiana Harbor</u></b>
Antimony	< 20.	< 20.	< 20.
Arsenic	< 50.	< 50.	< 50.
Barium	73.	41.	180.
Cadmium	< 3.	< 5.	24.
Chromium	51.	93.	430.
Copper	62.	60.	270.
Lead	110.	56.	750.
Mercury	0.68	0.20	1.6
Nickel	32.	53.	88.
Selenium	< 30.	< 30.	< 30.
Silver	< 5.	< 1.	< 5.
Sodium	5,200.	3,900.	12,000.
Zinc	160.	170.	3,500.

## TOXICITY CHARACTERISTIC LEACHING PROCEDURE ANALYSIS ON PRODUCT SOLIDS

The product solids from each sample were extracted using the Toxicity Characteristic Leaching Procedure (TCLP) in accordance with Federal Register, March 29, 1990. Each TCLP leachate was analyzed for metals content. The results from this analysis were as follows:

### Product Solids TCLP Leachate Analysis, mg/l

<u>Analyte</u>	<u>Buffalo River</u>	<u>Saginaw Bay</u>	<u>Indiana Harbor</u>	<u>Regulatory Level, mg/l</u>
Arsenic	< 0.5	< 0.5	< 0.5	5
Barium	0.11	0.14	0.04	100
Cadmium	< 0.03	< 0.03	< 0.03	1
Chromium	< 0.05	0.15	0.07	5
Lead	< 0.1	< 0.1	< 0.1	5
Mercury	< 0.002	< 0.002	0.0024	0.2
Selenium	< 0.3	< 0.3	< 0.3	1
Silver	< 0.01	< 0.01	0.05	5

As can be seen from the above data, all product solids readily passed the TCLP test for metals.

## DECANTATION OF WATER

The solvent recovered from each first extraction stage was separated into its aqueous and organic components. Only the extract from the first extraction stage had a significant amount of water in solution, so only the water in the first stage extract is recovered. There are two methods available to recover the water in the first stage extract. Each method has its advantages. The method chosen largely depends on the water content of the feed.

In the first method of recovering the water, the triethylamine mixture is heated to about 140 degrees F. The water phase is separated from the triethylamine/oil phase by decantation. At the elevated temperature, water is no longer miscible in triethylamine and settles to the bottom of the mixture. The heated mixture is allowed to stand and separate for 30 minutes in a 4-liter separatory funnel. The separatory funnel is immersed into a clear tank into which water from a temperature controlled water bath is pumped. Excess water from the tank drains back into the water bath. The temperature controlled bath is set at 140 degrees F. This low energy method is preferable for high water content feeds.

Decantation (the first method) was used for both Buffalo River and Indiana Harbor. The separation does not give only TEA/oil and water phases due to the presence of oil and solids in the mixture. In between the two phases is a 'rag' layer or emulsion where any solids present tend to collect and create a region where the TEA/Oil/Water separations is not distinct. The smaller this rag layer is in comparison to the TEA/Oil and water phases, the better the separation. For the Buffalo River sample the rag layer was only 2.6% of the entire TEA/Oil/Water mixture. For the Indiana Harbor sample the rag layer was 3.1%. In both cases, the rag layer is a very small fraction of the whole mixture, therefore, the separation of TEA/Oil from water was good.

In the second method of recovering water, the water from the feed is separated from the oil by evaporation as opposed to decantation. When the triethylamine/oil/water first stage extract is evaporated, as described in section SOLVENT EVAPORATION/PRODUCT OIL, the water forms an azeotrope with the distilled triethylamine, leaving the oil behind. The water is then separated from the triethylamine of the condensed triethylamine/water azeotrope by decantation. The triethylamine/water recovered from the Solvent Evaporation/Product Oil step is heated to 140 degrees F, then poured into a 4-liter separatory funnel. Separation occurs immediately, so no temperature control system is required. This separation is highly effective because there is virtually no oil or solids in the condensed triethylamine/water that would hinder the separation of triethylamine from water by decantation. This method is preferable for low water content feeds where the extra energy cost to evaporate the water is small. This method yields a much purer water stream, usually avoiding the requirement for possible post-treatment of the water.

Evaporation (the second method) was used for the Saginaw Bay sample because of its low water content (< 25%). The aqueous phase separated from the organic phase, producing excellent results for each.

## PRODUCT WATER

Removal of residual triethylamine from each decant water was accomplished by heating the water on a hot plate while maintaining an elevated pH. The elevated pH is necessary to ensure that the majority of the triethylamine remains in the volatile molecular form. Triethylamine/water azeotrope boils at about 170 degrees F. When the triethylamine is removed, the water temperature increases to 212 degrees F. Analysis of each stripped water, labeled Product Water, was conducted as follows:

- The triethylamine content was determined by packed column gas chromatography with a flame ionization detector.
- There was insufficient sample for Total Petroleum Hydrocarbons, Oil & Grease, PCB or total metals analyses since the bulk of the water was sent to a third party lab.

Results of these analyses were as follows:

### Product Water Analysis, mg/l

<u>Analyte</u>	<u>Buffalo River</u>	<u>Saginaw Bay</u>	<u>Indiana Harbor</u>
Triethylamine	7.	10.	13.
pH	10.3	10.4	10.8

## SOLVENT EVAPORATION/PRODUCT OIL

Recovery of product oil (the organic compounds in the feed) is normally accomplished in two steps. First, the bulk of the triethylamine is recovered by distillation. This is done by boiling the triethylamine/oil mixture in a Buchi Rotovapor<sup>®</sup> apparatus. The oil remains in the boiling flask of the Rotovapor while the triethylamine is condensed as it evaporates and is collected separately. Second, any residual triethylamine is stripped from the oil by adding water to the hot oil in the boiling flask of the Rotovapor. Water is added to form the low boiling triethylamine/water azeotrope. This second step was not used for these samples due to the low oil content of the feeds. With a low oil content feed, the amount of oil recovered is so small that effective stripping is not possible. The oil from the sample was kept in a solution of triethylamine with only a fraction being completely dried of solvent. In this way, the oil remains homogeneous and can be poured out of the rotovapor flask. This is vital for the integrity of the analyses on the oil, including PCBs from which a PCB mass balance is determined. The analysis of each product oil follows:

- The metals composition was determined by nitric acid digestion after ashing at 550 degrees C, followed by ICP analysis (EPA SW846, Method 6010).
- The PCB concentration was determined by dilution of the oil in hexane, followed by EPA SW846 (Test Methods for Evaluating Solid Waste), Method 3620, sulfuric acid and/or Florisil column cleanup. The prepared sample was then analyzed by EPA SW846, Method 8080.

The oil in a triethylamine diluent was analyzed and the results converted to a pure oil (triethylamine free) basis. The results were as follows:

### Product Oil Analysis, dry basis

<u>Analyte</u>	<u>Buffalo River</u>	<u>Saginaw Bay</u>	<u>Indiana Harbor</u>
PCBs, mg/kg	4.0	1,600	160
Metals, mg/kg			
Antimony	< 15.	< 20.	< 4.
Arsenic	< 40.	< 50.	< 10.
Barium	3.	15.	2.
Cadmium	3.	4.	< 0.6
Chromium	5.	58.	50.
Copper	110.	3,800.	12.
Lead	20.	290.	6.
Nickel	20.	1,400.	7.
Selenium	< 25.	< 30.	< 6.
Silver	< 0.8	39.	0.2
Sodium	400.	22,000.	44.
Zinc	19.	100.	5.

### III. MASS BALANCES

The data gathered during the bench-scale treatability test provides the data required to calculate mass balances. The mass balances have been segregated into four groups: solids, oil, water, and PCBs.

#### SOLIDS MASS BALANCE

The mass balance for solids is a comparison of the solids input during the test to the solids recovered after the test. The mass of solids input during the test includes the solids portion of the feed extracted and the solids portion of caustic soda added. The solids portion of the feed extracted was calculated by multiplying the weight of feed extracted by the solids content as determined by analysis. The solids portion of the caustic soda added was calculated by multiplying the weight of the 50 percent NaOH solution added by 0.50.

The mass of the solids recovered from the test is equivalent to the sum of the product solids and samples taken for stage-by-stage assays. A summary of this data follows:

	<u>Solids Mass Balance</u>		
	<u>Buffalo River</u>	<u>Saginaw Bay</u>	<u>Indiana Harbor</u>
Total Feed Extracted, Wet Basis	900 g	900 g	1,400 g
Solids Portion of Feed	530 g	690 g	584 g
Solids Portion of Caustic	+ 4.1 g	+ 4.1 g	+ 9.5 g
	<hr/>	<hr/>	<hr/>
Total Calculated Solids Input	= 534 g	= 694 g	= 594 g
.....			
Weight of Product Solids Recovered	461 g	666 g	485 g
Weight of Solids Samples Recovered	+ 57 g	+ 75 g	+ 26 g
	<hr/>	<hr/>	<hr/>
Total Solids Recovered	= 518 g	= 681 g	= 511 g
.....			
Recovery, %	97	98	86

## OIL MASS BALANCE

The oil mass balance was computed using the same method used in calculating the solids mass balance. The oil & grease content of each feed was determined by extracting a sample of the feed with methylene chloride. This oil & grease content (by  $\text{MeCl}_2$ ) was multiplied by the weight of the feed input to determine the amount of oil input. The mass of oil recovered from the test was equivalent to the product oil recovered. The residual oil in the product solids and product water was negligible when calculating an oil mass balance.

The oil mass balances (based on methylene chloride) were as follows:

### Oil Mass Balance

<u>Sample</u>	<u>Calculated Oil Input</u>	<u>Equivalent Product Oil Recovered</u>	<u>% Recovery</u>
Buffalo River	4.30 g	4.83 g	112 %
Saginaw Bay	3.24 g	4.44 g	137 %
Indiana Harbor	33.6 g	32.5 g	97 %

Virtually all of the PCBs from the sample now reside in the product oil. For each sample, the weight of PCB contaminated material was reduced from 900 grams (1400 grams for the Indiana Harbor sample) to 3.4-30 grams corresponding to a 47-270 times reduction in mass.

## WATER MASS BALANCE

The water mass balance was computed similarly to the method used for solids. The mass of water input came from the water in the feed, plus the water introduced with the caustic. The water portion of each feed was calculated by multiplying the weight of the feed by the water content as determined by analysis. The water portion of the caustic input was calculated by multiplying the weight of the 50 percent NaOH solution by half.

The mass of water recovered was equivalent to the sum of the decant water, residual water in the decant triethylamine/oil, the water contained in the rag layer, and residual water in the subsequent extraction extracts. A summary of this data follows:

### Water Mass Balance

	<u>Buffalo River</u>	<u>Saginaw Bay</u>	<u>Indiana Harbor</u>
Water Portion of Feed	365 g	207 g	784 g
Water Portion of Caustic	+ 4.0 g	+ 4.1 g	+ 9.5 g
	<hr/>	<hr/>	<hr/>
Total calculated water input	= 369 g	= 211 g	= 793 g
.....			
Water recovered from decant water	258 g	172 g	598 g
	<hr/>	<hr/>	<hr/>
Total water recovered	= 258 g	= 172 g	= 598 g
.....			
% Recovery	70	82	75

The recovery of water was low. The temperature tends to increase above the triethylamine/water miscibility limit when the treated solids are centrifuged. At these conditions, some water may have exited the centrifuge with the solids. This water was lost when the solids were dried. In addition, a portion of the water in the feed was left behind in the resin kettle after decantation of the first extraction since it is not possible to decant all the solvent from the solids. This water was lost when the solids were dried. This portion of the water lost in the dryer is not accounted for in the water mass balance. (In RCC's Pilot Unit, and Full-Scale Unit, all such water is recovered from the dryer.)

### **PCB MASS BALANCE**

The PCB mass balance was computed similarly to the method used for oil. The mass of PCBs input was calculated by multiplying the weight of each feed by the PCB concentration as determined by analysis. The PCBs recovered from the test reside in the product oil. The PCBs in the product solids, product water and recovered triethylamine were negligible when calculating a PCB mass balance. The mass of PCBs recovered in the oil was calculated by multiplying the weight of oil recovered by the PCB concentration as determined by analysis. The PCB mass balance for each sample was as follows:

**PCB Balance**

<b><u>Sample</u></b>	<b><u>Calculated PCBs Input</u></b>	<b><u>Calculated PCBs Recovered</u></b>	<b><u>Total PCB % Recovery</u></b>
Buffalo River	0.32 mg	0.22 mg	70 %
Saginaw Bay	14.2 mg	40.0 mg	280 %
Indiana Harbor	12.8 mg	8.3 mg	64 %

**SUMMARY OF MASS BALANCE CALCULATIONS**

The following table summarizes the mass balance calculations for each of the constituents considered. The mass balances were based on the amount of the fraction recovered from the simulation divided by the calculated input amount to the simulation.

**Mass Balance Summary, %**

<b><u>Sample</u></b>	<b><u>Solids</u></b>	<b><u>Oil</u></b>	<b><u>Water</u></b>	<b><u>PCBs</u></b>
Buffalo River	97	112	70	70
Saginaw Bay	98	137	82	280
Indiana Harbor	86	97	75	64

#### **IV. CONCLUSIONS**

**The PCB-contaminated sediment samples from the Buffalo River, Saginaw Bay and Indiana Harbor Sites are suitable for treatment with the B.E.S.T. solvent extraction process. No problems were observed during testing of the samples. Consequently, full-scale processing should be straightforward.**

- 1. The samples were chemically compatible with triethylamine.**
- 2. The total PCB concentrations in the samples tested, 'Buffalo River', 'Saginaw Bay' and 'Indiana Harbor' were 0.60, 21 and 22 mg/kg, respectively.**
- 3. After treatment, the PCB residual removal efficiencies were > 95% for the Buffalo River sample and 99% for the Saginaw Bay and Indiana Harbor samples.**
- 4. The PAH residual concentration in the treated product solids were < 0.2 mg/kg for the Buffalo River and Saginaw Bay samples and ranged from < 1 to < 3 mg/kg for the Indiana Harbor sample.**
- 5. All three treated solids readily passed the TCLP Toxicity Test for leaching of metals.**
- 6. Virtually all of the PCBs from the samples have been concentrated into the product oils. For each sample the weight of PCB contaminated material was reduced 47-270 times.**

**\* \* \* \* \***

## **APPENDIX B**

# **B.E.S.T.<sup>®</sup> BENCH SCALE TREATABILITY TEST PLAN**

The original Plan contained proprietary information. RCC has removed that information from this copy. For this reason, this copy contains blackened-out areas.

**February, 1990**

**! Registered in the U.S. Patent Office**

## 1.0 TEST OBJECTIVES

The test objectives are:

- o To achieve good separation of each type of sample's phase components (oil, water, solids) with low triethylamine residuals in each.
- o To record observations and data that will allow us to predict how a full-scale B.E.S.T. separation of the samples might proceed.
- o Take samples during the extraction tests and conduct analysis sufficient to allow for calculation of mass balances for oil, water, solids and other compounds of interest.
- o To calculate the extraction efficiency of compounds of interest (i.e., PCB's if present) achieved during the bench-scale workups in order to determine the number of stages appropriate for full scale treatment of the site materials.

Evaluation of attainment of these objectives will consist of analysis of the feed and products and observation of the bench-scale simulation in action.

## 2.0 TEST PLAN

The following tasks will be performed on the feed sample:

### 2.1 Characterize the Feed Sample

- o Phase compositional analysis (oil, water, solids)
- o Raw feed metals composition
- o Analysis of other compounds of interest (i.e., PCB's if present)

### 2.2 Perform Preliminary Tests

- o Sample pH adjustment characteristics
- o TEA/Feed compatibility study

### 2.3 Conduct a B.E.S.T. Bench-scale Treatability Test Workup

### 2.4 Feed Compositional Analysis

#### 2.4.1 Analyze the feed for percent oil, water, and solids.

Determine the total oil per Standard Methods for the Examination of Water and Wastewater, 16th Edition, Method 503D, with two exceptions; extend the extraction time from 4 to 16 hours and substitute methylene chloride for Freon based on RCC experience that methylene chloride is a superior solvent for oils and greases.

Determine the total water content by Karl Fisher titration. (The water content is generally not determined by a simple oven test since most oils are relatively volatile. The oven test is used if the ratio of water to oil present in the sample is relatively large.)

Determine the particulate solids content by rinsing a known quantity of feed through a Whatman GF/C filter under vacuum with acetone followed by methylene chloride, drying and weighing the residue.

Normalize the oil + water + solids concentration to 100% if it is 90-110%. Repeat the assays to determine the source of the error(s) if the oil + water + solids concentration is less than 90% or greater than 110%.

### 3.0 TEST PROCEDURE

#### 3.1 Feed Characterization

In conjunction with the performance of a B.E.S.T. laboratory-simulation, a data set must be developed for the sample. The data required includes compositional (oil/water/solids), metals analysis, as well as other compounds of interest.

- a. Determine the total oil per Standard Methods for the Examination of Water and Wastewater, 16th Edition, Method 503D, with two exceptions; extend the extraction time from 4 to 16 hours and substitute methylene chloride for Freon based on RCC experience that methylene chloride is a superior solvent for oils and greases.
- b. Determine the total water content by Karl Fisher titration. (The water content is not typically determined by a simple oven test since most oils are relatively volatile. An oven test will be used to cross-check the results of the Karl Fisher titration if the ratio of water to oil present in the sample is relatively large.)
- c. Determine the particulate solids content by rinsing a known quantity of feed through a Whatman GF/C filter under vacuum with acetone followed by methylene chloride, drying and weighing the residue.
- d. Normalize the oil + water + solids concentration to 100% if it is 90-110%. Repeat the assays to determine the source of the error(s) if the oil + water + solids concentration is less than 90% or greater than 110%.

##### 3.1.1 Feed Sample Metals Composition

Dry approximately 10-20 gms of the feed in the oven at 105°C. Use a ceramic crucible for this task. Dry for at least 6 hours. Record the initial sample weight.

After drying at 105°C put the sample into the muffle furnace and ash at 550°C for at least 3 hours. Once finished, let the sample cool to room temperature. Record the final sample (ash) weight.

Digest the ash with nitric acid as a prelude to analysis of metal concentrations by ICP.

### **3.1.2 TCLP Extraction Metals (SW 846 Method 1311)**

Perform preliminary extraction on a representative portion of each sample to determine the appropriate extraction fluid. After determining the correct extraction fluid, perform 18 hour TCLP extraction on a portion of representative sample.

After filtering the leachate, perform metals analysis using SW846 Method 6010.

### **3.1.2 PCB Analysis**

PCB analysis, if present, will be conducted in accordance with SW846 method 8080. Soxhlet extraction will be used (method 3540). The Aroclor type found in each sample should be recorded.

## **3.2 Preliminary Testing**

TEA can be ionized at some pH conditions. In the ionic form TEA is non-volatile and will not be recovered from the process product phase fractions. To determine the proper pH control requirements for each sample, a pH adjustment test is conducted.

TEA has the potential to react with some rare types of samples. To determine if this will pose a problem during the study a compatibility study will also be performed.

### **3.2.1 Adjustment Characteristics**

Measure the sample pH using pH paper if the sample is mostly solid, and pH probe if it is mostly liquid. If neither seem to be providing a good measurement (i.e., the pH paper color cannot be read or the pH probe readings keep drifting), then add 100 mls of distilled water to about 5 gms of sample, stir well, and remeasure pH.

Obtain 5-10 gms of sample and adjust pH with 5% NaOH solution. If the pH could not be measured as discussed above, be sure to add distilled water prior to the pH adjustment measurements.

After the sample pH has been adjusted to 11 or above, record the amount of caustic spent. Cover the sample with parafilm and leave mixing overnight. On the next day, check the pH again and adjust with caustic soda if needed. Record all amounts of caustic spent.

### **3.2.2 TEA/Feed Compatibility Test**

Mix about 5 gms of each type of sample with 50 mls of cold TEA. Make observations about the ability of the TEA to dissolve the sample.

Use a thermometer to measure the amount of temperature change when the sample is mixed with TEA.

Note any effervescence that takes place such as the formation of hydrogen. Note any unusual reactivity. If there is any effervescence or unusual reactivity, notify the Lab Director before proceeding.

### 3.3 B.E.S.T. Bench-scale Treatability Test Workup

#### 3.3.1 Pre-Treatment and First Wash

Adjust feed pH as determined previously.

Chill sample to below 40°F. Chill TEA to below 38°F.

Mix [redacted] grams of the sample with [redacted] litres of chilled TEA [redacted]. Mix for [redacted] minutes with the pneumatic mixer in the chiller bath (at < 40°F).

At the end of the first mixing stage, remove the particulates with the appropriate method determined during the optimization testing.

Decant and retain supernatant/centrate/filtrate. Keep chilled at < 40°F until heating and decantation can be performed.

Place the centrifuge cake or filter cake, if collected, back into the extraction beaker for additional wash stages.

#### 3.3.2 Second Wash

Mix recovered first extraction stage solids with [redacted] litres of fresh TEA. For the second and subsequent extractions the [redacted]. Use part of the TEA to transfer solids from the centrifuge bottles (if used) into the mixing container. Keep the mixture heated while mixing is in progress.

Mix for [redacted] minutes with the pneumatic mixer.

Perform particle removal from extraction mixture again as performed for the first stage extraction. If desired, collect a portion of the solids for later analysis.

#### 3.3.3 Third Wash and Solids Drying

Repeat section 3.3.2 as a third wash.

Dry the solids at 220°F (105°C) in the forced draft oven. Mix occasionally to facilitate TEA volatilization.

After the initial drying, add a portion of de-ionized water adequate to thoroughly wet the solids, then redry in order to further reduce residual TEA concentrations. To insure that the TEA residual in the dried solids will be low, treat the solids with caustic soda (applied with the de-ionized water) if the pH of these solids is less than 10. Add sufficient caustic soda to raise the pH to approximately 10.5. Determine the required amount of caustic soda on a small portion of the solids.

**NOTE:** Upon occasion, additional washes may be required to achieve required treatment standards.

### 3.3.4 Decantation

Use the large separatory funnel that is in the temperature controlled bath. Keep the water bath at 140°F.

Heat supernatant/centrate/filtrate from first wash (chilled to this point at 40°F) up to 140°F with continuous mixing on a hot plate. Pour into separatory funnel.

Allow 30 minutes quiescent residence time in the separatory funnel. Decant only if the layers appear to have stopped separating. If separation is still proceeding, wait until a better separation is achieved, and then decant.

Record observations of the speed of separation and measure 'rag' volume. Rag layer should ultimately be centrifuged and the resulting TEA/oil and water layers should be added to the appropriate decanted fraction prior to the stripping operation.

Record the weights of the TEA/oil, rag, and water fractions from the decantation.

### 3.3.5 Distillations

#### Water layer

Record initial water pH (should be >11).

Steam strip the water at 110°C (in the rotovap) until no TEA odor is detected in collected distilling drops. Record the initial water volume.

At this point, check water pH. If the pH is >10, distill for 15 minutes more and then terminate distillation.

If water pH is <10, adjust to >12 and continue distillation until no TEA is detected in the collected distillate. Check the latter every 15 minutes, then recheck water pH. If pH is still below 10, then repeat this section (d.) until water pH is >10.

#### Oil/TEA layer

Remove the bulk of TEA without steam at 110°C (in rotovap). Record initial and final oil/TEA volume.

Steam strip the oil at 110°C. Perform this operation by adding a known quantity of water (typically 5 mls) and then measuring the volume of distillate recovered. When all TEA is removed, the recovery of the distillate should be equal to the amount of water added.

Perform oil polishing by distilling without steam until the oil temperature reaches 120°C to facilitate excess water removal. Record the final product oil weight.

Be sure to collect some of the final distillate for measurement of the extent of any volatile organic carryover.

## **4.0 ANALYTICAL REQUIREMENTS**

### **4.1 Feed Analysis**

- o **Composition - % (wt) oil, water and solids by:**
  - 105°C total solid
  - Oil by Soxhlet extraction (by dichloromethane)
  - Solids by difference
- o **Total metals**
  - Dry at 105°C
  - Ash at 550°C
  - Nitric acid digest for ICP heavy metals determination
- o **Physical properties:**
  - pH
  - Specific Gravity
- o **pH Adjustment:**
  - Amount of caustic added to pH adjust the feed to pH of 11
- o **PCB's by Method 8080, if present**
- ~~o **TCLP Metals Extraction Analysis**~~

### **4.2 Product Solids Analysis**

- o **Residual Oil and Grease by Soxhlet extraction**
- o **Aqua regia digest for ICP total trace metals**
- o **TCLP Extraction Metals Analysis**
- o **PCB's, if present**
- o **Residual TEA**

### **4.3 Decant Water Analysis**

- o **PCB's, if present and if sample size permits**
- o **Oil & Grease by freon extraction (IR if volume is limited)**
- o **Total metals**

### **4.4 Oil Analysis**

- o **PCB's, if present**

## **5.0 SAFETY CONSIDERATIONS**

Because of the unknown reactivity of these samples, extreme care should be taken not to allow a dangerous situation to develop. Results of the compatibility study should be reviewed with the Lab Director prior to initiation of the full scale bench-scale extraction. The bulk of these simulations will be done in the laboratory hood to decrease TEA emissions. Prior to testing, the feed will be subjected to TEA-compatibility tests to verify that it does not react violently with TEA. Other safety precautions involving personnel conducting B.E.S.T. laboratory work that will be followed are described in RCC's Laboratory Safety Manual. If you have any other safety related concerns, contact the Lab Director.

**NOTE: Be sure to check that the ventilation system is working properly and wear appropriate protective equipment when handling these samples.**

**APPENDIX C**

**QUALITY ASSURANCE PROJECT PLAN  
FOR  
GLNPO - ASSESSMENT AND REMEDIATION OF  
CONTAMINATED SEDIMENT TECHNOLOGY  
DEMONSTRATION SUPPORT**

Revision II

February 15, 1991

Submitted to:

U.S. Environmental Protection Agency  
Great Lakes National Program Office  
230 S. Dearborn  
Chicago, Illinois 60604

Submitted by:

Science Applications International Corporation  
635 West Seventh Street, Suite 403  
Cincinnati, Ohio 45203

EPA Contract No. 68-C8-0061, Work Assignment No. 2-18  
SAIC Project No. 1-832-03-207-50

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# QUALITY ASSURANCE PROJECT PLAN APPROVALS

QA Project Plan Title: GLNPO Assessment and Remediation of Contaminated  
Sediment Technology Demonstration Support

Prepared by: Science Applications International Corporation (SAIC)

QA Project Category: II

Revision Date: January 9, 1990

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## 1.0 INTRODUCTION

The Great Lakes National Program Office (GLNPO) leads efforts to carry out the provisions of Section 118 of the Clean Water Act (CWA) and to fulfill U.S. obligations under the Great Lakes Water Quality Agreement (GLWQA) with Canada. Under Section 118(c)(3) of the CWA, GLNPO is responsible for undertaking a 5-year study and demonstration program for contaminated sediments. Five areas are specified for priority consideration in locating and conducting demonstration projects: Saginaw Bay, Michigan; Sheboygan Harbor, Wisconsin; Grand Calumet River, Indiana (aka: Indiana Harbor); Ashtabula River, Ohio; and Buffalo River, New York. In response, GLNPO has initiated an Assessment and Remediation of Contaminated Sediments (ARCS) Program. The ARCS Program will be carried out through a management structure including a Management Advisory Committee consisting of public interest, Federal and State agency representatives, an Activities Integration Committee which is made up of the chairpersons of the technical work groups, and technical work groups.

In order to obtain the broadest possible information base on which to make decisions, the ARCS Program will conduct bench-scale and pilot-scale demonstrations and utilize opportunities afforded by contaminated sediment remedial activities by others, such as the Corps of Engineers and the Superfund program, to evaluate the effectiveness of those activities. These bench-scale and pilot-scale tests will be developed and conducted under the guidance of the Engineering/Technology (ET) Work Group for ARCS.

SAIC has been contracted to supply technical support to the ET Work Group. The effort consists of conducting bench-scale treatability studies on designated sediments to evaluate the removal of specific organic contaminants.

Sediments have been obtained by GLNPO from various sites and represent the type of material that would be obtained for onsite treatment. The primary contaminants of these sediments are polychlorinated biphenyls (PCBs) and polynuclear aromatic hydrocarbons (PAHs). Analyses to date show PCB concentrations are less than 50 ppm. These sediments have been homogenized and packaged in smaller containers by EPA.

## 2.0 PROJECT DESCRIPTION

### 2.1 Background

SAIC and its subcontractors will conduct seven (7) bench-scale (several liters) tests on wet contaminated sediments using four treatment technologies.

The seven treatability tests (as currently planned) will utilize sediments from 4 sites (Saginaw River, Buffalo River, Indiana Harbor Canal, and Ashtabula River). Five sediments have been collected from these sites by GLNPO. These samples have been homogenized by the U.S. EPA and are being stored under refrigeration in 5 gallon containers by EPA in Duluth, MN.

These five sediments are currently being analyzed in the U.S. EPA, Environmental Research Laboratory in Duluth. The Duluth Laboratory is analyzing the sediments for total organic carbon/total inorganic carbon (TOC/TIC), particle size, density of dry material, total sulfur, acid volatile sulfide, oil and grease (O & G), total PCBs, PAHs (10), and metals including mercury. Table 2-1 is a summary of the data received to date.

A portion (small vial) of each residual of each treatability test may be retained and sent to the GLNPO office for "show" purposes. If available, sub-regulated quantities of the solid and oil residuals from each test treatability study may also be retained and shipped to EPA for possible further treatment studies.

The following is a list of technologies and the proposed number of sediment samples to be tested by each technology:

- a. B.E.S.T.<sup>TM</sup> Extraction Process on three samples (Buffalo River, Indiana Harbor, Saginaw TRP 6)
- b. Low Temperature Stripping (RETEC) on one sample (Ashtabula River)
- c. Wet Air Oxidation (Zimpro Passavant) on one sample (Indiana Harbor)
- d. Low Temperature Stripping (Soil Tech) on two samples (Buffalo River and Indiana Harbor)

Summaries of these technologies are included in Appendix A.

TABLE 2-1a. Preliminary Analytical Results on ARCS Sediments

Description	Concentration (Mg/kgm)(a)											Concentration (%) (a)	
	Total PCB	Total PAH	Cu	Cd	Ni	Fe (%)	Cr	Zn	Pb	TOC	O & G	Moisture (b)	
Saginaw 221	0.6	1.2	33	0.9	76	1.4	140	240	30	1.4	0.1	40.3	
Saginaw TRP6	6.0	3.1	81	4.7	110	0.9	200	200	47	1.2	0.3	31.1	
Ashtabula River	C	C	55	3.0	96	3.7	550	240	48	2.6	1.7	52.9	
Indiana Harbor	0.2	96	320	9.4	150	16	540	3300	780	21	5.8	61.0	
Buffalo River	0.4	5.6	85	1.9	57	3.9	110	200	94	2.0	0.5	41.5	

(a) Concentration in ppm and dry weight basis unless otherwise indicated.  
 (b) As received basis.

TABLE 2-1b. Preliminary Particle Size Distribution (%)

Description	Particle Size (a)						Median Diameter, u
	> 50 u	50-20 u	20-5 u	5-2 u	2-0.2 u	0.2-0.08 u	
Buffalo River	19.8	12.1	29.0	11.8	24.3	2.4	0.6

(a) u microns

## 2.2 Testing Program for Chemical Characterization

SAIC shall be primarily responsible for the physical and chemical characterization of both the sediment samples prior to testing and the residuals created during the tests. Analyses conducted by the vendors or subcontractors will not be depended on, but such data shall be reported whenever available.

Two different sets of chemical analyses will be conducted during the performance of the treatability tests: optimization test analyses and performance evaluation analysis. The Phase I optimization test analyses will be conducted by the subcontractor or vendor during the series of initial technology tests. The Phase II performance evaluation analyses will be conducted by SAIC (or its analytical subcontractor) on the raw sediment sample prior to the treatability test run at optimum conditions and on the end products produced by that particular test. These tests are described further in this section.

In order to assure objectivity and consistency of data obtained from multiple vendors running different technology tests, SAIC shall conduct analyses as described in Table 2-2 for characterization of the sediments and the end products of the treatability tests at optimum conditions (Phase II).

The analyses described for the solid fraction in Table 2-2 shall be performed by SAIC's analytical subcontractor once on a subsample taken from each sample sent to each vendor or subcontractor for treatability tests (Phase II). This subsample will be taken at the same time that the sample for the Phase II treatability study is taken by the vendor. This data will serve as the measure of the raw sediment quality for comparison to analyses of treated end products from each technology test that may be conducted on sediments from a particular area of concern.

Each bench-scale technology test may actually involve the performance of multiple laboratory simulations. During the initial tests (Phase I), any analyses performed by the

vendor or subcontractor shall be reported, as available. For the tests run at optimum conditions (Phase II), SAIC shall conduct the full suite of analyses, as detailed in Table 2-2, on the end products if sufficient quantities are produced by the technology. Quotes solicited for each technology specified that a minimum 300 grams dry basis of treated solid had to be produced for SAIC's analyses. Table 2-3 shows the apportionment of the 300 grams for the solid analyses. The quantity of water is depended on the sediments and the individual technologies. To do all the analyses listed in Table 2-2, and associated QC, approximately 10 liters of water are required. Table 2-4 listed specified sample volumes for each analysis, and gives a priority to each analysis. It is possible that only the PCB and PAH analysis and associated QC will be performed on the water samples. If any oil residue is produced, it will be analyzed by dilution with appropriate sample cleanup steps for PCBs and PAHs.

The data generated by SAIC's analyses of the untreated sediment and the treated end products from the test at optimum conditions will be primarily relied upon to determine treatment efficiencies. Vendor- or subcontractor-generated data will not be relied upon but shall be reported when available.

### 2.3 Required Permits

Because of the small quantities of sediments required for the bench-scale treatability tests, SAIC anticipates that no formal permits will be required to conduct these tests. If this is not the case and permits (such as TSCA, RD&D or RCRA permits) are required, the subcontractor will notify SAIC and the TPM will be notified to obtain approval for acquisition of the permit(s).

All unused sediment samples requested by SAIC for the treatability test and all testing residuals, except those requested by the TPM for "show" purposes and those requested by the TPM for possible further testing, will be properly disposed of per federal and state regulations.

TABLE 2-2

## Parameters and Detection Limits for Analysis of ARCS Technologies

<u>Parameter</u>	<u>Solid</u> <sup>1</sup>	<u>Water</u> <sup>2</sup>	<u>Oil</u> <sup>3</sup>
TOC/TIC	300	1000	
Total Solids <sup>4</sup>	1000		
Volatile Solids <sup>4</sup>	1000	1000	
Oil & Grease <sup>4</sup>	10	1000	
Total Cyanide	0.5	10	
Total Phosphorus	50	10	
Arsenic <sup>4</sup>	0.1	1	
Barium <sup>4</sup>	0.2	2	
Cadmium <sup>4</sup>	0.4	4	
Chromium <sup>4</sup>	0.7	7	
Copper <sup>4</sup>	0.6	6	
Iron (total) <sup>4</sup>	0.7	7	
Lead <sup>4</sup>	5	50	
Manganese <sup>4</sup>	0.2	2	
Mercury <sup>4</sup>	0.1	0.01	
Nickel <sup>4</sup>	2	20	
Selenium <sup>4</sup>	0.2	1	
Silver <sup>4</sup>	0.7	7	
Zinc <sup>4</sup>	0.2	2	
PCBs (total & Aroclors) <sup>4</sup>	0.02	0.07	0.1
PAHs (16) <sup>4,5</sup>	0.2	2	0.1
pH	full range	full range	
BOD <sub>5</sub>		1000	
Total Suspended Solids <sup>4</sup>		1000	
Conductivity		full range	

NOTES:

- <sup>1</sup> Detection limits for solids are ppm (mg/kg dry weight). The D.L.'s for metals should be obtainable by ICP except for As, Se, and Hg. If GFAA is used, the D.L.'s will be 2 mg/kgm except Hg, Cd, and Ag which will be 0.1 mg/kgm.
- <sup>2</sup> Detection limits for water are ppb (ug/l). The D.L.'s for metals should be obtainable by ICP except for As, Se, Hg. If GFAA is used D.L.'s will be 1 ug/L except Hg which will be 0.01 ug/L.
- <sup>3</sup> Detection limits for oil are ppm (mg/l).
- <sup>4</sup> Parameters tentatively identified for QC analyses.
- <sup>5</sup> Polynuclear aromatic hydrocarbons to be analyzed are the 16 compounds listed in Table 5-2.

TABLE 2-3

Solid Sample Quantities for Analyses

<u>Parameter</u>	<u>Initial Sample (g)</u>	<u>QC (g)</u>	<u>Total (g)</u>	<u>QC Approach</u>
TOC/TIC	15	--	15	None <sup>1</sup>
Total + Volatile Solids	5	10	15	Triplicate/Control
Oil & Grease	20	40	60	Triplicate/Control
Total Cyanide	10	--	10	None <sup>2</sup>
Total Phosphorous	5	--	5	None <sup>2</sup>
Metals (except Hg)	5	15	20	MS/Triplicate
Hg	1	3	4	MS/Triplicate
PCBs + PAHs	30	90(60) <sup>3</sup>	90	(3)
pH	20	--	20	None <sup>4</sup>
Subtotals	111	158(128)	269(239)	
Reserve	--	--	31(61)	
TOTAL	--	--	300	

<sup>1</sup> For sample set II that does not have such a limited quantity of solid, The QC described in footnote 3 will be implemented.

<sup>2</sup> For sample set II, MS/triplicate QC will be implemented.

<sup>3</sup> Quality control for untreated solids is Triplicate and spike and for treated solids matrix spike and matrix spike duplicate.

<sup>4</sup> For sample set II, Triplicate/Control sample QC will be implemented. The control sample may be an EPA QC check sample, an NBS - SRM, a standard laboratory reference solution, or other certified reference material.

TABLE 2-4

Sample Volumes Required and Priority Ranking for Water Analyses

<u>Parameter</u>	<u>Priority</u>	<u>Analysis Volume, ml</u>	<u>QC Volume, ml</u>	<u>QC Approach</u>
TOC/TIC	7	25	--	None (e)
Volatile Solids	5	d	d	Triplicate/Control
Oil & Grease	6	1000	2000	Triplicate/Control
Total Cyanide	7	500	--	None (f)
Total Phosphorus	7	50	--	None (f)
Arsenic	4	100	300	MS/Triplicate
Barium	2	100	300	MS/Triplicate
Cadmium	2	b	b	MS/Triplicate
Chromium	2	b	b	MS/Triplicate
Copper	2	b	b	MS/Triplicate
Iron (total)	2	b	b	MS/Triplicate
Lead	2	b	b	MS/Triplicate
Manganese	2	b	b	MS/Triplicate
Mercury	3	100	300	MS/Triplicate
Nickel	2	b	b	MS/Triplicate
Selenium	4	c	c	MS/Triplicate
Silver	2	b	b	MS/Triplicate
Zinc	2	b	b	MS/Triplicate
PCBs (total & Aroclors)	1	1,000	2,000	MS/MSD
PAHs (16)	1	a	a	MS/MSD
pH	7	25	--	None (f)
BOD	7	1,000	--	None (f)
Total Suspended Solids	5	200	400	Triplicate/Control
Conductivity	7	100	--	None (f)

Note:

- a) same aliquot as PCBs  
 b) same aliquot as Barium  
 c) same aliquot as Arsenic  
 d) same aliquot as Total Suspended Solids  
 e) see footnote 2, Table 2-3  
 f) see footnote 4, Table 2-3

#### 2.4 Purpose of Phase I Experimental Design

The purpose of the Phase I technology experimental design is for each subcontractor to establish a range of variables best suited for feasibly implementing their technology on a full-scale basis (Phase II). SAIC will send a quantity (specified by the vendor) of each sediment to the vendor to accomplish this. All data generated by the vendor during Phase I will be supplied to SAIC for inclusion in the report for that technology. This information will include the operating conditions/parameters, the input/output data for the contaminants of interest to show the range of effectiveness associated with various operating conditions, and the quantities of the input material and the various residuals resulting from the test. The optimum set of conditions to be used for Phase II will be reported to SAIC along with appropriate revisions to the Phase I experimental design to make it applicable to Phase II.

#### 2.5 Purpose of Phase II Treatability Test

SAIC will send another container of sediment(s) to the vendor (quantity to be specified by the vendor). This container will not be opened until a representative of SAIC arrives for the scheduled treatability test(s). Other observers from U.S. EPA, COE and/or the GLNPO may also be present during the Phase II treatability test(s).

The new sample will be homogenized and a sample equivalent to a minimum of 300 gm of dry solids will be set aside for characterization analyses (Table 2-2) by SAIC. SAIC will observe the treatability tests and obtain samples of process residuals for analyses (Table 2-2). The bench-scale test(s) must produce enough solid residual for all vendor requirements and a quantity equivalent to 300 gm of dry solids for SAIC analyses. SAIC can utilize up to 10 liters of water for analysis and 25 ml of the oil residual. The actual quantities of water and oil that will be produced are dependent on the initial sediment and the technology. All technologies except wet air oxidation are expected to produce an oil residual. Also, if additional solid and/or oil residue is available, EPA may ask for these materials to be sent to them for storage for possible future evaluation.

All data generated by the vendor during Phase II is to be supplied to SAIC for inclusion in the report for that technology. The vendor must stipulate in their work plan, prior to conducting the test(s), the process locations to be sampled, the frequency and the information being obtained.

All other residuals from both phases of the treatability study, including any untreated sediment, will be properly disposed of by the vendor.

SAIC shall oversee the treatability test assessment(s) by vendors or subcontractors, including all QA/QC aspects, monitoring and analysis. SAIC shall ensure compliance with the specific experimental design during the tests conducted by vendors or subcontractors. SAIC will make specific notes regarding the equipment being used, any pretreatment of the sediment(s), the operation of the equipment, and any post treatment of the residuals. SAIC personnel will pack the untreated sediment sample and the end product samples from the Phase II test for each technology in an appropriate fashion for shipment from the vendor or subcontractor to the laboratory SAIC is using for the analysis. Proper chain-of-custody procedures will be developed in the QAPjP and strictly followed by SAIC personnel.

SAIC plans to take photos of the equipment while at the vendor's location for inclusion in the report.

SAIC shall perform limited interpretation of technology test results, specifically the development of material and energy balances. No test of air or fugitive emissions will be done. For material balances, estimates of the mass distribution of the analytes of interest (Table 2-2) among the residuals will be made. The term *energy balance* is interpreted to mean an estimation by the vendor of the energy input into the process at a pilot- or full-scale.

SAIC shall collect any information available from the vendor or subcontractor concerning the actual or estimated costs of constructing and operating full-scale versions of the technology tested.

The purpose of this project is to test five technologies for removing organic contaminants (PCBs and PAHs) from sediments typical of locations around the Great Lakes. GLNPO is specifying the technologies and the sediment(s) to be treated by each technology. This study is only one part of a much larger program, and it is not necessarily intended to evaluate the complete treatment of these sediments. Other aspects or treatment options are being evaluated by a number of agencies, contractors, etc.

Therefore, this study is based on the following assumptions:

- The percent removal of the PCBs and PAHs from the solid residual is the most important object of this study.
- The untreated sediments and solid residuals are the most important matrices.
- If water and oil residuals are generated by a technology, the existence of an appropriate treatment or disposal option for these residuals is assumed. PAHs and PCBs will be determined in these residuals as a cross check of their fate in treating the solids.

Based on the intents of this study, the critical measurements are PAHs, PCBs, metals, total solids, volatile solids, and oil and grease in the untreated and treated solids.

## 2.6 Organization and Responsibilities

A project organization and authority chart is shown in Figure 2-1. The Environmental Monitoring Systems Laboratory (EMSL) is cooperating with GLNPO and SAIC on this evaluation. Mr. Thomas Wagner is the SAIC Work Assignment Manager and is responsible for the technical and budgeting aspects of this work assignment. Mr. Clyde Dial is QA Manager and is responsible for QA oversight on this work assignment.

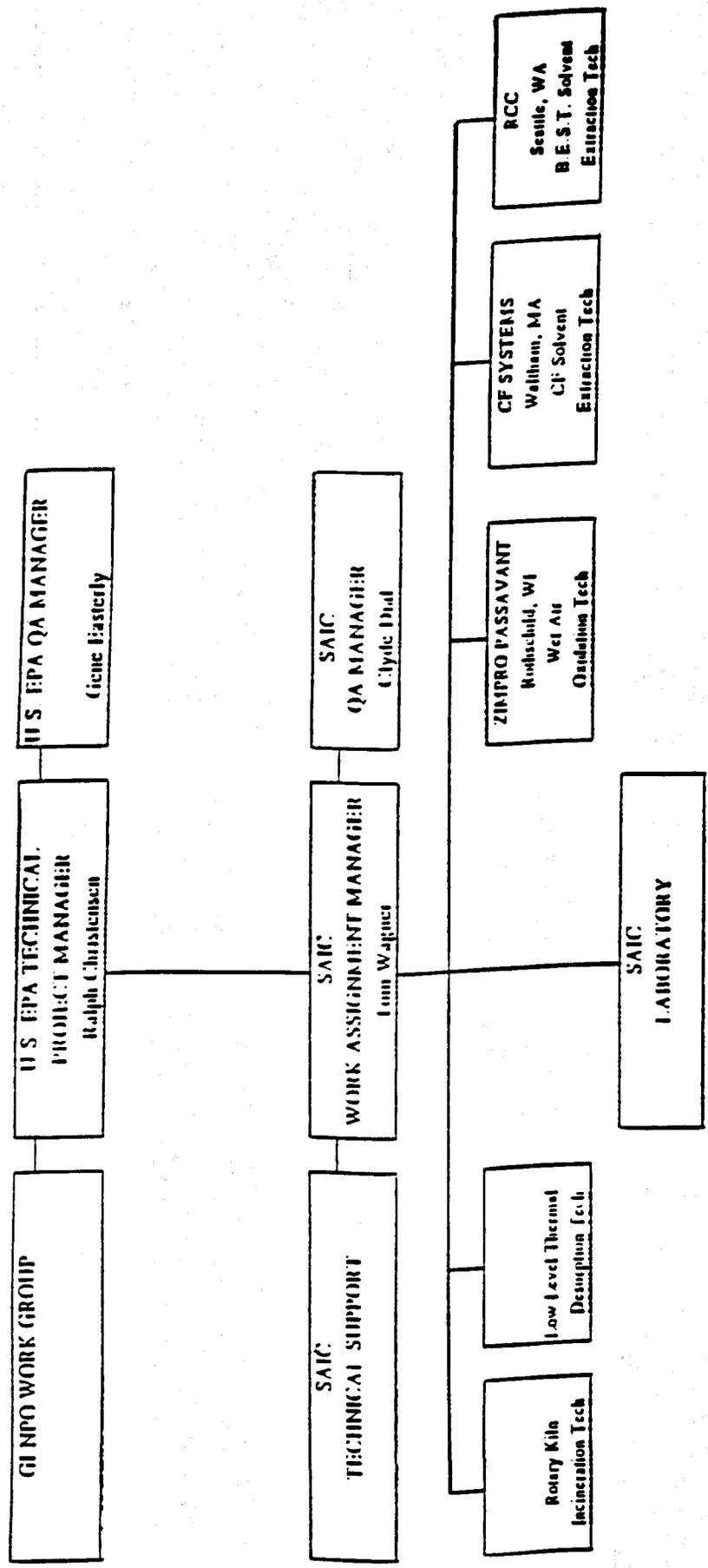


Figure 2-1. Project Organization

2.7 Schedule

The Phase I experimental designs are scheduled for mid to late February 1990, and the Phase II Treatability Tests are scheduled for March and April 1991.

### 3.0 QUALITY ASSURANCE OBJECTIVES

#### 3.1 Precision, Accuracy, Completeness, and Method Detection Limits

Objectives for accuracy, precision, method detection limits, and completeness for the critical measurements of solids are listed in Table 3-1. Accuracy (as percent recovery) will be determined from matrix spike recovery for PAHs, PCBs and metals, and from laboratory control samples (certified reference material- CRM) for the remaining analyses. Precision (as relative standard deviation) will be determined from the results of triplicate analyses for PAHs, PCBs, solids (total, volatile and/or suspended), oil and grease, and metals. Matrix spike and matrix spike duplicate analyses will be used for treated solids for PCBs and PAHs. The completeness will be determined from the number of data meeting the criteria in Table 3-1 divided by the number of samples that undergo performance evaluation analyses.

#### 3.2 Representativeness and Comparability

Representativeness and Comparability are qualitative parameters. The sediment samples have already been collected and have been reported to be representative of the areas to be remediated. The data obtained in this program will be comparable because all the methods are taken from a standard EPA reference manual and all the analyses will be conducted at the same laboratory. Reporting units for each analysis are specified in Section 6 of this document and are consistent with standard reporting units in this program.

#### 3.3 Method Detection Limits

The target detection limits (TDLs) were specified by GLNPO (Table 2-2). Based on the analytical methods appropriate for the analyses and the amount of samples specified in the methods, the detection limits listed in Table 3-1 should be achievable. Generally the instrument detection limits are defined as 3 times the standard deviation of 15 blanks or standards with a concentration within a factor of 10 of the IDL.

**TABLE 3-1. Quality Assurance Objectives for Critical Measurements  
 (Sediments and Treated Solids)**

Parameter	Method (a)	Accuracy (b) (as % recovery)	Precision (c) %	Method Detection Limit (d) (mg/kgm)	Completeness %
Total Solids	160.3	80-120	20	1000	90
Volatile Solids	160.4	80-120	20	1000	90
Oil & Grease	9071	80-120	20	10	90
Arsenic	3050/7060	85-115	20	0.1	90
Barium	3050/6010	85-115	20	0.2	90
Cadmium	3050/6010	85-115	20	0.4	90
Chromium	3050/6010	85-115	20	0.7	90
Copper	3050/6010	85-115	20	0.6	90
Iron (total)	3050/6010	85-115	20	0.7	90
Lead	3050/6010	85-115	20	5	90
Manganese	3050/6010	85-115	20	0.2	90
Mercury	7471	85-115	20	0.1	90
Nickel	3050/6010	85-115	20	2	90
Selenium	3050/7740	85-115	20	0.2	90
Silver	3050/6010	85-115	20	0.7	90
Zinc	3050/6010	85-115	20	0.2	90
PCBs (total & Aroclors (c))	3540 or 3550/8080	70-130	20	0.02	80
PAHs (Table 5-2)	3540 or 3550/ 8270 or 8100	70-130	20	0.2	90

- (a) References are to "Methods for Chemical Analysis of Water and Wastes", EPA/600/4-79/020 or "Test Methods for Evaluating Solid Waste", SW-846, 3rd. Ed.
- (b) Determined from MS or MS/MSD analyses for metals, PAHs, and PCBs; others determined from laboratory control samples.
- (c) Determined as relative percent standard deviation of triplicate analyses, except PAHs and PCBs in treated solids where MS/MSD will be used.
- (d) See Footnotes 1 and 2 of Table 2-2
- (e) Detection limits based on extraction of 30 gram samples.

#### 4.0 SAMPLE TRANSFER AND PREPARATION PROCEDURES

As described in Section 2, SAIC will receive a number of 5 gallon containers of previously homogenized sediments from the U. S. EPA in Duluth, Minnesota. The number of containers of each sediment is dependent on the final determination by GLNPO of which sediments will be tested by the various technologies. Only if smaller portions of sediments are requested by the vendors will these containers be opened by SAIC. If smaller portions are required, SAIC will resuspend the solids and water within an individual container by rolling, tumbling, and stirring of the contents. The final stirring will be in the original containers using a metal stirrer as would be used to mix a 5 gallon container of paint. The metal stirrer is appropriate because metals are not the primary constituents of concern in these treatability tests.

The *Chain of Custody Record* shown in Figure 4-1 will be completed for each cooler shipped to the subcontractor or vendor that will conduct the optimization and performance evaluation tests. The samples obtained from the vendor for analysis will be labeled as shown in Figure 4-2. The labels will document the sample I.D., time and date of collection, and the location from where the sample was taken. The amount/type of preservative that was added will also be recorded.

SAIC personnel will pack and ship the untreated sediment and the end product samples (residuals) from the optimum conditions test for each technology. The amount of preservative will be recorded. Samples will be labeled (see Figure 4-2) and shipped by overnight delivery service to the laboratory in coolers containing ice. If "blue ice" is used in the coolers, samples will be initially cooled with regular ice prior to being packed in the coolers with blue ice. The *Chain of Custody Record* (Figure 4-1) will be completed for each cooler shipped to the laboratory.

Solid, sediment and oil samples require no preservative other than cooling to 4° C. The appropriate types of containers (solid and liquids), holding times, and preservatives for water samples are listed in Table 4-1.

**TABLE 4-1. Sample Containers, Preservation and Holding Times**

<i>Parameter</i>	<i>Container</i>	<i>Preservation of Water Samples</i>	<i>Holding Time</i>
TOC	P,G	Cool 4° C, H <sub>2</sub> SO <sub>4</sub> to pH < 2	28 days
Solids (Total, Volatile & Suspended)	P,G	Cool 4° C	7 days
Oil and Grease	G	Cool 4° C, H <sub>2</sub> SO <sub>4</sub> to pH < 2	28 days
Total Cyanide	P,G	Cool 4° C, NaOH to pH > 12 0.6g Ascorbic acid	14 days
Total Phosphorous	P,G	Cool 4° C, H <sub>2</sub> SO <sub>4</sub> to pH < 2	28 days
Metals (except Cr VI)	P,G	HNO <sub>3</sub> to pH < 2	6 months except Hg (Hg 28 days)
Cr (VI)	P,G	Cool 4° C	24 hours
PAHs & PCBs	G teflon lined cap	Cool 4° C, store in dark	Extract within 7 days Analyze within 40 days
BOD <sub>5</sub>	P,G	Cool 4° C	48 hours
pH	P,G		Performed immediately
Conductivity	P,G	Cool 4° C	28 days





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---

*Sample No.:* \_\_\_\_\_

*Sample Location/Date/Time:* \_\_\_\_\_

*Project Location/No.:* \_\_\_\_\_

*Analysis:* \_\_\_\_\_

---

*Collection Method:* \_\_\_\_\_ *Purge Volume:* \_\_\_\_\_

*Preservative:* \_\_\_\_\_

*Comments:* \_\_\_\_\_

*Collector's Initials* \_\_\_\_\_

Figure 4-2. Example Sample Label

## 5.0 ANALYTICAL PROCEDURES AND CALIBRATION

Analytical procedures for all critical measurements are referenced in Table 3-1. The non-critical measurements are for any residual water and oil remaining after the performance evaluation tests and some additional analyses on the solid samples. The EPA procedures are specified in Table 5-1.

The required calibration for all analyses are specified in the methods and will be followed. All instruments will be calibrated as specified in the methods prior to performing any analysis of the samples. Internal QC checks, including initial calibration and continuing calibration checks, for the critical measurements are listed in Table 7-1.

Table 5-2 contains the minimum list of the sixteen PAHs that must be determined by either analytical method. Additional compounds may be included, but none of these sixteen may be deleted from the target list.

The laboratory is responsible for maintaining a preventive maintenance program consistent with manufacturers recommendations for all instruments required for this program. In addition, they are responsible for having a sufficient supply of routine spare parts necessary for the operation of the analytical equipment in order to complete the analysis in a timely fashion.

**TABLE 5-1**

**Analytical Methods for Critical and Non-critical Measurements**

<i>Parameter</i>	<i>Methods<sup>a</sup></i>		
	<i>Solid</i>	<i>Water</i>	<i>Oil</i>
TOC	9060	9060	NA
Total Solids	160.3	NA	NA
Volatile Solids	160.4	160.4	NA
Oil and Grease	9071	413.1	NA
Total Cyanide	9010	9010	NA
Total Phosphorous	365.2	365.2	NA
Arsenic	3050/7060	7060	NA
Mercury	7471	7470	NA
Selenium	3050/7740	7740	NA
Other Metals	3050/6010	3010/6010 (7760 Ag)	NA
PCBs	3540 or 3550/8080	3510 or 3520/8080	3580/8080
PAHs	3540 or 3550/ 8270 or 8100 <sup>b</sup>	3510 or 3520/ 8270 or 8100 <sup>b</sup>	3580/8270
pH	9045	9040	NA
BOD	NA	405.1	NA
Total Suspended Solids	NA	160.2	NA
Conductivity	NA	9050	NA

(a) References are to "Methods for Chemical Analysis of Water and Wastes", EPA/600/4-79/020 or "Test Methods for Evaluating Solid Waste", SW-846, 3rd. Ed.

(b) Where options for methods are given, -Either is acceptable if the detection limits given in Table 2-2 can be achieved.

NA - Not analyzed

**TABLE 5-2**

**List of PAHs<sup>a</sup>**

---

Acenaphthene	Chrysene
Acenaphthylene	Dibenzo(a,h)anthracene
Anthracene	Fluoranthene
Benzo(a)anthracene	Fluorene
Benzo(a)pyrene	Inden(1,2,3-cd)pyrene
Benzo(b)fluoranthene	Naphthalene
Benzo(k)fluoranthene	Phenanthrene
Benzo(ghi)perylene	Pyrene

---

- <sup>a</sup> PAH analyses must determine these 16 compounds at a minimum.

## 6.0 DATA REDUCTION, VALIDATION AND REPORTING

Data will be reduced by the procedures specified in the methods and reported by the laboratory in the units also specified in the methods. The work assignment manager or his designer will review the results and compare the QC results with those listed in Table 3-1. Any discrepancies will be discussed with the QA Manager.

All data will be reviewed to ensure that the correct codes and units have been included. All organic and inorganic data for solids will be reported as mg/kgm except TOC, oil & grease (O&G), moisture and iron that will be reported as percent and pH that will be reported in standard pH units. All metals and organics in water samples will be reported as ug/l. TOC, solids (suspended and volatile), O&G, cyanide, phosphorus, and BOD will be reported as mg/l. Conductivity will be reported as umhos/cm and pH as standard pH units. After reduction, data will be placed in tables or arrays and reviewed again for anomalous values. Any inconsistencies discovered will be resolved immediately, if possible, by seeking clarification from the sample collection personnel responsible for data collection, and/or the analytical laboratory.

Data Tables in the report will be delivered in hard copy and on discs. The discs will be either in Lotus files or WordPerfect 5.1 files.

## 7.0 INTERNAL QUALITY CONTROL CHECKS

The internal QC checks appropriate for the measurement methods to be utilized for this project are summarized in Table 7-1. These items are taken from the methods and the QC program outlined in Section 3 of this QAPjP.

For the GLNPO program, the following QC measures and limits are employed:

- |                             |  |
|-----------------------------|--|
| on-going calibration checks | <ul style="list-style-type: none"><li>- beginning, middle, and end of sample set for metals, pH, TOC/TIC, total cyanide, and total P</li><li>- mid-calibration range standard</li><li>- <math>\pm 10\%</math> limit unless otherwise stated</li><li>- <math>\pm 0.1</math> pH unit for pH</li><li>- <math>\pm 10</math> umhos/cm for conductivity at 25° C</li></ul> |
|                             | <ul style="list-style-type: none"><li>- beginning, every 12, and end of sample set for PCBs and PAHs</li><li>- mid calibration range standard</li><li>- <math>\pm 10\%</math> limit</li></ul>  |
| method blanks               | <ul style="list-style-type: none"><li>- one per sample set for PCBs and PAHs</li><li>- &lt; MDL limit unless otherwise stated</li><li>- beginning, middle and end for metals, TOC/TIC, total P, total cyanide, and pH</li><li>- beginning, middle and end for conductivity with acceptance limits of &lt; 1 umho/cm</li></ul>  |
| matrix spikes               | <ul style="list-style-type: none"><li>- one per sample set</li><li>- 1 to 1.5 times the estimated concentration of sample</li><li>- <math>\pm 15\%</math> limit for metals; <math>\pm 30\%</math> for PCBs and PAHs</li></ul>  |
| replicates                  | <ul style="list-style-type: none"><li>- triplicate analyses</li><li>- RSD <math>\leq 20\%</math> unless otherwise stated</li><li>- one per sample set</li><li>- <math>\pm 0.1</math> pH unit for pH</li><li>- <math>\pm 2</math> umhos/cm for conductivity</li></ul>   |

- QC sample (CRM) - - minimum of one per sample set
- ± 20% of known CRM
- ± 0.1 pH unit for pH
- ± 1 umhos/cm for conductivity
  
- surrogate spikes - added to each sample
- (PCBs and PAHs only) - ± 30% recovery

The surrogate for PCB analysis is tetrachlorometaxylene and the internal standard is 1,2,3-trichlorobenzene.

Table 7-2 shows an analytical matrix that will be completed for each technology tested. For example, consider the case of a bench scale treatability test of (1 kilogram) Indiana harbor sediment by low temperature stripping. Based on the data presented in Table 2-1a and assuming complete separation and recovery of oil, water, and solid, a 1 kilogram sample of untreated sediment will produce 58 grams of oil, 610 ml of water, and 332 grams of dry treated solids. For the purpose of this program, this sample set consists of 1 untreated solid, 1 treated solid, and the water and oil generated by the process. Table 7-3 is a completed analytical matrix for this test. Table 7-3 is based on Tables 2-2 and 2-4 and the QC approach described in this QA plan. The analysis of the water sample in this example is severely limited by the relatively small amount of sample obtained.

Table 7-4 is a matrix summarizing the anticipated samples to be analyzed for this project. The sets for each technology (see section 2.1) are:

- I B.E.S.T.
- II ReTec
- III Wet Air Oxidation
- IV Soil Tech

The Soil Tech process will process treated soils at two distinct points. Therefore, four treated solids are produced from the two untreated sediments.

TABLE 7-1. Internal QC Checks for Measurements

Parameter	Method (a)	Initial Calibration	Calibration Checks	Method Blank	MS/MSD	Triplicate Sample Analysis	QC Sample	Surrogate Spikes
Solids (Total & Volatile)	160.3	Balance (Yearly)	Balance Each Day	Yes	NA	Yes	Yes	NA
	160.4							
Oil & Grease	9071	See Above	See Above	Yes	NA	Yes	Yes	NA
Metals	6010	2 points	Every 10th Sample	Yes	MS only	Yes	Yes	NA
Metals	7000 series	4 points	Every 10th Sample	Yes	MS only	Yes	Yes	NA
PCBs (b)	8080	5 points	Every 10th Sample	Yes	Yes (treated) MS only (untreated)	NA (treated)	Yes	Yes
						Yes (untreated)		
PAHs	8270 or 8100	5 points	Every 12 Hours	Yes	Yes (treated) MS only (untreated)	NA (treated)	Yes	Yes
						Yes (untreated)		

(a) References are to "Methods for Chemical Analysis of Water and Wastes", EPA/600/4-79/020 or "Test Methods for Evaluating Solid Waste", SW-846, 3rd. Ed.

(b) Second column confirmation of positive results is required.  
 NA - Not Applicable

TABLE 7-1. Internal QC Checks for Measurements (continued)

Parameter	Method (a)	Initial Calibration	Calibration Checks	Method Blank	MS/MSD	Triplicate Sample Analysis	QC Sample	Surrogate Spikes
pH	9045/9040	2 points	Every 10th Sample	NA	NA	NA	Yes	NA
Conductivity	9050	1 point	Every 15th Sample	NA	NA	NA	Yes	NA
Cyanide	9010	7 points	Every 15th Sample	Yes	NA	NA	Yes	NA
Phosphorous	365.2	9 points	Every 15th Sample	Yes	NA	NA	Yes	NA
TOC/TIC	9060	3 points	Every 15th Sample	Yes	NA	NA	Yes	NA

(a) References are to "Methods for Chemical Analysis of Water and Wastes", EPA/600/4-79/020 or "Test Methods for Evaluating Solid Waste", SW-846, 3rd. Ed.  
 NA - Not Applicable

TABLE 7-2. Analytical Matrix

Parameters	QC Sample and Method Blank	Untreated Sediment	MS	Tripli- cate	Treated Solids	MS	MSD	Tripli- cate	Water	MS	MSD	Tripli- cate	Oil	MS	Tripli- cate	
Total Solids (Moisture)																
Volatile Solids																
O & G																
Metals																
PCBs																
PAHs																
TOC																
Total Cyanide																
Total Phosphorous																
pH																
BOD																
Total Suspended Solids																
Conductivity																

TABLE 7-3. Example

Parameters	QC Sample and Method Blank	Untreated Sediment	MS	Tripli- cate	Treated Solids	MS	MSD	Tripli- cate	Water	MS	MSD	Tripli- cate	Oil	MS	Tripli- cate	
Total Solids (Moisture)	Yes	I		X	I			X								
Volatile Solids	Yes	I		X	I			X								
O & G	Yes	I		X	I			X								
Metals	Yes	I	X	X	I	X		X								
PCBs	Yes	I	X	X	I	X	X		I				I	X		X
PAHs	Yes	I	X	X	I	X	X		I				I	X		X
TOC	Yes	I			I											
Total Cyanide	Yes	I			I											
Total Phosphorous	Yes	I			I											
pH	Yes	I			I											
BOD																
Total Suspended Solids																
Conductivity																

TABLE 7-4. Analytical and QC Sample Matrix for GLNPO Treatability Studies (numbers of samples)

SAMPLE SET	TOC/TIC		TOTAL SOLIDS		VOL SOLIDS		O & G		TOTAL CYANIDE		TOTAL PHOS		METALS		PCBs		PAH		PH		BOD		TSS		COND		
	S(a)	QC(b)	S	QC	S	QC	S	QC	S	QC	S	QC	S	QC	S	QC	S	QC	S	QC	S	QC	S	QC	S	QC	
SET I																											
Untreated S.	3	-	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	-	-	-	-	-	-
Treated S.	3	-	3	2	3	2	3	2	3	-	3	3	3	2	3	2	3	2	3	2	3	-	-	-	-	-	-
Water	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Oil	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
SET IV																											
Untreated S.	2	7	2	3	2	3	2	3	2	-	2	3	2	3	2	3	2	3	2	3	2	-	-	-	-	-	-
Treated S.	4	-	4	2	4	2	4	2	4	-	4	3	4	2	4	2	4	2	4	2	4	-	-	-	-	-	-
Water	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Oil	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
SET II																											
Untreated S.	1	3	1	3	1	3	1	3	1	3	1	3	1	3	1	3	1	3	1	3	1	3	-	-	-	-	-
Treated S.	1	2	1	2	1	2	1	2	1	3	1	3	1	3	1	2	1	2	1	2	1	2	-	-	-	-	-
Water	1	3	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Oil	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
SET III																											
Untreated S.	1	-	1	3	1	3	1	3	1	-	1	3	1	3	1	3	1	3	1	3	1	-	-	-	-	-	-
Treated S.	1	-	1	2	1	2	1	2	1	-	1	3	1	3	1	2	1	2	1	2	1	-	-	-	-	-	-
Water	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Oil	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
TOTALS																											
Solids	16	5	16	20	16	20	16	20	16	6	16	6	16	24	16	20	16	20	16	5	16	5	-	-	-	-	-
Water	1	3	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Oil	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-

(a) Number of original samples.  
 (b) Number of quality control samples. A "3" represents two additional replicates (triplicate determination) and a spike or control sample analysis resulting in an additional three QC analyses. A "2" represents matrix spike/matrix spike duplicate analysis scheme resulting in an additional two QC analyses. A "1" indicates a blank spike or other control sample analysis resulting in one additional QC analysis.  
 (c) Treated and untreated solids does not apply, and only one control sample per set will be analyzed.

## 8.0 PERFORMANCE AND SYSTEM AUDITS

The laboratory will perform internal reviews by the QA officer or a designee. These reviews should include, as a minimum, periodic checks on the analysts to assess whether they are aware of and are implementing the QA requirements specified in the ARCS QA program.

The laboratory will be prepared to participate in a systems audit to be conducted by the SAIC QA Officer or his designee and/or ARCS QA Officer.

The vendors of the various technologies have all been advised that a number of representatives from SAIC, GLNPO, and other organizations will be present during Phase II of the treatability studies. Thus the ARCS QA officer can be present during Phase II of any or all of the treatability studies.

## 9.0 CALCULATION OF DATA QUALITY INDICATORS

### 9.1 Accuracy

Accuracy for PAHs, PCB and metals will be determined as the percent recovery of matrix spike samples. The percent recovery is calculated according to the following equation:

$$\% R = 100\% \times \frac{C_i - C_o}{C_t}$$

where

- %R = percent recovery
- $C_i$  = measured concentration in spiked sample aliquot
- $C_o$  = measured concentration in unspiked sample aliquot
- $C_t$  = actual concentration for spike added

Accuracy for the other critical measurements will be determined from laboratory control samples according to the equation:

$$\% R = 100\% \frac{C_m}{C_t}$$

where

- %R = percent recovery
- $C_m$  = measured concentration of standard reference material
- $C_t$  = actual concentration for standard reference material

### 9.2 Precision

Precision will be determined from the difference of percent recovery values of MS and MSDs for PAHs and PCBs or triplicate laboratory analyses. The following equations will be used for all parameters:

When 2 values are available:

$$RPD = \frac{[C_1 - C_2] \times 100\%}{[C_1 + C_2] / 2}$$

where

- RPD = Relative percent difference
- C<sub>1</sub> = The larger of two observed values
- C<sub>2</sub> = The smaller of the two observed values

When more than 2 values are available:

$$S = \sqrt{\frac{\sum_{i=1}^N X_i^2 - \frac{1}{N} \left( \sum_{i=1}^N X_i \right)^2}{N - 1}}$$

where

- S = standard deviation
- X<sub>i</sub> = individual measurement result
- N = number of measurements

Relative standard deviation may also be reported. If so, it will be calculated as follows:

$$RSD = 100 \frac{S}{\bar{X}}$$

where

RSD = relative standard deviation, expressed in percent

S = standard deviation

X = arithmetic mean of replicate measurement.

### 9.3 Completeness

Completeness will be calculated as the percent of valid data points obtained from the total number of samples obtained.

$$\% \text{ Completeness} = \frac{\text{VDP}}{\text{TDP}} \times 100$$

where

VDP = number of valid data points

TDP = total number of samples obtained.

## 10.0 CORRECTIVE ACTION

Corrective actions will be initiated whenever quality control limits (e.g., calibration acceptance criteria) or QA objectives (e.g., precision, as determined by analysis of duplicate matrix spike samples) for a particular type of critical measurement are not being met.

Corrective actions may result from any of the following functions:

- Data Review
- Performance evaluation audits
- Technical systems audits
- Interlaboratory/interfield comparison studies

All corrective action procedures consist of six elements:

- Recognition that a Quality Problem exists
- Identification of the cause of the problem
- Determination of the appropriate corrective action
- Implementation of the corrective action
- Verification of the corrective action
- Documentation of the corrective action

For these treatability studies after initial recognition of a data quality problem, the data calculation will be checked first. If an error is found, the data will be recalculated and no further action will be taken. If no calculation error is found, further investigation will be conducted. Depending on the cause and the availability of the appropriate samples, reanalysis or flagging of the original data will be utilized.

All corrective action initiations, resolutions, etc. will be implemented immediately and will be reported in Sections One and Two (Difficulties Encountered and Corrective Actions Taken, respectively) in the existing monthly progress reporting mechanisms established between SAIC, EPA-RREL, GLNPO, AND THE ARCS QA officer and in the QA section of the final report. The QA Manager will determine if a correction action has resolved the QC problem.

## 11.0 QA/QC REPORTS TO MANAGEMENT

This section describes the periodic reporting mechanism, reporting frequencies, and the final project report which will be used to keep project management personnel informed of sampling and analytical progress, critical measurement systems performance, identified problem conditions, corrective actions, and up-to-date results of QA/QC assessments. As a minimum, the reports will include, when applicable:

- Changes to the QA Project Plan, if any.
- Limitations or constraints on the applicability of the data, if any.
- The status of QA/QC programs, accomplishments and corrective actions.
- Assessment of data quality in terms of precision, accuracy, completeness, method detection limit, representativeness, and comparability.
- The final report shall include a separate QA section that summarizes the data quality indicators that document the QA/QC activities that lend support to the credibility of the data and the validity of the conclusions.

For convenience, any QA/QC reporting will be incorporated into the already well-established monthly progress reporting system between SAIC and EPA-RREL for all TESC Work Assignments. In addition, copies of monthly reports will be sent to the ARCS QA officer. Any information pertaining to the above-listed categories will be reported under Sections One through Three (Difficulties Encountered, Corrective Actions Taken, and Current Activities, respectively) in the monthly reports.

**APPENDIX A**  
**TECHNOLOGY SUMMARIES**

### B.E.S.T.<sup>TM</sup> Process Description

The B.E.S.T.<sup>TM</sup> process is a patented solvent extraction technology utilizing triethylamine as the solvent. Triethylamine is an aliphatic amine that is produced by reacting ethyl alcohol and ammonia. The key to success of the B.E.S.T.<sup>TM</sup> process is triethylamine's property of inverse miscibility. At temperatures below 65°F, triethylamine is completely soluble with water. Above this temperature, triethylamine and water are only partially miscible. The property of inverse miscibility can be utilized since cold triethylamine can simultaneously solvate oil and water.

The B.E.S.T.<sup>TM</sup> process produces a single phase extraction solution which is a homogeneous mixture of triethylamine and the water and oil (containing the organic contaminants, such as PCBs, PNAs, and VOCs) present in the feed material. In cases where the extraction efficiencies of other solvent extraction systems are hindered by emulsions, which have the effect of partially occluding the solute (oil containing the organic contaminants), triethylamine can achieve intimate contact at nearly ambient temperatures and pressures. This allows the B.E.S.T.<sup>TM</sup> process to handle feed mixtures with high water content without penalty in extraction efficiency. This process is expected to yield solid, water, and oil residuals.

### Low Temperature Stripping

Low-temperature stripping (LTS) is a means to physically separate volatile and semivolatile contaminants from soil, sediments, sludges, and filter cakes. For wastes containing up to 10% organics or less, LTS can be used alone for site remediation.

LTS is applicable to organic wastes and generally is not used for treating inorganics and metals. The technology heats contaminated media to temperatures between 200-1000°F, driving off water and volatile contaminants. Offgases may be burned in an afterburner, condensed to reduce the volume to be disposed, or captured by carbon adsorption beds. For these treatability studies, only processes that capture the contaminants driven off will

be considered. The process (for these treatability studies) is expected to yield solid, water, and oil residuals.

### Wet Air Oxidation

Wet air oxidation is a process that accomplishes an aqueous phase oxidation of organic or inorganic substances at elevated temperatures and pressures. The usual temperature range varies from approximately 350 to 600°F (175 to 320°C). System pressures of 300 psig to well over 300 psig may be required. However, testing has been done at temperatures exceeding the critical point for water to limit the amount of evaporation of water, depending on the desired reaction temperature. Compressed air or pure oxygen is the source of oxygen that serves as the oxidizing agent in the wet air oxidation process. This process is expected to yield only solid and water residuals.

## APPENDIX D

### B.E.S.T.<sup>®</sup> TREATABILITY STUDY

#### ANALYTICAL METHODS

##### Analytical Methods Used In B.E.S.T.<sup>®</sup> Workups

###### Feed Analysis

Moisture:	Karl Fisher titration or Gravimetric @ 105°C, 16 hrs
Oil & Grease:	Both EPA/SW846 Method 9071 and Methylene Chloride Soxhlet gravimetric (16 hr. extraction)
Ash Content:	Gravimetric @ 550°C for 16 hrs.
Metals Analysis:	Digestion: EPA SW846/3050 or, alternatively, ash digestion at 550°C, followed by heating with nitric acid Analysis: EPA SW846/6010
Polynuclear Aromatic Hydrocarbons*:	EPA SW846/8100 (Methylene Chloride Extraction)
Benzene, Toluene, Xylene*:	EPA SW846/8020
PCB*:	EPA SW846/8080, Method 3540 extraction (soxhlet extraction with 1:1 acetone:hexane for 16 hours)

###### Product Analysis

###### Product Water

Total Solids:	Standard methods 209A
Total Dissolved Solids:	Standard methods 209B
Total Organic Carbon:	EPA 600/415.2
Oil & Grease:	EPA 600/413.1 or 413.2 depending on level of sample and quantity of sample available
Triethylamine:	See the attached RCC method
Metals:	Digestion: SW846/3005 Analysis: SW846/6010

\* If present

# GC METHODS FOR TRIETHYLAMINE (TEA) ANALYSIS IN AQUEOUS SOLUTIONS, SOLIDS AND OILS

## I Summary of Methods

Triethylamine (TEA) can be determined using Gas Chromatography with flame ionization detection. Aqueous solutions can be injected directly onto a packed column after pH adjustment and filtration. Solids are extracted into water without pH adjustment and then are analyzed using the same column and parameters as aqueous solutions. Oils are dissolved in a solvent (typically methylene chloride) and analyzed, using a megabore HP-1 Methyl Silicone column.

## II TEA In Aqueous Solutions

### A. Equipment and Operating Parameters

1. Gas Chromatograph: Hewlett Packard 5890A with 3392A Integrator
2. Column: 4% Carbowax-20M, 0.8% KOH, 60/80 Carbopack B
3. Injector Temp.: 200°C
4. Detector: Flame Ionization Detector (FID), set at 300°C
5. Oven Temperature and Time:  
Initial Temp: 90°C, Initial Time: 0 minutes  
Final Temp: 170°C, Final Time: 30 minutes  
Rate: 5°/min.
6. Column Flow: ~30ml/min.
7. TEA Peak Retention Time: Approximately 8 minutes

### B. Procedure

1. Standardization
  - a) Inject 1 microliter of 73 mg/l TEA. This standard is prepared by serial dilution from pure TEA (successive 1:100 dilutions from pure TEA, which is 730,000 mg/l TEA).
  - b) Use the peak area at approximately 8 min. retention time to quantitate TEA. Repetitive standards injections should agree to within 10%.

### **Product Oil**

**Triethylamine:** See the attached RCC method

**Viscosity:** Brookfield

**Water:** Karl Fisher titration

**Suspended Solids:** Filtration/Gravimetric (Whatman GF/C)

**Metals:** Product oil is diluted 1:10 with Xylene and filtered through GF/C filter, then analyzed with organometallic standards on ICP or, alternatively, ash digestion at 550°C, followed by heating with nitric acid

### **Product Solids**

**Residual Triethylamine:** See the attached RCC method

**Oil & Grease:** Both SW846/9071 and Methylene Chloride Soxhlet gravimetric (16 hr extractions)

**Metals:** Digestion: 1 gm sample refluxed with 15 mls Aqua Regia  
Analysis: SW846/6010

**TCLP:** SW846/1311 followed by 3010 & 6010

**Polynuclear Aromatic Hydrocarbons\*:** EPA SW846/8100 (Methylene Chloride Extraction)

**Benzene, Toluene, Xylene\*:** EPA SW846/8020

**PCB\*:** EPA SW846/8080, Method 3540 extraction (soxhlet extraction with 1:1 acetone:hexane for 16 hours)

\* If present

## 2. Sample Preparation and Analysis

- a) If necessary, dilute the sample in deionized, distilled water until the TEA concentration is at or below 73 ppm and record the dilution factor.
- b) Inject 1 microliter sample.
- c) Inject a standard at least once every 10 samples and at the end of an analytical sequence.

## 3. Quantitation

Quantitate the TEA using direct comparison of peak area.

$$\frac{\text{Peak Area of Standard}}{\text{mg/l Standard}} = \frac{\text{Peak Area of Sample}}{\text{(mg/l of Sample)}} \times (\text{dilution factor})$$

## III TEA in Solids

### A. Equipment and Operating Parameters

The same equipment and parameters are used as in aqueous solutions. Additional equipment includes the following:

1. Shaker Bath (example: Forma Scientific model 2564).
2. 50 ml Erlenmeyer flasks.
3. Cover with Parafilm.

### B. Procedure

1. Standardization: as in II.B.1.
2. Sample preparation and analysis.
  - a. Weigh out 3-5 g solids into an Erlenmeyer flask. Record exact weight.
  - b. Add 25 ml distilled water.
  - c. Cover with 1 layer of parafilm.

- d. In the shaker bath, shake vigorously at ambient temperature for 1 hour.
- e. Let the mixture stand quiescent allowing the solids to settle.
- f. Continue as in II.B.2.

### 3. Quantitation

Quantitation is the same as that for aqueous solutions, correcting for the extraction of the solids into the water.

$$\frac{\text{Peak Area of Standard}}{\mu\text{g/g TEA in Standard}} = \frac{\text{Peak Area of Extract}}{\mu\text{g/g TEA of Extract}}$$

$$\mu\text{g/gm TEA in Solids} = (\mu\text{g/ml TEA in Extract}) \times \frac{(\text{mls of Extraction Water})}{(\text{g of Solids})}$$

## IV TEA In Oil

### A. Equipment and Operating Parameters

1. Gas Chromatograph: Hewlett Packard 5890 with 3392A Integrator
2. Column: Megabore 15m x .53mm J&W BD1 Methyl Silicone
3. Injector Temp: 200°C
4. Detector: Flame Ionization Detector (FID), set at 300°C
5. Oven Temperature and Time:  
Initial Temp: 35°C, Initial Time: 12 minutes  
Final Temp: 250°C, Final Time: 20 minutes  
Rate: 25°C/min.
6. Column Flow: 2 ml/min.
7. Makeup Gas: 20 ml/min.
8. TEA Peak Retention Time: Approximately 7-8 min.

## B. Procedure

### 1. Standardization

- a) Inject 1 microliter 73 mg/l TEA (dissolved in GC grade methylene chloride). This standard is prepared by serial dilution from pure TEA (successive 1:100 dilutions into methylene chloride from pure TEA, which is 730,000 mg/l TEA).
- b) Use the peak area at approximately 7-8 minutes retention time to quantitate TEA. Repetitive standard injections should agree to within 10%.

### 2. Sample Preparation and Analysis

- a) Dissolve the oil in methylene chloride such that the TEA concentration is at or below 73 ppm.
- b) Inject 1 microliter sample.
- c) Inject a standard at least once every 10 samples.

### 3. Quantitation

Quantitate the TEA using direct comparison of peak area.

$$\frac{\text{Peak Area of Standard}}{\text{mg/l of Standard}} = \frac{\text{Peak Area of Sample}}{\text{mg/l of Sample}}$$



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METALS IN UNTREATED SEDIMENT

(Concentrations in ug/g dry weight)

MSL Code	Sponsor ID	Ag		As		Ba		Cd		Cr		Cu		%Fe		Hg		Mn		Ni		Pb		Se		Zn	
		AA	AF	XF	XF	AF	AF	AA	AA	AF	AF	AF	AF	AF	AF	AF	CVAA	AF	AF	AF	AF	AF	AF	AA	AA	AF	AF
MDL		0.007	2.5	2.5	43	0.006	3.3	5.5	0.26	0.0003	5.6	7.5	6.2	0.22	7.8												
361-5, Rep 1	S-US-RCC, Rep 1	0.87	2.5 U	322	4.39	93	55.8	0.780	0.162	162	58.1	43.0	0.22 U	125.7													
361-5, Rep 2	S-US-RCC, Rep 2	0.85	2.65	322	4.08	112	57.3	0.795	0.178	159	55.2	42.5	0.22 U	132.6													
361-5, Rep 3	S-US-RCC, Rep 3	0.80	2.5 U	321	3.96	117	63.4	0.816	0.160	173	61.5	50.9	0.22 U	162.1													
361-6	B-US-RCC	0.31	12.7	413	2.10	109	70.2	4.290	0.551	667	43.1	101.9	0.74	190.3													
361-7	I-US-RCC	4.84	22.8	317	8.58	2270	188	18.770	1.528	3230	12.9	582.0	0.22 U	2380													
Method Blank		0.016	NA	NA	0.006	NA	NA	NA	0.00086	NA	NA	NA	0.22 U	NA													

STANDARD REFERENCE MATERIAL

1646 SRM	certified value	0.126	11.3	406	0.42	78	19.8	3.36	0.066	339	31.1	26.1	0.75	126.3														
		NC	11.6	NC	0.36	76	18	3.35	0.063	375	32	28.2	NC	138														
		NC	±1.3	NC	±0.07	±3	±3	±0.1	±0.012	±20	±3	±1.8	NC	±6														

MATRIX SPIKE RESULTS

Amount Spiked	2	NS	NS	NS	2	NS	NS	NS	1.972	NS	NS	NS	2.72	NS														
361-5*	0.84	NS	NS	NS	4.15	NS	NS	NS	0.166	NS	NS	NS	0.22 U	NS														
361-5 + Spike	3.33	NS	NS	NS	6.43	NS	NS	NS	2.207	NS	NS	NS	3.10	NS														
Amount Recovered	2.49	NS	NS	NS	2.28	NS	NS	NS	2.041	NS	NS	NS	3.10	NS														
Percent Recovery	125%	NS	NS	NS	114%	NS	NS	NS	103%	NS	NS	NS	114%	NS														

REPLICATE ANALYSES

361-5, Rep 1	S-US-RCC, Rep 1	0.87	1.86	322	4.39	93	55.8	0.780	0.162	162	58.1	43.0	0.22 U	125.7															
361-5, Rep 2	S-US-RCC, Rep 2	0.85	2.65	322	4.08	112	57.3	0.765	0.178	159	55.2	42.5	0.22 U	132.6															
361-5, Rep 3	S-US-RCC, Rep 3	0.80	2.12	321	3.96	117	63.4	0.816	0.160	173	61.5	50.9	0.22 U	162.1															
	FSD%	4%	18%	0%	5%	12%	7%	3%	6%	4%	5%	10%	NA	14%															

J - Values detected below MDL.

U - Below detection limit

NA - Not analyzed/applicable

\* - Mean of triplicated sample

NS - Not spiked

NOTE: All Metals results are "blank corrected."

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CONVENTIONALS IN TREATED SEDIMENT

MSL Code	Sponsor ID	% Moisture	pH	% Total Volatile Solids	Oil & Grease (mg/kg)	TOC % weight	Total Cyanide (mg/kg)	Total Phosphorus (mg P/kg)
MDL		0.01%	NA	0.001%	0.1	0.007%	0.001	0.001
361-8, Rep 1	S-TS-RCC, Rep 1	0.16%	10.73	1.76%	297	0.58%	50.72	63
361-8, Rep 2	S-TS-RCC, Rep 2	NA	NA	1.70%	293	NA	NA	NA
361-8, Rep 3	S-TS-RCC, Rep 3	NA	NA	1.74%	208	NA	NA	NA
361-9	B-TS-RCC	3.72%	10.30	3.91%	238	1.21%	8.11	20
361-10	I-TS-RCC	0.50%	10.25	9.06%	470	13.36%	68.5	7214
Method Blank		NA	NA	NA	0.1	0.011%	0.001 U	0.089

STANDARD REFERENCE MATERIAL

MESS-1 SRM		NA	NA	NA	NA	2.45	NA	NA
In-house Concensus Value *		NA	NA	NA	NA	2.3	NA	NA

REPLICATE ANALYSES

361-8, Rep 1	S-TS-RCC, Rep 1	0.16%	10.73	1.76%	297	0.58%	50.72	63
361-8, Rep 2	S-TS-RCC, Rep 2	NA	NA	1.70%	293	NA	NA	NA
361-8, Rep 3	S-TS-RCC, Rep 3	NA	NA	1.74%	208	NA	NA	NA
	RSD%	NA	NA	2%	19%	NA	NA	NA

NA = Not analyzed

U = Below detection limit

\* = TOC value for MESS determined based on past In-house analyses. Not a statistical determination.

# = Mean for replicated sample.

NOTE: All Conventional results are reported on a dry weight basis.

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METALS IN TREATED SEDIMENT

(Concentrations in ug/g dry weight)

MSL Code	Sponsor ID	Ag		As		Ba		Cd		Cr		Cu		%Fe		Hg		Mn		Ni		Pb		Se		Zn			
		AA	MF	MF	MF	AA	MF	AA	MF	AA	MF	AA	MF	MF	CVAL	MF	AA	MF											
MDL		0.007	2.5	4.3	0.006	3.3	5.5	0.0003	5.8	7.5	6.2	0.22	7.8																
361-8, Rep 1	S-TS-RCC, Rep 1	1.03	2.77	325	4.38	130	68.6	0.505	181	71.5	53.3	0.22 U	194.0																
361-8, Rep 2	S-TS-RCC, Rep 2	0.76	2.87	321	4.17	113	68.3	0.290	177	64.1	45.0	0.22 U	165.1																
361-8, Rep 3	S-TS-RCC, Rep 3	0.67	2.92	310	4.24	110	57.4	0.209	172	57.2	41.4	0.22 U	147.0																
361-9	B-TS-RCC	0.24	14.6	398	2.11	113	61.2	4.42	684	42.1	101.5	0.87	189.7																
361-10	I-TS-RCC	4.34	29.0	290	6.97	1708	223	8.25	2540	10 U	656.0	4.94	2810.0																
Method Blank		0.020	NA	NA	0.008	NA	NA	NA	0.00013	NA	NA	0.22 U	NA																

STANDARD REFERENCE MATERIAL

1646 SRM	certified value	0.113	13.1	411	0.42	65	22.3	3.47	350	36.1	28.6	0.74	133.4																		
		NC	11.6	NC	0.38	76	18	3.35	375	32	28.2	NC	138																		
		NC	±1.3	NC	±0.07	±3	±3	±0.1	±20	±3	±1.8	NC	±6																		

MATRIX SPIKE RESULTS

Amount Spiked		2	NS	NS	2	NS	NS	NS	1.987	NS	NS	2.74	NS																		
361-8*		0.82	NS	NS	4.26	NS	NS	NS	0.335	NS	NS	0.22 U	NS																		
361-8 + Spike		3.22	NS	NS	6.25	NS	NS	NS	2.282	NS	NS	2.99	NS																		
Amount Recovered		2.4	NS	NS	1.99	NS	NS	NS	1.947	NS	NS	2.99	NS																		
Percent Recovery		120%	NS	NS	100%	NS	NS	NS	98%	NS	NS	109%	NS																		

REPLICATE ANALYSES

361-8, Rep 1	S-TS-RCC, Rep 1	1.03	2.77	325	4.38	130	68.6	0.855	181	71.5	53.3	0.22 U	194.0																			
361-8, Rep 2	S-TS-RCC, Rep 2	0.76	2.87	321	4.17	113	68.3	0.827	177	64.1	45.0	0.22 U	165.1																			
361-8, Rep 3	S-TS-RCC, Rep 3	0.67	2.92	310	4.24	110	57.4	0.795	172	57.2	41.4	0.22 U	147.0																			
	RSD%	23%	3%	2%	3%	8%	8%	4%	46%	11%	13%	NA	14%																			

J - Values detected below MDL.

U - Below detection limit

NA - Not analyzed/applicable

\* - Mean of triplicated sample

NS - Not spiked

NOTE: All Metals results are "blank corrected."

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PAH IN UNTREATED SEDIMENT

MSL Code	Sponsor ID	Naphthalene	Acenaphthylene	Acenaphthene	Fluorene	Phenanthrene	Anthracene
361-5, Rep 1 R	S-US-RCC, Rep 1	26 B	15	19	33	249 B	64
361-5, Rep 2 R	S-US-RCC, Rep 2	24 B	14 U	20 U	32	302 B	70
361-5, Rep 3 R	S-US-RCC, Rep 3	27 B	18	22 U	33	249 B	63
361-6	B-US-RCC	107 B	79 U	113 U	160	1018 B	547
361-7	I-US-RCC	4402 B	2322	4396	4819	15152 B	5630
Method Blank-3		581	58 U	83 U	73 U	100	64 U
Method Blank-R		11	11 U	16 U	13 U	9	9 U

STANDARD REFERENCE MATERIAL

SRM-NIST1941	certified value	63	550	164
		NC	577	202

MATRIX SPIKE RESULTS

Amount Spiked	361-5 #	361-5 + Spike R	Amount Recovered	Percent Recovery
933	26	406	380	41%
16 < 18	577	562	577	69%
933	90 < 22	535	545	62%
933	33	628	595	57%
933	267	600	64%	63%

- R - Re-extracted sample results.
- # - Mean of triplicated sample.
- B - Analyte detected in Blank associated with sample.
- U - Below detection limits.
- NC - Not certified.
- \* - Value outside of Internal QC limits (40-120%).

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PAH IN UNTREATED SEDIMENT

High Molecular Weight PAHs (ng/g dry wt)

MSL Code	Sponsor ID	Fluoranthene	Pyrene	Benzo(a)anthracene	Chrysene	Benzo(b)fluoranthene	Benzo(k)fluoranthene	Benzo(a)pyrene	Indeno(1,2,3-c,d)pyrene	Dibenz(a,h)anthracene	Benzo(g,h,i)perylene
361-5, Rep 1 R	S-US-RCC, Rep 1	376 B	425 B	184	265 B	238 B	180 B	224 B	201 B	44	113 B
361-5, Rep 2 R	S-US-RCC, Rep 2	464 B	491 B	210	299 B	270 B	200 B	250 B	231 B	47	129 B
361-5, Rep 3 R	S-US-RCC, Rep 3	351 B	402 B	163	242 B	217 B	158 B	200 B	180 B	30	109 B
361-6	B-US-RCC	1197 B	1157 B	861	1037	878	733	887	607	205	495
361-7	I-US-RCC	32032 B	32022 B	18282	24399	19107	13350	20581	14853	5224	13767
Method Blank-3		44	39	37 U	36 U	27 U	23 U	30 U	27 U	35 U	26 U
Method Blank-R		9	9	5 U	5	6	5	5	5	4 U	6

STANDARD REFERENCE MATERIAL

SRM-NIST1941	certified value	1114	1034	481	703	766	603	500	498	141	421
		1220	1080	550	NC	780	444	670	569	NC	516

Amount Spiked

$\bar{x}$

361-5 #

361-5 + Spike R

Amount Recovered

Percent Recovery

R - Re-extracted sample results.

# - Mean of triplicated sample

B - Analyte detected in Blank associated with sample.

U - Below detection limits.

NC - Not certified.

. - Value outside of internal QC limits (40-120%).

933	933	933	933	933	933	933	933	933	933	933	933
387	439	186	269	242	179	225	207	225	207	43	117
1075	1077	919	958	1044	648	987	648	987	1095	912	680
678	637	733	688	802	669	762	669	762	888	889	543
73%	68%	79%	74%	86%	72%	82%	72%	82%	95%	93%	58%

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PAH IN UNTREATED SEDIMENT

MSL Code	Sponsor ID	Surrogate Recovery %		
		DB Naphthalene	D10 Acenaphthalene	D12 Perylene
361-5, Rep 1 R	S-US-RCC, Rep 1	37%*	53%	79%
361-5, Rep 2 R	S-US-RCC, Rep 2	34%*	47%	83%
361-5, Rep 3 R	S-US-RCC, Rep 3	37%*	50%	76%
361-6	B-US-RCC	25%*	45%	84%
361-7	I-US-RCC	27%*	48%	80%
Method Blank-3		26%*	26%*	90%
Method Blank-R		51%	62%	72%

STANDARD REFERENCE MATERIAL

SRM-NIST1941 28%\* 47% 74%

MATRIX SPIKE RESULTS

Amount Spiked	NA	NA	NA
361-5 #	36%	50%	79%
361-5 + Spike R	40%	54%	88%
Amount Recovered	NA	NA	NA
Percent Recovery	NA	NA	NA

R = Re-extracted sample results.  
\* = Value outside of Internal QC limits (40-120%).  
NA = Not applicable.

SAIC-GLNPO (CF #361)

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PAH IN TREATED SEDIMENT

Low Molecular Weight PAHs (ng/g dry wt.)

MSL Code	Sponsor ID	Naphthalene	Acenaphthylene	Acenaphthene	Fluorene	Phenanthrene	Anthracene
361-8 R	S-TS-RCC	24 B	14 U	19 U	16 U	99 B	17
361-9 R	B-TS-RCC	37 B	15 U	21 U	18 U	68 B	30
361-10	I-TS-RCC	2253	121	726	1078	5642	1474
Method Blank-4		38 U	39 U	56 U	49 U	33 U	36 U
Method Blank-R		11	11 U	16 U	13 U	9	9 U

STANDARD REFERENCE MATERIAL

SRM-NIST1941	certified value	54	60 U	63	550	165
		NC	NC	NC	577	202

MATRIX SPIKE RESULTS

Amount Spiked	2222	2222	2222	2222	2222	2222	2222
361-8	24	14 U	18 U	16 U	99	17	17
361-8 + Spike R	723	1004	933	1185	2052	1658	1658
Amount Recovered	699	1004	933	1185	1954	1642	1642
Percent Recovery	31%	45%	42%	53%	86%	74%	74%
Amount Spiked	1799	1799	1799	1799	1799	1799	1799
361-8 DUP	24	14 U	19 U	16 U	99	17	17
361-8 + Spike DUP R	999	1262	1149	1281	1468	1370	1370
Amount Recovered	975	1249	1130	1265	1369	1353	1353
Percent Recovery	54%	69%	63%	70%	76%	75%	75%

R = Re-extracted sample results.

U = Below detection limits

NC = Not certified

. = Value outside of Internal QC limits (40-120%)

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PAH IN TREATED SEDIMENT

High Molecular Weight PAHs (ng/g dry wt.)

MSL Code	Sponsor ID	Fluoranthene	Pyrene	Benzo(a)anthracene	Chrysene	Benzo(b)fluoranthene	Benzo(k)fluoranthene	Benzo(e)pyrene	Indeno(1,2,3-c,d)pyrene	Dibenzo(a,h)anthracene	Benzo(g,h,i)perylene
361-8 R	S-TS-RCC	138 B	120 B	67	68 B	97 B	66 B	76 B	90 B	16	60 B
361-9 R	B-TS-RCC	45 B	39 B	22	39 B	27 B	4 B	18 B	18 B	6	14 B
361-10	I-TS-RCC	3105	3553	3128	3992	1887	1316	3220	1360	1930	2354
Method Blank-R		25 U	26 U	25 U	24 U	18 U	16 U	20 U	19 U	24 U	18
Method Blank-R		9	9	5 U	5	6	5	5	5	4 U	6

STANDARD REFERENCE MATERIAL

SRM-NIST1941	certified value	1114	1034	481	703	768	603	500	498	141	421
		1220	1080	550	NC	780	444	670	569	NC	516

MATRIX SPIKE RESULTS

Amount Spiked	2222	2222	2222	2222	2222	2222	2222	2222	2222	2222	2222
361-8R	138	120	57	68	97	66	76	90	16	60	60
361-8 + Spike R	2855	2555	2654	2589	2870	2367	2684	2939	2810	1766	1766
Amount Recovered	2717	2434	2506	2502	2773	2301	2508	2849	2594	1726	1726
Percent Recovery	122%*	110%	117%	113%	125%*	104%	113%	128%*	117%	78%	78%
Amount Spiked	1799	1799	1799	1799	1799	1799	1799	1799	1799	1799	1799
361-8 R DUP	138	120	57	68	97	66	76	90	16	60	60
361-8 + Spike DUP R	1689	1594	1611	1611	1734	1509	1506	1703	1639	1065	1065
Amount Recovered	1561	1473	1632	1523	1637	1443	1430	1613	1622	1005	1005
Percent Recovery	87%	82%	91%	85%	91%	80%	79%	90%	90%	56%	56%

R - Re-extracted sample results.

U - Below detection limits

NC - Not certified

\* - Value outside of internal QC limits (40-120%)

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PAH IN TREATED SEDIMENT

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MSL Code	Sponsor ID	Surrogate Recovery %		
		D8 Naphthalene	D10 Acenaphthalene	D12 Perylene
361-8 R	S-TS-RCC	41%	46%	67%
361-9 R	B-TS-RCC	31%*	43%	73%
361-10	I-TS-RCC	25%*	54%	85%
Method Blank-R		21%*	19%*	50%
Method Blank-R		51%	62%	72%

STANDARD REFERENCE MATERIAL

SRM-NIST1941

28%\* 47% 74%

MATRIX SPIKE RESULTS

Amount Spiked	NA	NA	NA
361-8R	41%	46%	67%
361-8 + Spike R	31%*	40%	108%
Amount Recovered	NA	NA	NA
Percent Recovery	NA	NA	NA
Amount Spiked	NA	NA	NA
361-8 R DUP	41%	46%	67%
361-8 + Spike DUP R	56%	64%	87%
Amount Recovered	NA	NA	NA
Percent Recovery	NA	NA	NA

R = Re-extracted sample results.

\* = Values outside of Internal QC limits (40-120%).

NA = Not applicable.

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PAH IN WATER

Low Molecular Weight PAHs (ng/L)		Sponsor ID					
MSL Code		Naphthalene	Acenaphthylene	Acenaphthene	Fluorene	Phenanthrene	Anthracene

361-1	S-WR-RCC	1441 B	538 U	679 U	641 U	421 U	508 U
361-2	B-WR-RCC	988 B	342 U	432 U	408 U	288 U	323 U
361-11	I-WR-RCC	301 B	495	165	278	2719	997

Method Blank-2

		1767	963 U	1216 U	1148 U	754 U	910 U
--	--	------	-------	--------	--------	-------	-------

**MATRIX SPIKE RESULTS**

Amount Spiked	5000	5000	5000	5000	5000	5000	5000
Blank-2	1767	963 U	1216 U	1148 U	754 U	910 U	
Blank-2 + Spike	2552	1072	1143 U	1108	1504	1420	
Amount Recovered	785	1072	1143 U	1108	1504	1420	
Percent Recovery	16%*	21%*	0%*	22%*	30%*	28%*	

B - Analyte present in method blank associated with sample.

U - Below detection limits

\* - Value outside of Internal QC limits (40-120%).

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PAH IN WATER

MSL Code	Sponsor ID	Surrogate Recovery %		
		D8 Naphthalene	D10 Acenaphthalene	D12 Perylene
361-1	S-WR-RCC	39%	44%	130%
361-2	B-WR-RCC	35%	37%	111%
361-11	I-WR-RCC	20%	28%	90%

Method Blank-2 37% 36% 83%

**MATRIX SPIKE RESULTS**

Amount Spiked	NA	NA	NA
Blank-2	37%	38%	83%
Blank-2 + Spike	20%	20%	109%
Amount Recovered	NA	NA	NA
Percent Recovery	NA	NA	NA

\* - Value outside of Internal QC limits (40-120%).  
NA - Not applicable.

SAIC-GLNPO (CF #361)

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PAH IN WATER

High Molecular Weight PAHs (ng/L)

MSL Code	Sponsor ID	Fluoranthene	Pyrene	Benzo(a)anthracene	Chrysene	Benzo(b)fluoranthene	Benzo(k)fluoranthene	Benzo(e)pyrene	Indeno(1,2,3-c,d)pyrene	Dibenzo(a,h)anthracene	Benzo(g,h,i)perylene
361-1	S-WR-RCC	402	405 U	436 U	360 U	361 U	290 U	387 U	348 U	377 U	289
361-2	B-WR-RCC	256 U	258 U	269 U	242	230 U	185 U	247 U	221 U	240 U	184
361-11	I-WR-RCC	17056	17998	9418	10879	8796	3968	6181	3235	762	2841

Method Blank-2 721 U 726 U 813 U 680 U 648 U 520 U 694 U 623 U 676 U 618 U

MATRIX SPIKE RESULTS

Amount Spiked	5000	5000	5000	5000	5000	5000	5000	5000	5000	5000	5000
Blank-2	721 U	726 U	813 U	680 U	648 U	520 U	694 U	623 U	676 U	618 U	519
Blank-2 + Spike	3816 %	3839 %	6978	5752	8829	5579	4794	5886	5911	5911	4854
Amount Recovered	3816 %	3839 %	6978	5752	8829	5579	4794	5886	5911	5911	4336
Percent Recovery	76%	77%	140%*	115%	133%*	112%	96%	114%	116%	116%	87%

\* - Analyte present in method blank associated with sample.

U - Below detection limits

\* - Value outside of Internal QC limits (40-120%).

## PAH IN OIL

Low Molecular Weight PAHs (ng/ml)		Sample Density (g/ml)	Sample Density (g/ml)					
MSL Code	Sponsor ID		Naphthalene	Acenaphthylene	Acenaphthene	Fluorene	Phenanthrene	Anthracene
361-3	S-OR-RCC	0.7525	603 U	764 U	965 U	1250	11800	7210
361-4 R	B-OR-RCC	0.6903	369 U	396 U	557 U	1063	6943	5465
361-12, Rep 1	I-OR-RCC, Rep 1	0.7301	12127 DU	20391 DU	18291 DU	21142 D	92741 D	47155 D
361-12, Rep 2	I-OR-RCC, Rep 2	0.7301	11178 DU	22779 D	22184 D	24980 D	97299 D	51320 D
361-12, Rep 3	I-OR-RCC, Rep 3	0.7301	11539 DU	19859 D	18557 D	21992 D	90220 D	45668 D
Method Blank			1774 DU	1902 U	2675 DU	2241 DU	1312 DU	1469 DU

## OIL CONCENTRATIONS ON % OIL BASIS

Low Molecular Weight PAHs (ug/kg oil)		% Oil (%)	% Oil (%)					
MSL Code	Sponsor ID		Naphthalene	Acenaphthylene	Acenaphthene	Fluorene	Phenanthrene	Anthracene
361-3	S-OR-RCC	9.25	8664 U	10977 U	13865 U	17960	169540 D	103592 D
361-4 R	B-OR-RCC	6.29	8508 U	9121 U	12832 U	24483	159968	125930
361-12, Rep 1	I-OR-RCC, Rep 1	59.98	27694 DU	46565 D	41770 DU	48280 D	211786 D	107884 D
361-12, Rep 2	I-OR-RCC, Rep 2	59.98	25526 DU	52019 D	50860 D	57045 D	222195 D	117214 D
361-12, Rep 3	I-OR-RCC, Rep 3	59.98	28351 DU	45351 D	42377 D	50222 D	208029 D	104289 D

## MATRIX SPIKE RESULTS

Amount Spiked	50000	50000	50000	50000	50000	50000	50000	50000
361-3	603 U	764 U	965 U	1250	11800	7210	11800	7210
361-3 + Spike	6280	16800	19300	32400	56700	61800	56700	61800
Amount Recovered	5280	16800	19300	31150	44900	54500	44900	54500
Percent Recovery	11%*	34%*	39%*	62%	90%	89%	90%	109%

R - Re-extracted sample results.

D - Samples diluted 1:10 and re-run.

U - Below detection limits.

\* - Outside of Internal QC limits (40-120%).

## PAH IN OIL

MSL Code	Sponsor ID	Sample Density (g/ml)	High Molecular Weight PAHs (ng/ml)									
			Fluoranthene	Pyrene	Benzo(e)anthracene	Chrysene	Benzo(b)fluoranthene	Benzo(k)fluoranthene	Benzo(a)pyrene	Indeno(1,2,3-c,d)pyrene	Dibenzo(a,h)anthracene	Benzo(g,h,i)perylene
361-3	S-OR-RCC	0.7525	19500	17900	6070	9980	9710	6430	7920	6300	2160	4840
361-4 R	B-OR-RCC	0.6903	8715	7932	3679	5229	3771	2968	3684	2928	579	1708 09
361-12, Rep 1	I-OR-RCC, Rep 1	0.7301	277651 D	266075 D	151390 D	210791 D	189639 D	126840 D	189962 D	159145 D	32883 D	96127 D
361-12, Rep 2	I-OR-RCC, Rep 2	0.7301	287695 D	274485 D	160159 D	220510 D	199895 D	129943 D	199435 D	165497 D	35296 D	98294 D
361-12, Rep 3	I-OR-RCC, Rep 3	0.7301	270489 D	258486 D	146980 D	204599 D	177787 D	122228 D	180974 D	150758 D	30903 D	90713 D

Method Blank

898 DU 895 DU 845 DU 805 DU 645 DU 529 DU 688 DU 707 DU 694 DU 1430 D

## OIL CONCENTRATIONS ON % OIL BASIS

MSL Code	Sponsor ID	% Oil (%)	Low Molecular Weight PAHs (ug/kg oil)									
			Fluoranthene	Pyrene	Benzo(e)anthracene	Chrysene	Benzo(b)fluoranthene	Benzo(k)fluoranthene	Benzo(a)pyrene	Indeno(1,2,3-c,d)pyrene	Dibenzo(a,h)anthracene	Benzo(g,h,i)perylene
361-3	S-OR-RCC	9.25	280172	257184	115948	143391	125144	92385	113793	90517	31034	69540
361-4 R	B-OR-RCC	6.29	200814	182759	89370	120480	86890	86386	84679	87455	13335	39357
361-12, Rep 1	I-OR-RCC, Rep 1	59.98	634051 D	607616 D	345718 D	481388 D	433085 D	289955 D	433802 D	363428 D	75092 D	219518 D
361-12, Rep 2	I-OR-RCC, Rep 2	59.98	656988 D	626776 D	365743 D	503562 D	456485 D	296741 D	453151 D	377933 D	80603 D	224467 D
361-12, Rep 3	I-OR-RCC, Rep 3	59.98	617696 D	590240 D	335647 D	467228 D	405999 D	279123 D	413277 D	344275 D	70571 D	207155 D

## MATRIX SPIKE RESULTS

Amount Spiked	50000	50000	50000	50000	50000	50000	50000	50000	50000	50000	50000	50000
361-3	19500	17900	8070	9980	8710	6430	7920	6300	2160	4840	1708 09	4840
361-3 + Spike	83600	77500	78600	66100	75800	64300	72700	77100	81400	64700	81400	64700
Amount Recovered	64100	59600	70530	56120	67090	64780	70800	70240	59860	79240	59860	59860
Percent Recovery	128%*	119%	141%*	112%	134%*	116%	130%*	142%*	158%*	120%	158%*	120%

R - Re-extracted sample results.

D - Samples diluted 1:10 and re-run.

U - Below detection limits.

\* - Outside of Internal QC limits (40-120%).

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PAH IN OIL

MSL Code	Sponsor ID	Surrogate Recovery %		
		D8 Naphthalene	D10 Acenaphthalene	D12 Perylene
361-3	S-OR-RCC	6%*	46%	161%
361-4 R	B-OR-RCC	22%*	39%*	95%
361-12, Rep 1	I-OR-RCC, Rep 1	23% D*	61% D	123% D*
361-12, Rep 2	I-OR-RCC, Rep 2	30% D	63% D	106% D
361-12, Rep 3	I-OR-RCC, Rep 3	19% D*	59% D	108% D
Method Blank		60% D	118% D	73% D

OIL CONCENTRATIONS ON % OIL BASIS

Low Molecular Weight PAHs (ug/kg oil)

MSL Code	Sponsor ID	Surrogate Recovery %		
		D8 Naphthalene	D10 Acenaphthalene	D12 Perylene
361-3	S-OR-RCC	6%*	46%	161%
361-4 R	B-OR-RCC	22%*	39%*	95%
361-12, Rep 1	I-OR-RCC, Rep 1	23% D*	61% D	123% D*
361-12, Rep 2	I-OR-RCC, Rep 2	30% D	63% D	106% D
361-12, Rep 3	I-OR-RCC, Rep 3	19% D*	59% D	108% D

MATRIX SPIKE RESULTS

Amount Spiked	NA	NA	NA
361-3	6%*	46%	161%
361-3 + Spike	11%*	38%*	139%
Amount Recovered	NA	NA	NA
Percent Recovery	NA	NA	NA

R = Re-extracted sample results.

D = Samples diluted 1:10 and re-run.

\* = Outside of Internal QC limits (40 : 20%).

NA = Not applicable.

RE-PROCESSED RESULTS (1/92)

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PCBs IN UNTREATED SEDIMENT

Concentrations in ug/kg dry weight

MSL Code	Sponsor ID	Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260	Tetrachloro-m-Xylene	Octachloro-naphthalene
361-5, Rep 1	S-US-RCC, Rep 1	25372	200 U	2591 E	100 U	60.9%	84.2%
361-5, Rep 2	S-US-RCC, Rep 2	18249	200 U	1691 E	100 U	58.0%	94.1%
361-5, Rep 3	S-US-RCC, Rep 3	17666	200 U	2025 E	100 U	59.6%	89.5%
361-6	B-US-RCC	200 U	325	100 U	100 U	63.3%	66.2%
361-7	I-US-RCC	200 U	11257	3746 E	100 U	61.7%	57.1%
Blank-3		200 U	200 U	100 U	100 U	256.1%*	122.7%*

STANDARD REFERENCE MATERIAL

SRM-1 (HS-2)

certified value

MATRIX SPIKE RESULTS

Amount Spiked	40 U	40 U	132	127	42.4%
361-5#	NS	NS	6098	NS	NA
361-5 + Spike	NS	NS	2102	NS	89.3%
Amount Recovered	NS	NS	8129	NS	61.0%
Percent Recovery	NS	NS	4027	NS	NA
	NS	NS	66%	NS	NA
Amount Spiked	NS	NS	3333	NS	NA
Blank-3	NS	NS	100 U	NS	258.1%*
Blank-3 + Spike	NS	NS	2498	NS	48.2%
Amount Recovered	NS	NS	2498	NS	NA
Percent Recovery	NS	NS	75%	NS	NA

REPLICATE ANALYSES

361-5, Rep 1	S-US-RCC, Rep 1	25372	200 U	2591	100 U	60.9%	84.2%
361-5, Rep 2	S-US-RCC, Rep 2	16249	200 U	1691	100 U	56.0%	94.1%
361-5, Rep 3	S-US-RCC, Rep 3	17666	200 U	2025	100 U	59.6%	89.5%
		RSD%	25%	22%	0%	4%	6%

U = Below detection limits.

E = Values due to residuals from high Aroclors 1242 and 1248 levels.

\* = Value outside of Internal QC limits (40-120%).

NC = Not certified.

# = Mean of 3 replicates.

NS = Not spiked. NA = Not applicable.

**RE-PROCESSED RESULTS (1/92)**  
**PCBs IN TREATED SEDIMENT**

B.E.S.T  
 SAIC-GLNPO (CF #361)

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Concentrations in ug/kg dry weight

MSL Code	Sponsor ID	Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260	% Tetrachloro-m-Xylene	% Surrogate Recovery Octachloro-naphthalene
361-8	S-TS-RCC	205	100 U	30 J	50 U	35.2%*	71.2%
361-9	B-TS-RCC	100 U	100 U	50 U	50 U	34.5%*	90.0%
361-10	I-TS-RCC	100 U	440	50 U	50 U	89.7%	50.3%
Blank-4		200 U	200 U	100 U	100 U	21.0%*	71.5%

**STANDARD REFERENCE MATERIAL**

SRM-2 (HS-2)	certified value	200 U	200 U	100 U	100 U	44.8%	41.7%
		NC	NC	111	NC	NC	NC

**MATRIX SPIKE RESULTS**

Amount Spiked		NS	NS	2404	NS	NA	NA
361-8		NS	NS	30 J	NS	35.2%*	71.2%
361-8 + Spike		NS	NS	2060	NS	72.3%	89.2%
Amount Recovered		NS	NS	2030	NS	NA	NA
Percent Recovery		NS	NS	84%	NS	NA	NA
Amount Spiked		NS	NS	2273	NS	NA	NA
361-8		NS	NS	30 J	NS	35.2%*	71.2%
361-8 + Spike DUP		NS	NS	1169	NS	30.2%*	55.4%
Amount Recovered		NS	NS	1139	NS	NA	NA
Percent Recovery		NS	NS	50%	NS	NA	NA

U = Below detection limits.  
 J = Detected below detection limit.  
 \* = Value outside of Internal QC limits (40-120%).  
 \*\* = Early eluting peaks present that do not match Aroclor pattern. Quantity estimated at ~1000-2000 ppb, based on TCX response factor.  
 NC = Not certified.  
 NS = Not spiked.  
 NA = Not applicable.

RE-PROCESSED RESULTS (1/92)

PCBs IN WATER

Concentrations in ug/L

B.E.S.T  
SAIC-GLNPO (CF #361)

2/18/92

MSL Code	Sponsor ID	Aroclor				% Surrogate Recovery	
		1242	1248	1254	1260	Tetrachloro- m-Xylene	Octachloro- naphthalene
361-1	S-WR-RCC	0.2 U	0.2 U	0.1 U	0.1 U	41.2%	114.1%
361-2	B-WR-RCC	0.2 U	0.2 U	0.1 U	0.1 U	35.2%*	98.0%
361-11	I-WR-RCC	0.2 U	4.8	0.1 U	0.1 U	43.5%	86.0%
Blank-1		0.2 U	0.2 U	0.1 U	0.1 U	36.5%*	98.0%

MATRIX SPIKE RESULTS

Amount Spiked

Blank-1

Blank-1+ Spike

Amount Recovered

Percent Recovery

NS	NS	NS	NS	NS	NS	NS	NS
NS	NS	NS	NS	50	NS	NA	NA
NS	NS	NS	NS	0.1 U	NS	36.5%*	98.0%
NS	NS	NS	NS	42.6	NS	33.6%*	123.8%*
NS	NS	NS	NS	42.6	NS	NA	NA
NS	NS	NS	NS	85%	NS	NA	NA

U = Below detection limits.

\* = Value outside of internal QC limits (40-120%).

NC = Not certified.

NS = Not spiked.

NA = Not applicable.

**RE-PROCESSED RESULTS (1/92)**

**B.E.S.T**

**2/28/92**

**SAIC-GLNPO (CF #361)**

**PCBs IN OIL**

Concentrations in ug/L

MSL Code	Sponsor ID	Sample Density (g/ml)	Aroclor			% Surrogate Recovery		
			1242	1248	1254	1260	Tetrachloro-m-Xylene	Octachloro-naphthalene
361-3	S-OR-RCC	0.7525	318320	2000 U	30789	1000 U	65.9%	90.5%
361-4	B-OR-RCC	0.6903	2000 U	2702	1000 U	1000 U	71.5%	91.9%
361-12, Rep 1	I-OR-RCC, Rep 1	0.7301	2000 U	118616	1000 U	1000 U	93.7%	96.9%
361-12, Rep 2	I-OR-RCC, Rep 2	0.7301	2000 U	120743	1000 U	1000 U	85.5%	83.7%
361-12, Rep 3	I-OR-RCC, Rep 3	0.7301	2000 U	112109	1000 U	1000 U	85.2%	90.4%
Blank-1 Oil			2000 U	2000 U	1000 U	1000 U	48.6%	96.7%

**OIL CONCENTRATIONS ON % OIL BASIS**

Concentrations in ug/kg oil		% Oil (%)	Aroclor			% Surrogate Recovery	
MSL Code	Sponsor ID		1242	1248	1254	1260	Tetrachloro-m-Xylene
361-3	S-OR-RCC	4573563	28736 U	442371	14368 U	65.9%	90.5%
361-4	B-OR-RCC	46083 U	62258	23041 U	23041 U	71.5%	91.9%
361-12, Rep 1	I-OR-RCC, Rep 1	4587 U	270875	2284 U	2284 U	83.7%	96.9%
361-12, Rep 2	I-OR-RCC, Rep 2	4587 U	275732	2284 U	2284 U	85.5%	83.7%
361-12, Rep 3	I-OR-RCC, Rep 3	4587 U	256015	2284 U	2284 U	85.2%	90.4%

**MATRIX SPIKE RESULTS**

Amount Spiked	Amount Recovered	Percent Recovery
361-3	NS	NS
361-3+ Spike	NS	NS
Amount Recovered	50000	68%
Percent Recovery	30789	61.2%
	64912	NA
	34123	NA
	68%	NA

**REPLICATE ANALYSES**

Sample	Amount	% Recovery
361-12, Rep 1	118616	93.7%
361-12, Rep 2	120743	85.5%
361-12, Rep 3	112109	85.2%
	4%	5%
	NA	7%

U = Below detection limits.  
 NC = Not certified.  
 NS = Not spiked.  
 NA = Not applicable.

## APPENDIX F

### QUALITY ASSURANCE/QUALITY CONTROL

In order to obtain data of known quality to be used in evaluating the different technologies for the different sediments, a Quality Assurance Project Plan (QAPP) was prepared. The QAPP specified the guidelines to be used to ensure that each measurement system was in control. In order to show the effectiveness of the different technologies, the following measurements were identified in the QAPP as critical - PAHs, PCBs, metals, total solids, volatile solids, and oil and grease in the untreated and treated sediments. Other parameters analyzed in the sediments included pH, TOC, total cyanide, and total phosphorus. If water and oil residuals were generated by a technology, then PAHs and PCBs were determined as a check on their fate in treating the sediments. Each of these measurements and the associated quality control (QC) data will be discussed in this section.

Also included in this section are a discussion of the modifications and deviations from the QAPP and the results of a laboratory audit performed. Any possible effects of findings on data quality will be presented.

#### PROCEDURES USED FOR ASSESSING DATA QUALITY

The indicators used to assess the quality of the data generated for this project are accuracy, precision, completeness, representativeness, and comparability. All indicators will be discussed generally in this section; specific results for accuracy, precision, and completeness will be summarized in later sections.

##### Accuracy

Accuracy is the degree of agreement of a measured value with the true or expected value. Accuracy for this project will be expressed as a percent recovery (%R).

Accuracy was determined during this project using matrix spikes (MS) and/or standard reference materials (SRMs). Matrix spikes are aliquots of sample spiked with a known concentration of target analyte(s) used to document the accuracy of a method in a given sample matrix. For matrix spikes, recovery is calculated as follows:

$$\%R = \frac{C_1 - C_o}{C_t} \times 100$$

where:  $C_1$  = measured concentration in spiked sample aliquot  
 $C_o$  = measured concentration in unspiked sample aliquot  
 $C_t$  = actual concentration of spike added

An SRM is a known matrix spiked with representative target analytes used to document laboratory performance. For SRMs, recovery is calculated as follows:

$$\%R = \frac{C_m}{C_t} \times 100$$

where:  $C_m$  = measured concentration of SRM  
 $C_t$  = actual concentration of SRM

In addition, for the organic analyses, surrogates were added to all samples and blanks to monitor extraction efficiencies. Surrogates are compounds which are similar to target analytes in chemical composition and behavior. Surrogate recoveries will be calculated as shown above for SRMs.

### Precision

Precision is the agreement among a set of replicate measurements without assumption of knowledge of the true value. When the number of replicates is two, precision is determined using the relative percent difference (RPD):

$$RPD = \frac{(C_1 - C_2) \times 100}{(C_1 + C_2) / 2}$$

where:  $C_1$  = the larger of two observed values  
 $C_2$  = the smaller of two observed values

When the number of replicates is three or greater, precision is determined using the relative standard deviation (RSD):

$$RSD = \frac{S}{X} \times 100$$

where:  $S$  = standard deviation of replicates  
 $X$  = mean of replicates

Precision was determined during this project using triplicate analyses for those samples suspected to be high in target analytes (i.e., untreated sediments). Matrix spike and matrix spike duplicate (MSD) analyses were performed on those samples suspected to be low in target analytes (i.e., treated sediments). A MSD is a second spiked sample aliquot with a known concentration of target analyte used to document accuracy and precision in a given sample matrix.

### Completeness

Completeness is defined as the percentage of valid data points to the total number of data points obtained.

$$\% \text{ Completeness} = \frac{\text{VDP}}{\text{TDP}} \times 100$$

where: VDP = number of valid data points

TDP = total data points obtained

For this project, completeness was determined for each parameter for each technology evaluated.

(Add more)

### Representativeness

Representativeness refers to the degree with which analytical results accurately and precisely represent actual conditions present at locations chosen for sample collection. Sediment samples were collected prior to this demonstration and were reported to be representative of the areas to be remediated.

### Comparability

Comparability expresses the extent with which one data set can be compared to another. As will be discussed in more detail in the section MODIFICATIONS AND DEVIATIONS FROM THE QAPP, the data generated are comparable within this project and within other projects conducted for the ARCS Program.

## **ANALYTICAL QUALITY CONTROL**

The following sections summarize and discuss analytical procedures and the results of the QC indicators of accuracy and precision for each measurement parameter.

### PAH Procedures

Sediments and waters were extracted and analyzed using modified SW-846 procedures as described in the section MODIFICATIONS AND DEVIATIONS FROM THE QAPP. Oils were diluted 1:10 in hexane. Three radiolabelled PAH surrogates were added to all samples and blanks prior to extraction. Daily mass tuning was performed using decafluorotriphenylphosphine (DFTPP) to meet the criteria specified in Method 8270. The instrument was calibrated at five levels for the sixteen polynuclear aromatic hydrocarbons (PAHs). The RSD of the response factors for each PAH was required to be <25 percent. Calibrations were verified every 12 hours for each PAH; criteria for % difference from the initial calibration was <25 percent. An internal standard, hexamethyl benzene, was added prior to cleanup and was used to correct PAH

concentrations for loss during cleanup and extract matrix effects. Quantification was performed using Selective Ion Monitoring (SIM).

#### PAH QC Results and Discussion

Surrogate recoveries for all PAH samples for the B.E.S.T. demonstration are summarized in Table QA-1. If more than one of the three surrogates fell outside the control limits used, corrective action (reanalysis) was necessary. (Insufficient sample remained for reanalysis of water residuals). Surrogate recoveries were generally low for samples and method blanks, indicating a possible analytical problem rather than matrix effects. An investigation indicated possible problems with the evaporator used to concentrate the extracts. This concentration step was not performed for the oil residuals and, as can be seen in Table QA-1, acceptable surrogate recoveries for the method blank were obtained. In summary, low surrogate recoveries indicate that PAH target concentrations may be biased somewhat low. Since both the untreated and treated sediments were affected similarly, relative removal percentages should be valid.

As required by the QAPP, triplicate analyses of the Saginaw River untreated sediment (S-US-RCC) were performed to assess precision. These results are summarized in Table QA-2. A matrix spike was performed on this same sample to assess accuracy. These results are included in Table QA-2. All RSD and spike recoveries fell within the control limits specified. It should be noted that these QC analyses were reanalyses; the initial analysis yielded unacceptable surrogates and poor precision. The effect of missed holding times on data quality will be discussed in the section HOLDING TIMES.

As required by the QAPP, a matrix spike and a matrix spike duplicate (MS/MD) analysis was performed for the treated Saginaw River sediment (S-TS-RCC). These results are presented in Table QA-3. While recoveries were generally acceptable, RPDs were consistently outside the control limits specified in the QAPP. These RPDs, however, are generally within acceptance criteria specified in Method 8270; data should be of sufficient quality to support project results. It should be noted that spike levels averaged up to one hundred times the target concentrations and that accuracy and precision data for this MS/MSD may not be indicative of the accuracy and precision obtainable at the target concentrations.

Due to the minimal amount of water generated by the B.E.S.T. process, no QC analyses were performed on this matrix.

The QAPP specified that triplicate analyses and a matrix spike be performed on the Saginaw River oil residual (S-OR-RCC). This sample was spiked but triplicate analyses were performed on the Indiana Harbor oil residual. These results are summarized in Tables QA-4 and QA-5, respectively.

One certified NIST standard reference material (SRM) was extracted and analyzed with the sediment samples. The recoveries for this standard are summarized in Table QA-6.

Method blanks were extracted and analyzed with each set of samples extracted. Insignificant

TABLE QA-1. PAH SURROGATE RECOVERIES, PERCENT

Sample	d8-Napthalene	d10-Acenapthalene	d12-Perylene	Control Limits
S-US-RCC, Rep. 1	37 *	53	79	40 - 120
S-US-RCC, Rep. 2	34 *	47	83	
S-US-RCC, Rep. 3	37 *	50	76	
B-US-RCC	25 *	45	64	
I-US-RCC	27 *	48	60	
Method Blank	26 *	26 *	90	
Method Blank	25 *	24 *	90	
S-TS-RCC	41	46	67	40 - 120
B-TS-RCC	31 *	43	73	
I-TS-RCC	25 *	54	85	
Method Blank	21 *	19 *	50	
S-WR-RCC	39 *	44	130 *	40 - 120
B-WR-RCC	35 *	37 *	111	
I-WR-RCC	20 *	28 *	90	
Method Blank	37 *	38 *	83	
S-OR-RCC	8 *	46	161 *	40 - 120
B-OR-RCC	22 *	39 *	95	
I-OR-RCC, Rep. 1	23 *	61	123 *	
I-OR-RCC, Rep. 2	30 *	63	106	
I-OR-RCC, Rep. 3	19 *	59	108	
Method Blank	60	118	73	

\* Outside Control Limits

quantities of some PAHs were found in a few blanks; total concentrations are unaffected. No corrections for method blanks were performed.

TABLE QA-2. PAH REPLICATE AND SPIKE RESULTS FOR S-US-RCC

Compound	Replicate 1 dry ppb	Replicate 2 dry ppb	Replicate 3 dry ppb	Mean	RSD	Precision Control Limits	% Recovery	Accuracy Control Limits
Napthalene	26	24	27	26	6.0	20	41	40 - 120 %
Acenaphthylene	15	20U	18	NC	NC		62	
Acenaphthene	19	20U	30U	NC	NC		57	
Fluorene	33	32	33	33	1.8		64	
Phenanthrene	249	302	249	267	11		64	
Anthracene	64	70	63	66	5.7		63	
Fluoranthene	376	464	351	397	14		73	
Pyrene	425	491	402	439	11		68	
Benzo(a)anthracene	184	210	163	186	13		79	
Chrysene	265	299	242	269	11		74	
Benzo(b)fluoranthene	238	270	217	242	11		86	
Benzo(k)fluoranthene	180	200	158	179	12		72	
Benzo(a)pyrene	224	250	200	225	11		82	
Indeno(1,2,3,c,d)pyrene	201	231	188	207	11		95	
Dibenzo(a,h)anthracene	44	47	39	43	9.4		93	
Benzo(g,h,i)perylene	113	129	109	117	9.1		58	

NC = Not Calculated

U = Undetected

TABLE QA-3. PAH MS/MSD RESULTS FOR S-TS-RCC

Compound	MS Recovery	MSD Recovery	RPD	Accuracy Control Limits	Precision Control Limits
Naphthalene	31 *	54	32 *	40 - 120%	20
Acenaphthylene	45	70	43 *		
Acenaphthene	42	64	42 *		
Fluorene	53	71	29 *		
Phenanthrene	88	76	15		
Anthracene	74	75	1.3		
Fluoranthene	122 *	87	33 *		
Pyrene	110	82	29 *		
Benzo(a)anthracene	117	91	25 *		
Chrysene	113	85	28 *		
Benzo(b)fluoranthene	125 *	91	31 *		
Benzo(k)fluoranthene	104	80	26 *		
Benzo(a)pyrene	113	79	35 *		
Indeno(1,2,3,c,d)pyrene	128 *	90	35 *		
Dibenzo(a,h)anthracene	117	90	26 *		
Benzo(g,h,i)perylene	78	56	33 *		

\* Outside Control Limits

## PCBs

### PCB Procedures

Sediments and waters were extracted and analyzed using modified SW-846 procedures as described in the section MODIFICATIONS AND DEVIATIONS FROM THE QAPP. Oil were diluted 1:10 in hexane. Two surrogates, tetrachloro-m-xylene and octachloronaphthalene, were added to all samples and blanks prior to extraction. The gas chromatograph (GC) employed electron capture detection (ECD) and was calibrated at three levels for each of four Aroclors (1242, 1248, 1254, 1260). The RSD of the response factors for each Aroclor was required to be <25 percent. Calibrations were verified after every ten samples; criteria for % difference from the initial calibration was <25 percent. An internal standard, dibromooctafluorobiphenyl, was added prior to cleanup and was used to correct PCB concentrations for loss during cleanup and extract matrix effects. Quantification of Aroclors was performed on two columns (DB-5, primary and 608, confirmation) as a confirmation of their presence.

**TABLE QA-4. PAH MS RESULTS FOR S-OR-RCC**

Compound	MS Recovery %	Control Limits
Naphthalene	11	Not Specified
Acenaphthylene	34	
Acenaphthene	39	
Fluorene	62	
Phenanthrene	90	
Anthracene	109	
Fluoranthene	128	
Pyrene	119	
Benzo(a)anthracene	141	
Chrysene	112	
Benzo(b)fluoranthene	134	
Benzo(k)fluoranthene	116	
Benzo(a)pyrene	130	
Indeno(1,2,3,c,d)pyrene	142	
Dibenzo(a,h)anthracene	158	
Benzo(g,h,i)perylene	120	

**PCB QC Results and Discussion**

Surrogate recoveries for all PCB samples for the B.E.S.T. demonstration are summarized in Table QA-7. If both recoveries fell outside the control limits used, correction action (reanalysis) was necessary. All samples were acceptable with respect to the surrogate criteria used.

As required by the QAPP, triplicate analyses of the Saginaw River untreated sediment (S-US-RCC) were performed to assess precision. These results are summarized in Table QA-8. A matrix spike using Aroclor 1254 was performed on the same sample to assess accuracy. These results are included in Table QA-8. RSDs were outside specified control limits but within precision specified in Method 8080; data should be of sufficient quality to support project results.

As required by the QAPP, a matrix spike and a matrix spike duplicate (MS/MSD) analysis was performed for the treated Saginaw Rive sediment (S-TS-RCC). These results are presented in Table QA-9. The RPD was outside control limits; however, no Aroclor 1254 was found in the sample and the data is not impacted. Matrix spike recoveries were acceptable. Due to the minimal amount of water generated by the B.E.S.T. process, no QC analyses were performed on this matrix.

The QAPP specified that triplicate analyses and a matrix spike be performed on the Saginaw River

oil residual (S-OR-RCC). This sample was spiked but triplicate analyses were performed on the Indiana Harbor oil residual. These results are summarized in Tables QA-10 and QA-11, respectively.

One standard reference material (SRM) certified by the National Research Council of Canada (NRCC) for Aroclor 1254 was extracted and analyzed twice with the sediment samples. Recoveries of 82.8% and 78.6% were obtained. The average of 80.7% fell within the 80-120% criteria specified in the QAPP.

Method blanks were extracted and analyzed with each set of samples extracted. No PCBs were found in any method blanks.

## **METALS**

### Metals Procedure

Sediments were prepared for metals analysis by freeze-drying, blending, and grinding. Sediments for As, Cl, Hg, and Se were digested using nitric and hydrofluoric acids. The digestates were analyzed for As, Cd, and Se by graphite furnace atomic absorption (GFAA) by SW-846 Method 7000 series using Zeeman Background correction. The digestates were analyzed for mercury by cold vapor AA (CVAA) using SW-846 Method 7470.

Sediments for As, Ba, Cr, Cu, Fe, Mn, Ni, Pb, and Zn were analyzed by energy-dispersive X-Ray fluorescence (XRF) following the method of Sanders (1987). The XRF analysis was performed on a 0.5g aliquot of dried, ground sediment pressed into a pellet with a diameter of 2 cm.

### Metals QC Results and Discussion

Triplicate analyses of the Saginaw River untreated sediment (S-US-RCC) and treated sediment (S-TS-RCC) were performed to assess precision. Matrix spikes were analyzed for the same samples to assess accuracy. Results are summarized in Tables QA-12 and QA-13. It should be noted that the sediments were not spiked for XRF analysis.

Accuracy and precision results for metals were acceptable with only a few minor exceptions, as shown in Tables QA-12 and QA-13. These exceptions have little, if any, impact on data quality and project results.

One NIST certified standard reference material (SRM) was digested and/or analyzed twice with the sediment samples for XRF, GFAA, and CVAA analyses. These results are presented in Table QA-14.

Method blanks were digested and analyzed for the metals analyzed by GFAA and CVAA. (Method blanks are not applicable to XRF analysis). If analyte was detected in the method blank, blank correction was performed. Minimal amounts of some metals were detected; data quality is not affected.

TABLE QA-5. PAH REPLICATE<sup>(a)</sup> RESULTS FOR I-OR-RCC ng/ml

Compound	Replicate 1 ppb	Replicate 2 ppb	Replicate 3 ppb	RSD	Control Limits
Napthalene	30000 U	30000 U	30000 U	NC	Not Specified
Acenaphthylene	46600	52000	45400	7.4	
Acenaphthene	50000 U	50100	42400	NC	
Fluorene	48300	57000	50200	8.9	
Phenanthrene	212000	222000	206000	3.8	
Anthracene	108000	117000	104000	6.1	
Fluoranthene	634000	657000	618000	3.1	
Pyrene	608000	627000	590000	3.0	
Benzo(a)anthracene	346000	366000	336000	4.4	
Chrysene	481000	504000	467000	3.8	
Benzo(b)fluoranthene	433000	456000	406000	5.8	
Benzo(k)fluoranthene	290000	297000	279000	3.1	
Benzo(a)pyrene	434000	453000	413000	4.6	
Indeno(1,2,3,c,d)pyrene	363000	378000	344000	4.7	
Dibenzo(a,h)anthracene	75100	80600	70600	6.7	
Benzo(g,h,i)perylene	220000	224000	207000	4.1	

(a) Replicate results represent values after correction for percent oil concentration.

NC = Not Calculated

U = Undetected

TABLE QA-6. PAH SRM RESULTS

Compound	Recovery, %	Control Limits
Naphthalene	NC	80 - 120%
Acenaphthylene	NC	
Acenaphthene	NC	
Fluorene	NC	
Phenanthrene	95	
Anthracene	81	
Fluoranthene	91	
Pyrene	96	
Benzo(a)anthracene	87	
Chrysene	NC	
Benzo(b)fluoranthene	98	
Benzo(k)fluoranthene	136 *	
Benzo(a)pyrene	75 *	
Indeno(1,2,3,c,d)pyrene	88	
Dibenzo(a,h)anthracene	NC	
Benzo(g,h,i)perylene	82	

NC = Not Certified

\* Outside Control Limits

TABLE QA-7. PCB SURROGATE RECOVERIES, PERCENT

Sample	Tetrachloro-m-xylene	Octachloronaphthalene	Control Limits
S-US-RCC, Rep. 1	53	85	40 - 120
S-US-RCC, Rep. 2	56	94	
S-US-RCC, Rep. 3	51	92	
B-US-RCC	63	70	
I-US-RCC	62	61	
Method Blank	25 *	130 *	
S-TS-RCC	35 *	76	40 - 120
B-TS-RCC	34 *	96	
I-TS-RCC	89	54	
Method Blank	21 *	76	
S-WR-RCC	41	124 *	40 - 120
B-WR-RCC	35 *	109	
I-WR-RCC	44	98	
Method Blank	36 *	107	
S-OR-RCC	65	100	40 - 120
B-OR-RCC	71	103	
I-OR-RCC, Rep. 1	92	107	
I-OR-RCC, Rep. 2	86	97	
I-OR-RCC, Rep. 3	84	102	
Method Blank	48	105	

\* Outside Control Limits

TABLE QA-8. PCB REPLICATE AND SPIKE RESULTS FOR S-US-RCC

Aroclor	Replicate 1 ppb dry	Replicate 2 ppb dry	Replicate 3 ppb dry	Mean	RSD	Precision Control Limits	% Recovery	Accuracy Control Limits
1242/1248						20		--
1254						20		40 - 120
1260						20		--

TABLE QA-9. PCB MS/MSD RESULTS FOR S-TS-RCC

PCB	MS Recovery	MSD Recovery	PRD	Accuracy Control Limits	Precision Control Limits
Aroclor 1254				40 - 120%	20

**TABLE QA-10. PCB MS RESULT FOR S-OR-RCC**

PCB	MS Recovery	Control Limits
Aroclor 1254		Not Specified

**OIL AND GREASE**

Oil and Grease Procedures

Sediment samples were extracted with freon using Soxhlet extraction according to SW-846 Method 9071. The extract was analyzed for oil and grease by infra-red (IR) as outlined in Method 418.1 (Methods for Chemical Analysis of Water and Wastes, 1983).

Oil and Grease QC Results and Discussion

Both the untreated and treated Saginaw River sediments (S-UC-RCC and S-TS-RCC) were analyzed for oil and grease in triplicate. In addition, matrix spike was performed for S-TS-RCC. These results are summarized in Table QA-15. All QC results fell within specified control limits.

**TOTAL VOLATILE SOLIDS**

Total Volatile Solid Procedures

Sediments were analyzed for total volatile solids (TVS) following the procedures in Method 160.4. (Methods for Chemical Analysis of Water and Waste, 1983) modified for sediments. An aliquot of sediment was dried and then ignited at 550°C. The loss of weight on ignition was then determined.

Total Volatile Solid QC Results and Discussion

Both the untreated and treated Saginaw River sediments (S-US-RCC and S-TS-RCC) were analyzed for TVS in triplicate. Results are summarized in Table QA-16. Both RSDs fell within specified control limits.

**AUDIT FINDINGS**

An audit of the Battelle-Marine Sciences Laboratory was conducted on September 25 and 26, 1991. Participants included EPA, GLNPO, and SAIC personnel. The path of a sample from receipt to reporting was observed specifically for samples from these bench-scale treatability tests. Two concerns were identified in the organic laboratory: 1) the preparation, storage, record-keeping, and replacement of

TABLE QA-11. PCB REPLICATE RESULTS FOR I-OR-RCC

Aroclor	Replicate 1, ppb	Replicate 2, ppb	Replicate 3, ppb	Mean	RSD	Control Limits
1242/1248						20
1254						20
1260						20

TABLE QA-12. METALS REPLICATE AND SPIKE RESULTS FOR S-US-RCC

Metal	Method	Replicate 1, ppm dry	Replicate 2, ppm dry	Replicate 3, ppm dry	Mean	RSD	Precision Control Limits	% Recovery	Accuracy Control Limits
Ag	GFAA	0.87	0.85	0.80					
As	XRF	1.86	2.65	2.12					
Ba	XRF	322	322	321					
Cd	GFAA	4.39	4.08	3.96					
Cr	XRF	93	112	117					
Cu	XRF	55.8	57.3	63.4					
Fe(I)	XRF	0.780	0.765	0.816					
Hg	CVAA	0.162	0.178	0.160					
Mn	XRF	162	159	173					
Ni	XRF	58.1	55.2	61.5					
Pb	XRF	43.0	42.5	50.9					
Se	GFAA	0.3 U	0.3 U	0.3 U					
Zn	XRF	126	133	162					

\* Outside Control Limits NS = Not Spiked NC = Not Calculated

(1) Results in Percent for Fe U = Undetected

TABLE QA-13. METALS REPLICATE AND SPIKE RESULTS FOR S-TS-RCC

Metal	Method	Replicate 1, ppm dry	Replicate 2, ppm dry	Replicate 3, ppm dry	Mean	RSD	Precision Control Limits	% Recovery	Accuracy Control Limits
Ag	GFAA	1.03	0.76	0.67	0.82	23	20	120*	85 - 115
As	XRF	2.77	2.87	2.92	2.85	2.7		NS	--
Ba	XRF	325	321	310	319	2.4		NS	--
Cd	GFAA	4.38	4.17	4.24	4.26	2.5		100	85 - 115
Cr	XRF	130	113	110	118	9.2		NS	--
Cu	XRF	68.6	66.3	57.4	64.1	9.2		NS	--
Fe(1)	XRF	0.855	0.827	0.795	0.826	3.6		NS	--
Hg	CVAA	0.505	0.290	0.209	0.335	46*		98	85 - 115
Mn	XRF	181	177	172	177	2.6		NS	--
Ni	XRF	71.5	64.1	57.2	64.3	11		NS	--
Pb	XRF	53.3	45.0	41.4	46.6	13		NS	--
Se	GFAA	0.3 U	0.3 U	0.3 U	0.3 U	NC		101	85 - 115
Zn	XRF	194	165	147	169	14		NS	--

\* Outside Control Limits

(1) Results in Percent for Fe

NS = Not Spiked

U = Undetected

NC = Not Calculated

**TABLE QA-14. METALS SRM RESULTS, % RECOVERY**

Metal	SRM-1	SRM-2	Control Limits
Ag	NC	NC	80 - 120%
As	113	97.4	
Ba	NC	NC	
Cd	117	117	
Cr	85.5	103	
Cu	124*	110	
Fe	104	100	
Hg	105	105	
Mn	93.3	90.4	
Ni	113	97.2	
Pb	101	99.6	
Se	NC	NC	
Zn	96.7	93.0	

\* Outside control limits.

NC = not certified.

standards is not well-documented; and 2) the nonstandard procedures used to extract, clean up and analyze samples needs to be documented with reported data.

During the audit, the use of nonstandard procedures was discussed. It was concluded that data comparability within this project and within the ARCS program should not be an issue, as the Battelle laboratory has performed all analyses to date. However, comparability to data generated outside the ARCS program is not possible.

**MODIFICATIONS AND DEVIATIONS FROM THE QAPP**

Laboratory activities significantly deviated from the approved QAPP in two areas--analytical procedures and quality assurance (QA) objectives. Specific deviations and their effect on data quality are discussed in this section.

TABLE QA-15. OIL AND GREASE REPLICATES AND SPIKE RESULTS FOR S-US-RCC AND S-TS-RCC

Sample	Replicate 1, ppm dry	Replicate 2, ppm dry	Replicate 3, ppm dry	Mean	RSD	Precision Control Limits	% Recovery	Accuracy Control Limits
S-US-RCC	1480	1320	1260	1350	8.4	20	114	80 - 120%
S-TS-RCC	297	293	206	265	19	20	NS	---

NS = Not Spiked

TABLE QA-16. TVS REPLICATES FOR S-US-RCC AND S-TS-RCC

Sample	Replicate 1, % dry	Replicate 2, % dry	Replicate 3, % dry	Mean	RSD	Control Limits
S-US-RCC	2.24	2.03	2.01	2.09	6.1	20
S-TS-RCC	1.76	1.70	1.74	1.73	1.8	20

## ANALYTICAL PROCEDURES

The Assessment and Remediation of Contaminated Sediments (ARCS) Program was initiated by the Great Lakes National Program Office (GLNPO) to conduct bench-scale and pilot-scale demonstrations for contaminated sediments. To date, all laboratory analyses performed in support of the ARCS Program have been done at the Battelle-Marine Sciences Laboratory (MSL) in Sequim, Washington. Standard procedures used by Battelle-MSL often do not follow those procedures identified in SW-846 and the QAPP. While these nonstandard procedures yield results of acceptable quality, comparability with analyses performed outside the ARCS Program is not possible.

### PAH Analysis

- Samples were co-extracted with PCB samples using a modified SW-846 extraction procedure which entailed rolling of the sample in methylene chloride and an additional clean-up step using high pressure liquid chromatography (HPLC). An internal standard, hexamethyl benzene, was added prior to this clean-up step to monitor losses through the HPLC. Final results were corrected for the recovery of this internal standard. A second internal standard, d12-phenanthrene, was added prior to analysis; however, no corrections were made based on its recovery. Neither of these internal standards are specified in Method 8270.
- SW-846 Method 8270 was modified to quantify the samples using Selective Ion Monitoring (SIM) Gas Chromatography/Mass Spectrometry (GC/MS). This modification results in improved detection limits.
- Three radiolabelled PAH compounds were used as surrogates rather than those recommended in Method 8270. Recoveries of these compounds should better represent the recoveries of target PAHs.

### PCB Analysis

- Samples were extracted using the modified extraction procedures as described for the PAH analysis. An internal standard, dibromooctafluorobiphenyl, was added prior to the HPLC clean-up to monitor losses. Final results were corrected for the recovery of this standard. A second internal standard, 1,2,3-trichlorobenzene (required by QAPP) was added prior to analysis; however, no corrections were made based on its recovery.
- Quantification of PCBs was not done on a total basis as required by SW-846 Method 8080 but by quantifying four peaks for each Aroclor and averaging these results. (Add max)
- A three-point calibration for each peak was used instead of the five-point calibration required by Method 8080. This modification should have minimal effect on data quality.

- The surrogate required by the QAPP, tetrachloro-m-xylene, was used. A second surrogate, octochloronaphthalene, was also added to monitor extraction efficiency.

#### Metals Analysis

- Nine of the 13 metals analyzed for sediment samples were measured by energy-dispersive X-Ray fluorescence (XRF) - As, Ba, Cr, Cu, Fe, Mn, Ni, Pb, and Zn. This procedure yields a total metals concentration instead of the recoverable metals determined by SW-846 methods.
- Sediments for Ag, Cd, Hg, and Se were subjected to an acid digestion using nitric and hydrofluoric acids. This digestion again yields total rather than recoverable metals.

#### Oil and Grease

- Oil and grease extracts for sediments were analyzed using infrared (IR) detection rather than the gravimetric procedures specified in the QAPP. This should have no effect on data quality.

#### QUALITY ASSURANCE OBJECTIVES

Many of the QA objectives and internal QC checks criteria specified in the QAPP (particularly for organic analyses) are not routinely achievable by standard or nonstandard methods. To avoid excessive reanalyses (both costly and time-consuming), acceptance criteria established internally by Battelle were used for this project. These internal limits are adequate for use in determining whether or not project results are valid.

#### PAH Analysis

- Both surrogate and matrix spike objectives for PAHs were specified in the QAPP to be 70-130%. For surrogates, Battelle actually used internal limits of 40-120%, with one of the three surrogates out of limits being acceptable. If more than one surrogate did not fall within 40-120%, reanalysis was required. For matrix spikes, internal limits of 40-120% were also used; no reanalyses however, were performed based on exceedences of these limits.
- Limits for continuing calibration checks were specified as  $\pm 10\%$  in the QAPP; limits of  $\pm 25\%$  were used.

#### PCB Analysis

- Both surrogate and matrix spike objectives for PCBs were specified in the QAPP to be 70-130%. For surrogates, Battelle actually used internal limits of 40-120%. If both surrogates exceeded these limits, re-extraction was performed. For matrix spikes, internal limits of 40-120% were also used; no reanalyses, however, were performed based on exceedences of these limits.

- Limits for continuing calibration checks were specified as  $\pm 10\%$  in the QAPP; limits of  $\pm 25\%$  were used.

#### Metals Analysis

- Samples analyzed by XRF cannot be spiked. Therefore, no measure of sample accuracy was obtained for those metals previously identified as being analyzed by XRF. An SRM was analyzed, providing a means to measure method accuracy for eight of the nine metals determined by XRF (all but Ba).

# **Data Verification Report For Assessment and Remediation of Contaminated Sediment Program**

**Report Number 8  
(SAIC, Bench-Scale Tests)**

**By**

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## ABSTRACT

Data submitted by the Science Applications International Corporation (SAIC) of Cincinnati, Ohio, have been verified for compliance of the QA/QC requirements of the Assessment and Remediation of Contaminated Sediment (ARCS) program. This data set includes results from bench-scale technology demonstration tests on wet contaminated sediments using four treatment technologies, namely, B.E.S.T. (extraction process), RETEC (low temperature stripping), ZIMPRO (wet air oxidation), and Soil Tech (low temperature stripping). The primary contaminants in these sediments were polychlorinated biphenyls (PCBs) and polynuclear aromatic hydrocarbons (PAHs). In addition, metal contents and conventionals (% moisture, pH, % total volatile solids, oil and grease, total organic carbon (TOC), total cyanide, and total phosphorus) in these sediments were also considered for this project. The objective of the bench-scale technology demonstration study was to evaluate four different treatment techniques for removing different organic contaminants from sediments. Both treated and untreated sediment samples were analyzed to determine treatment efficiencies.

A total of seven sediment samples from four different areas of concerns (Buffalo River, Ashtabula River, Indiana Harbor, and Saginaw River) were analyzed under the bench-scale technology demonstration project. The samples from these areas of concern (AOCs) were collected by the Great Lakes National Program Office (GLNPO) in Chicago, IL, and sample homogenization was performed by the U. S. EPA in Duluth, MN. SAIC was primarily responsible for the characterization of the sediment samples prior to testing and for the residues created during the test. The solid fraction analyses were performed by SAIC's analytical subcontractor Battelle-Marine Sciences Laboratory of Sequim, Washington, and Analytical Resources Incorporated of Seattle, Washington.

The submitted data sets represent analyses of untreated sediments, as well as solid, water, and oil residues obtained by using different treatments. The verified data set is divided into several parameter groups by sampled media. The data verifications are presented in parameter groups that include: metals, PCBs, conventionals, and PAHs.

The results of the verified data are presented as a combination of an evaluation (or rating) number and any appropriate data flags that may be applicable. The templates used to assess each individual analyte are attached in case the data user needs the verified data of a single parameter instead of a parameter group.

## INTRODUCTION

The bench-scale technology demonstration project was undertaken to evaluate the efficiencies of four techniques used for the removal of specific contaminants from wet sediments collected from designated Great Lakes areas of concern. Four different sediment treatment techniques, namely, B.E.S.T (Basic Extraction Sludge Technology), RETEC, ZIMPRO, and Soil Tech were considered for evaluation. B.E.S.T. is a solvent extraction process, RETEC and Soil Tech are low temperature stripping techniques, and ZIMPRO is a wet air oxidation technique. Wet sediments were collected by the Great Lakes National Program Office (GLNPO) from four Great Lakes sites, namely, the Buffalo River in New York, the Saginaw River/Bay (referred to as Saginaw River throughout the following discussions) in Michigan, the Grand Calumet River/Indiana Harbor (referred to as Indiana Harbor throughout the following discussions) in Indiana, and the Ashtabula River in Ohio. The four techniques were used to treat the sediment samples from these four sites. The sediment samples represent the sediment that would be obtained for on-site treatment.

The B.E.S.T. process is a patented solvent extraction technology that uses the inverse miscibility of triethylamine as a solvent. At 65° F, triethylamine is completely soluble in water and above this temperature, triethylamine and water are partially miscible. This property of inverse miscibility is used since cold triethylamine can simultaneously solvate oil and water. RETEC and the Soil Tech (low temperature stripping) are techniques to separate volatile and semivolatile contaminants from soils, sediments, sludges and filter cakes. The low temperature stripping (LTS) technology heats contaminated media to temperatures between 100 -200° F, evaporating off water and volatile organic contaminants. The resultant gas may be burned in an afterburner and condensed to a reduced volume for disposal or can be captured by carbon absorption beds. For these treatability studies, only the processes that capture the driven off contaminants were considered. The ZIMPRO (wet air oxidation) process accomplishes an aqueous phase oxidation of organic and inorganic compounds at elevated temperatures and pressures. The temperature range for this process is between 350 to 600° F (175 to 320° C). System pressure of 300 psi to well over 300 psi may be required. In this process, air or pure oxygen is used as an oxidizing agent.

Samples for the technology demonstration projects were obtained by GLNPO (Chicago, Illinois) and were analyzed by Battelle-Marine Sciences Laboratory (Battelle-MSL, Sequim, WA) and by Analytical Resources Incorporated (Seattle, WA). To evaluate the bench-scale technologies, the sample analyses were divided into four parts: (1) raw untreated sediment samples, (2) treated sediments, (3) water residues, and (4) oil residues. The amount of residues available for the analyses depended upon the corresponding sediment samples and on the individual technology used to treat those sediment samples.

The analyses of sediment and residue parameters for these projects were divided into four different categories: (1) metals, including Ag, As, Ba, Cd, Cr, Cu, Fe, Hg, Mn, Ni, Pb, Se, and Zn; (2) polychlorinated biphenyls (PCBs); (3) polynuclear aromatic hydrocarbons (PAHs);

and (4) conventionals, including percent moisture, pH, percent total volatile, oil and grease, total organic carbon (TOC), total cyanide, and total phosphorus. Analyses of metals and conventionals were performed on treated and untreated sediment samples only for B.E.S.T., ZIMPRO, and Soil Tech, while for the RETEC process, analyses of metals and conventionals were performed on treated and untreated sediment samples as well as water residue samples.

No oil residues were produced by the ZIMPRO technique (wet air oxidation treatment technique), while in the other three techniques, oil residues were analyzed after appropriate sample cleanup steps for PCBs and PAHs.

## QUALITY ASSURANCE AND QUALITY CONTROL REQUIREMENTS

The objective behind all quality assurance and quality control (QA/QC) requirements is to ensure that all data satisfy predetermined data quality objectives. These requirements are dependent on the data collection process itself. Under the bench-scale technology demonstration project, QA/QC requirements were established for:

1. Detection limits,
2. Precision,
3. Accuracy,
4. Blank analyses,
5. Surrogate and matrix spike analyses, and
6. Calibration
  - a) initial
  - b) ongoing.

Four parameter groups analyzed in the sediment and water residue phases were of interest in the bench-scale technology demonstration project. These groups included: (a) metals, (b) PCBs, (c) PAHs, and (d) conventionals. The conventionals included: percent moisture, pH, percent total volatile, oil and grease, TOC, total cyanide, and total phosphorus. In addition, total solids, total suspended solids, and conductivity were included in the conventionals group for RETEC conventional analyses. The analyses for metals and conventionals were performed for solids only, except for RETEC, where metals and conventionals were analyzed in solid and water residue phases. Parameter groups analyzed in the oil residue phase are PCBs and PAHs. The objective of these analyses was to characterize samples both before and after each treatment was applied.

The detection limits for metals, PCBs, PAHs, and conventionals (where appropriate) were defined as, three times the standard deviation for 15 replicate analyses of a sample with an analyte concentration within a factor of 10 above the expected or required limit of detection. Individual parameter detection limits are presented in the approved quality assurance project plan for SAIC on file at the Great Lakes National Program Office in Chicago, IL.

Precision requirements were based on analytical triplicate analyses for all parameters of sediment samples and treated residues, at the rate of 1 per 20 samples. The results of the triplicate analyses provided the precision for the analytical laboratory. An acceptable limit was the coefficient of variation less than or equal to 20 percent. The precision requirement was established for all variable types in this project. For treated sediments, the relative percent difference (RPD) between the matrix spike and matrix spike duplicate was used as a measure of precision with an acceptance limit of less than 20% .

Accuracy was defined as the difference between the expected value of the experimental observation and its "true" value. Accuracy in this project was required to be assessed for each variable type using analysis of certified reference materials, where available, at the rate of 1 per 20 samples. Acceptable results must agree within 20 percent of the certified range. Since no PCBs and PAHs were expected to be detected in the treated sediment, matrix spikes and matrix spike duplicate analyses were required during the analyses of treated sediment for the organic parameters. Matrix spike analyses were used as a measure of accuracy for treated sediment analyses, with an acceptance limit of  $\pm 30\%$  from the known value.

Matrix spikes were required to be used at a rate of 1 per 20 samples and to be within plus or minus 15 percent of the spiking value for metals and 70 to 130 percent of the spiking value for organics (PCBs and PAHs).

Surrogate spike analyses were only required for each sample in organic analyses. The acceptable limits for the surrogate recovery was between 70 and 130 percent of the known concentration.

The observed values should have been less than the method detection limit for each parameter for method blanks (run at the beginning, middle, and end of each analytical run).

The ongoing calibration checks were required at the beginning, middle, and end of a set of sample analyses for all variable types. The maximum acceptable difference was  $\pm 10\%$  of the known concentration value in the mid-calibration range. Initial calibration acceptance limits, for metals, was the  $\geq 0.97$  coefficient of determination for the calibration curve, while a %RSD of the response factors of less than or equal to 25% was required for organics.

## RESULTS AND DISCUSSION

The ARCS QA program was formally adopted for use when SAIC received final approval from the GLNPO on May 31, 1991. An evaluation scale, based upon the QA program developed for the ARCS program, was developed to evaluate the success of the data collection process in meeting the QA/QC requirements of the ARCS program. The following section discusses how to interpret the data verification results.

## The Verification Process and Evaluation Scale

For verification purposes, the data set from each technology was divided into 4 different sample media as follows:

1. Untreated sediment,
2. Treated sediment,
3. Water residue, and
4. Oil residue.

The verification process included QA/QC compliance checking for accuracy, precision, matrix spike analysis, surrogate spike analysis, blank analysis, detection limits, initial and ongoing calibration checks, and holding times as well as checks on calculational correctness and validity on a per parameter/analyte basis. Compliance checks were performed to ensure that the QA/QC measurements and samples: (a) met their specified acceptance limits; (b) had reported results that were supported by the raw data; and (c) were analyzed following good laboratory practices, where checking was possible. Upon completion of the verification process, a final rating was assigned for each of the individual categories. The final ratings are presented as a combination of a number value and a flag list.

The numerical value for the rating of a given parameter was assigned based upon the successful completion of each required QA/QC sample or measurement. The QA/QC samples were broken down into four different sample groups, namely, accuracy, precision, blanks, and spike recoveries. A fifth category was included for QA/QC measurements to address the successful completion of instrument calibrations (both initial and ongoing) and the determination of method detection limits. If the laboratory successfully met the acceptance criteria of 50 percent or more of the parameters in a given QA/QC sample group, then the laboratory received the full value for that category. For example, if 50 percent or more of the reagent blanks for the metals in sediment analyses had measured values below the method detection limit, then three points were awarded for that category, assuming reagent blanks were the only blank samples analyzed by the laboratory. The individual point values for each QA/QC sample type or measurement and the minimum acceptance levels for each category are presented in Appendix B. The final numerical rating presented for each parameter category is the summation of the point values from each of the five categories.

Along with each numerical rating, a list of appropriate flags has been attached to the final rating value (Appendix C). The flag indicates where discrepancies exist between the laboratory data and the acceptance limits of the required QA program. Different flags are presented for each category of QA sample (accuracy, precision, blanks, and spike recoveries) and for the QA/QC measurements (instrument calibration and detection limit determination). The flags have a letter and subscript configuration, such as A<sub>1</sub>. The letter of the flag represents the category of the discrepancy while the subscript designates the form of the discrepancy. For example, the A flags indicate discrepancies in the use of accuracy checking samples, such as reference materials or standards. A flag with a subscript of 1 indicates that the laboratory failed to meet

the acceptance criteria. Using the example of the A<sub>1</sub> flag, this flag would then indicate a failure of the laboratory to meet the QA/QC requirements for the use of reference materials in their appraisal of accuracy. A flag with the subscript 0 indicates that no information was received (or no standards were available in the case of accuracy) from the analytical laboratory, and therefore, no points could be allotted towards the final calculated rating value for that particular category. It should be noted that the 0 flag does not necessarily indicate that the analytical laboratory did not perform the QA/QC analyses, only that no information was received from the laboratory.

The subscript 9 flag indicates that the sample category or QA/QC measurement is not applicable to that particular parameter or parameter group (Appendix C). For example, an S<sub>9</sub> flag indicates that a matrix spike for that given parameter or analyte is not applicable, such as was the case for percent moisture. Where subscript 9 flags occur, an adjustment to the passing and maximum scores (to be discussed) for a parameter group was made and will be reported in the appropriate tables.

A complete presentation of the QA/QC rating factors (point values by sample type) and the various data flags and their subscripts are presented in Appendices B and C, respectively. A more complete discussion of the rating scale can be found in the report submitted to the RA/M workgroup by Schumacher and Conkling entitled, "User's Guide to the Quality Assurance/Quality Control Evaluation Scale of Historical Data Sets."

Individual parameter flags are presented in the templates found in Appendix D. The objective of the presentation of the individual flag templates is to help the data user make a determination regarding the useability of the data set for any given purpose and to provide the data user with a means to assess any individual parameter that may be of specific interest.

### The Interpretation and Use of the Final Verified Data Rating Values

The data verification scale was developed to allow for the proper rating of the verified data and the subsequent interpretation and evaluation of the ratings. Two different interpretations can be made using the ratings provided in this report, namely, the actual or "true" rating and the potential rating. The first interpretation is based upon the formal ARCS QA program, while the second interpretation scale is based upon the "full potential" value of the submitted data set. In the following sections, each interpretation of the results will be discussed.

#### Data Interpretation Based upon the Formal ARCS QA Program

For each of the four parameter categories, the data were initially verified for QA/QC compliance following the requirements specified in the signed QAPP submitted by SAIC and the ARCS QAMP on file at the GLNPO in Chicago, Illinois.

Table 1 provides the verified data ratings for each variable class for the four different technologies studied based on the current ARCS QA program. The ratings of these variable

classes are presented to provide the data user with a means for comparing the ARCS QA program-based verified results with other data sets, using the same or similar parameters, that were generated prior to and after the initiation of the formal ARCS QA program.

Table 2 provides the data user with the full compliance and acceptable scores presented for each parameter group based upon the current ARCS QA program. The full compliance score represents the numerical rating value if all required QA/QC samples and measurements were performed by the analytical laboratory and successfully met all the QA/QC requirements of the ARCS QA program. An acceptable score is lower than the full compliance score and accounts for laboratory error that can be reasonably expected during an analysis of multiple samples. Any final rating value less than the acceptable score indicates that problems were identified in the data that could adversely effect the quality of the data. The acceptable score was set at 60 percent of the full compliance score. To determine the percentage of QA/QC samples and measurements successfully analyzed for a given parameter versus the number analyzed following the complete ARCS QA protocols, divide the numerical rating received by the full compliance score. An acceptable data set, in this case, has a rating of 60 percent or greater.

In some cases, all the QA/QC requirements may not be applicable (e.g., matrix spikes for percent solids are not applicable). If this is the case, a flag with the subscript 9 was used, and the full compliance and acceptable scores were adjusted by lowering the score on appropriate number of points for nonrequired sample type, as identified in Appendix B. An example of this situation is % moisture, as indicated in Table 1, the subscript 9 flag has been applied to accuracy, blank, detection limit, and spike samples. Therefore, the full compliance and acceptable scores (Table 2) are only based upon the possible points for the successful completion of the remaining QA/QC samples that have cumulative points value of 8 (Appendix B).

#### Data Interpretation Based upon the "Potential" Value of the Data Set

A second interpretation scale has been presented to allow the data user to establish the "full potential" value of the submitted data set. The numerical value and associated flags presented in the first interpretation can be considered as an absolute rating for that data set or parameter. These ratings were based upon all the data submitted to Environmental Monitoring Systems Laboratory - Las Vegas (EMSL-LV) and to Lockheed for review by the analytical laboratory. If one or more parameter or parameter groups qualifying flags had the subscript of 5, 6, 9, or 0 (Appendix C), the required information was not available or not applicable at the time of sample analysis, and consequently was not included during the data verification and review process. The equivalent point value(s) for each individual sample type may be added to the reported point sum to give the data user the full potential value of the data set. This process assumes that if the "missing" QA/QC samples or measurements were performed, the results would fall within the ARCS QA program specified acceptance limits. For example, if the point value (including qualifying flags) for the metals was 6-B<sub>0</sub> C<sub>0</sub> D<sub>0</sub> S<sub>0</sub>, then the data user could potentially add 14 points to the score since the blank analyses, spike information, detection limit,

and calibration (initial and ongoing) information was not available for verification. The resulting data would then have a rating of 20.

**TABLE 1. Verified Data Ratings Based on the Current ARCS QA Program**

Untreated Sediments	B.E.S.T.	ZIMPRO	Soil Tech	RETEC
Metals	12-C <sub>0</sub> D <sub>0</sub>	12-C <sub>0</sub> D <sub>0</sub>	12-C <sub>0</sub> D <sub>0</sub>	12-C <sub>0</sub> D <sub>0</sub>
%Moisture	0-A, B, C <sub>0</sub> D, P <sub>0</sub> S <sub>0</sub>	3-A, B, C <sub>0</sub> D, S <sub>0</sub>	0-A, B, C <sub>0</sub> D, P <sub>0</sub> S <sub>0</sub>	3-A, B, C <sub>0</sub> D, S <sub>0</sub>
pH	0-A, B, C <sub>0</sub> D, P <sub>0</sub> S <sub>0</sub>	0-A, B, C <sub>0</sub> D, P <sub>0</sub> S <sub>0</sub>	0-A, B, C <sub>0</sub> D, P <sub>0</sub> S <sub>0</sub>	3-A, B, C <sub>0</sub> D, S <sub>0</sub>
%TVS	6-A, C <sub>0</sub> D, S <sub>0</sub>	3-A, B, C <sub>0</sub> D, S <sub>0</sub>	6-A, C <sub>0</sub> D, S <sub>0</sub>	6-A, C <sub>0</sub> D, S <sub>0</sub>
Oil and grease	15-A, C <sub>6</sub>	6-A, B, C <sub>6</sub> D <sub>1</sub> S <sub>1</sub>	6-A, B, C <sub>6</sub> D <sub>1</sub> S <sub>0</sub>	9-A, D <sub>0</sub> C <sub>6</sub> S <sub>0</sub>
TOC	12-C <sub>6</sub> P <sub>0</sub> S <sub>0</sub>	12-C <sub>6</sub> P <sub>0</sub> S <sub>0</sub>	12-C <sub>6</sub> P <sub>0</sub> S <sub>0</sub>	9-C <sub>6</sub> D <sub>0</sub> P <sub>0</sub> S <sub>0</sub>
Total cyanide	14-A <sub>0</sub> P <sub>0</sub>	14-A <sub>0</sub> P <sub>0</sub>	11-A <sub>0</sub> P <sub>0</sub> S <sub>0</sub>	8-A <sub>0</sub> D <sub>0</sub> P <sub>1</sub> S <sub>0</sub>
Total phosphorus	14-A <sub>0</sub> P <sub>0</sub>	14-A <sub>0</sub> P <sub>0</sub>	14-A <sub>0</sub> P <sub>0</sub>	11-A <sub>0</sub> D <sub>0</sub> S <sub>0</sub>
PCBs	17-B <sub>2</sub> D <sub>0</sub>	14-A <sub>1</sub> B <sub>2</sub> D <sub>0</sub>	14-A <sub>1</sub> B <sub>2</sub> D <sub>0</sub>	11-A <sub>1</sub> B <sub>2</sub> D <sub>0</sub> S <sub>0</sub>
PAHs	17-D <sub>0</sub> S <sub>2</sub>	11-B <sub>2</sub> D <sub>0</sub> S <sub>1</sub> S <sub>2</sub>	17-D <sub>0</sub> S <sub>2</sub>	20-D <sub>0</sub>
<b>Treated Sediments</b>				
Metals	12-C <sub>0</sub> D <sub>0</sub>	12-C <sub>0</sub> D <sub>0</sub>	12-C <sub>0</sub> D <sub>0</sub>	12-C <sub>0</sub> D <sub>0</sub>
%Moisture	0-A, B, C <sub>0</sub> D, P <sub>0</sub> S <sub>0</sub>	0-A, B, C <sub>0</sub> D, P <sub>0</sub> S <sub>0</sub>	3-A, B, C <sub>0</sub> D, S <sub>0</sub>	3-A, B, C <sub>0</sub> D, S <sub>0</sub>
pH	0-A, B, C <sub>0</sub> D, P <sub>0</sub> S <sub>0</sub>	3-A, B, C <sub>0</sub> D, S <sub>0</sub>	0-A, B, C <sub>0</sub> D, P <sub>0</sub> S <sub>0</sub>	3-A, B, C <sub>0</sub> D, S <sub>0</sub>
%TVS	6-A, C <sub>0</sub> D, S <sub>0</sub>	3-A, B, C <sub>0</sub> D, S <sub>0</sub>	6-A, C <sub>0</sub> D, S <sub>0</sub>	6-A, C <sub>0</sub> D, S <sub>0</sub>
Oil and grease	15-A, C <sub>6</sub>	6-A, B, C <sub>6</sub> D <sub>1</sub> S <sub>1</sub>	9-A, B, C <sub>6</sub> D <sub>1</sub>	6-A, C <sub>6</sub> D <sub>0</sub> P <sub>1</sub> S <sub>0</sub>
TOC	12-C <sub>6</sub> P <sub>0</sub> S <sub>0</sub>	12-C <sub>6</sub> P <sub>0</sub> S <sub>0</sub>	12-C <sub>6</sub> P <sub>0</sub> S <sub>0</sub>	12-C <sub>6</sub> D <sub>0</sub> S <sub>0</sub>
Total cyanide	14-A <sub>0</sub> P <sub>0</sub>	14-A <sub>0</sub> P <sub>0</sub>	14-A <sub>0</sub> P <sub>0</sub>	11-A <sub>0</sub> D <sub>0</sub> P <sub>0</sub>
Total phosphorus	14-A <sub>0</sub> P <sub>0</sub>	14-A <sub>0</sub> P <sub>0</sub>	14-A <sub>0</sub> P <sub>0</sub>	14-A <sub>0</sub> D <sub>0</sub>
PCBs	14-B <sub>2</sub> D <sub>0</sub> P <sub>1</sub>	11-A <sub>1</sub> B <sub>2</sub> D <sub>0</sub> P <sub>1</sub>	14-B <sub>2</sub> D <sub>0</sub> P <sub>1</sub>	14-A <sub>1</sub> B <sub>2</sub> D <sub>0</sub>
PAHs	14-D <sub>0</sub> P <sub>1</sub> S <sub>2</sub>	17-D <sub>0</sub> S <sub>2</sub>	14-D <sub>0</sub> P <sub>1</sub> S <sub>2</sub>	20-D <sub>0</sub>

**TABLE 1. Verified Data Rating Based on the Current ARCS Program  
(Continued)**

<b>Water residue</b>				
Metals	**	**	**	20
%Moisture	**	**	**	**
pH	**	**	**	3-A, B, C, D, S,
Total Suspended Solids	**	**	**	6-A, C, D, S,
%TVS	**	**	**	6-A, C, D, S,
Total Solids				6-A, C, D, S,
Oil and grease	**	**	**	12-A, C, D,
TOC	**	**	**	9-A, C, D, S,
Total cyanide	**	**	**	14-A, D,
Total phosphorus	**	**	**	14-A, D,
Conductivity	**	**	**	9-A, C, D, S,
PCBs	14-B <sub>2</sub> D <sub>0</sub> P <sub>0</sub>	14-B <sub>2</sub> D <sub>0</sub> P <sub>0</sub>	5-A <sub>1</sub> B <sub>2</sub> D <sub>0</sub> P <sub>0</sub> S <sub>1</sub> S <sub>6</sub>	5-A <sub>0</sub> B <sub>2</sub> D <sub>0</sub> P <sub>0</sub> S <sub>5</sub> S <sub>6</sub>
PAHs	11-A <sub>0</sub> D <sub>0</sub> P <sub>1</sub> S <sub>2</sub>	17-D <sub>0</sub> S <sub>2</sub>	17-D <sub>0</sub> P <sub>0</sub>	11-A <sub>1</sub> D <sub>0</sub> P <sub>1</sub> S <sub>1</sub>
<b>Oil residue</b>				
PCBs	11-A <sub>1</sub> B <sub>2</sub> D <sub>0</sub> S <sub>1</sub>	*	17-B <sub>2</sub> D <sub>0</sub>	11-B <sub>2</sub> D <sub>0</sub> P <sub>0</sub> S <sub>5</sub>
PAHs	11-A <sub>0</sub> B <sub>2</sub> D <sub>0</sub> S <sub>2</sub>	*	14-B <sub>2</sub> D <sub>0</sub> S <sub>2</sub>	17-B <sub>2</sub> D <sub>0</sub>

\* No oil residue was produced by this treatment

\*\* Analyses were not conducted for this treatment

**TABLE 2. Full Compliance and Acceptable Scores Based on the Current ARCS QA Program**

Variable Class	Full Compliance	Acceptable
Metals in Treated Sediment	20	12
Metals in Untreated Sediment	20	12
%Moisture	8	5
pH	8	5
%TVS	9	6
Oil and grease	17	11
TOC	17	11
Total cyanide	20	12
Total phosphorus	20	12
Conductivity	14	9
Suspended Solids	9	6
Total Solids	9	6
PAHs	23	14
PCBs	23	14

Table 3 presents the verified data ratings for each variable class in the four technologies based on their full potential value. All data qualifying flags with the subscripts 5, 6, 9, or 0 have been removed. The appropriate point values for each of the 5, 6, or 0 flags (Appendices B and C) were added to the final rating scores for each parameter or parameter group. In contrast, the removal of the subscript 9 flags resulted in an adjustment to the full compliance and acceptable scores, and not in an addition to the calculated point scores since these analyses were not applicable to the methodologies used by the laboratory (Table 2).

**TABLE 3. Verified Data Ratings Based on the Full Potential of the Data set**

<b>Untreated Sediments</b>	<b>B.E.S.T.</b>	<b>ZIMPRO</b>	<b>Soil Tech</b>	<b>RETEC</b>
Metals	20	20	20	20
%Moisture	8	8	8	8
pH	8	8	8	8
%TVS	6	6	6	6
Oil and grease	17	8-B <sub>2</sub> D <sub>1</sub> S <sub>1</sub>	11-B <sub>2</sub> D <sub>1</sub>	17
TOC	17	17	17	17
Total cyanide	20	20	20	17-P <sub>1</sub>
Total phosphorus	20	20	20	20
PCBs	20-B <sub>2</sub>	17-A <sub>1</sub> B <sub>2</sub>	17-A <sub>1</sub> B <sub>2</sub>	17-A <sub>1</sub> B <sub>2</sub>
PAHs	20-S <sub>2</sub>	14-B <sub>2</sub> S <sub>1</sub> S <sub>2</sub>	20-S <sub>2</sub>	23
<b>Treated Sediments</b>				
Metals	20	20	20	20
%Moisture	8	8	8	8
pH	8	8	8	8
%TVS	6	6	6	6
Oil and grease	17	8-B <sub>2</sub> D <sub>1</sub> S <sub>1</sub>	11-B <sub>2</sub> D <sub>1</sub>	9-P <sub>1</sub>
TOC	17	17	17	17
Total cyanide	20	20	20	20
Total phosphorus	20	20	20	20
PCBs	17-B <sub>2</sub> P <sub>1</sub>	14-A <sub>1</sub> B <sub>2</sub> P <sub>1</sub>	17-B <sub>2</sub> P <sub>1</sub>	17-A <sub>1</sub> B <sub>2</sub>
PAHs	17-P <sub>1</sub> S <sub>2</sub>	20- S <sub>2</sub>	20-S <sub>2</sub>	23

**TABLE 3. Verified Data Ratings Based on the Full Potential of the Data set (continued)**

<b>Water residue</b>				
Metals	**	**	**	20
%Moisture	**	**	**	8
pH	**	**	**	8
%TVS	**	**	**	6
Oil and grease	**	**	**	17
TOC	**	**	**	17
Total cyanide	**	**	**	20
Total phosphorus	**	**	**	20
Conductivity	**	**	**	14
Suspended Solids	**	**	**	6
Total Solids	**	**	**	6
PCBs	20-B <sub>2</sub>	20-B <sub>2</sub>	14-A <sub>1</sub> B <sub>2</sub> S <sub>1</sub>	20-B <sub>2</sub>
PAHs	17-P <sub>1</sub> S <sub>2</sub>	20-S <sub>2</sub>	23	14-A <sub>1</sub> P <sub>1</sub> S <sub>1</sub>
<b>Oil residue</b>				
PCBs	14-A <sub>1</sub> B <sub>2</sub> S <sub>1</sub>	*	20-B <sub>2</sub>	20-B <sub>2</sub>
PAHs	17-B <sub>2</sub> S <sub>2</sub>	*	17-B <sub>2</sub> S <sub>2</sub>	20-B <sub>2</sub>

\* No oil residue was produced by this treatment

\*\* Analyses were not conducted for this treatment

To evaluate the data using the values presented in Table 3, the final ratings should be compared to the full compliance and acceptable scores presented in Table 2. The data user should bear in mind that these values are only the potential values of the data set and assumes that the "missing" QA/QC data could have been or were performed successfully by the laboratory. Any value falling below the acceptable value presented in Table 2 clearly indicates that major QA/QC violations were identified and the data should be used with a great deal of caution by the data user.

## Data Verification Results for Bench-scale Technology Demonstration Project

### B.E.S.T.

The B.E.S.T. technology was evaluated by analyzing sediment samples and their treated residues (treated sediments, water residues, and oil residues) for metals, conventionals, PCBs and PAHs. PCB and PAH analyses were performed for sediments, water, and oil residues. The metals and conventional analyses were performed for the sediment samples only.

In the majority of the cases studied, the accuracy objective was satisfactory for the metal analyses in treated and untreated sediments. Of the thirteen metals analyzed, accuracy information was not available for Ba, Se, and Ag. In both treated and untreated sediments, ten of the thirteen metal analyses (As, Cd, Cr, Cu, Fe, Pb, Mn, Hg, Ni, Pb, and Zn) satisfied ARCS specified QA/QC requirements for accuracy. Four of the thirteen metal analyses (Cd, Hg, Se, and Ag) satisfied QA/QC requirements for blank analyses, while the remaining nine metals (As, Ba, Cr, Cu, Fe, Mn, Ni, Pb, and Zn) were analyzed by XRF techniques. In all of the XRF analyses, results from blank sample analyses were not applicable. Both initial and ongoing calibration for Cd, Hg, Se, and Ag analyses met the ARCS QA/QC specifications for both treated and untreated sediments, while for the remaining nine metals (As, Ba, Cr, Cu, Fe, Mn, Ni, Pb, and Zn) calibration information was not available. Detection limits information for metal analyses in treated and untreated sediments were not available for verification except for Cd, Hg, Se, and Ag where detection limits were satisfactory. The precision information for the metal analyses in treated sediment was not available for Se, but was satisfactory for the remaining elements, with the exception of Hg, where precision information did not satisfy QA/QC requirements. The precision information for the metal analyses in untreated sediment was not available for Se, but was satisfactory for the remaining twelve metal (Ag, As, Ba, Cd, Cr, Cu, Fe, Hg, Mn, Ni, Pb, and Zn) analyses. The matrix spike information for both treated and untreated sediment analyses were satisfactory for Cd, Hg, and Se, were unsatisfactory for Ag, while the remaining nine metals (As, Ba, Cr, Cu, Fe, Mn, Ni, Pb, and Zn) were analyzed by XRF techniques. In all of the XRF analyses, results from matrix spike analyses were not applicable.

Of the seven conventional analyses, the accuracy information in both treated and untreated sediments was satisfactory for TOC and was not available for total cyanide, and total phosphorus. In the remaining four conventional analyses, accuracy was not applicable. In both sediments, five of the seven conventionals (%TVS, oil and grease, TOC, total cyanide, and total phosphorus) satisfied QA/QC requirements for blank analyses, and the blank information was not applicable for moisture, pH, and TVS. Both initial and ongoing calibration information was satisfactory for all conventional analyses in both treated and untreated sediments except for moisture and pH where calibration information was not available and for TOC and oil and grease where ongoing calibration information was not available. Detection limits were satisfactory for four (oil and grease, TOC, total cyanide, and total phosphorus) of the seven conventional

analyses in treated and untreated sediments, and were not applicable for moisture, pH, and TVS. The precision information was satisfactory for two (%TVS, oil and grease) of the seven conventional analyses in treated and untreated sediments. No precision information was available for the remaining five conventional analyses in treated or untreated sediments. The matrix spike information for both treated and untreated sediment analyses were satisfactory for oil and grease, total cyanide, and total phosphorus, while for the remaining four conventional analyses the matrix spike information was not applicable.

In treated sediments, untreated sediments, and water residues, the accuracy objective for PCBs was satisfactory for Aroclor 1254 analyses only and could be used to represent the whole PCB group. No accuracy information was available for the remaining three Aroclor analyses. In oil residues, accuracy information was not satisfactory for PCB analyses. In both sediments and in both residues, PCB analyses did not satisfy ARCS specified QA/QC requirements for blank analyses indicating potential contamination at the laboratory. Initial and ongoing calibration was satisfactory for all PCB analyses in both treated and untreated sediments as well as in water and oil residues. Detection limit information were not available for PCB analyses in treated and untreated sediments and for water and oil residues. In the untreated sediments, the precision information was satisfactory for Aroclors 1242 and 1254, and no precision information was available for Aroclors 1248 and 1260. In the treated sediments, the precision information was not satisfactory for Aroclor 1254, and no precision information was available for Aroclors 1242, 1248, and 1260. In water residues, no precision information was available for any of the Aroclors. In oil residues, the precision information was satisfactory for Aroclor 1248, and no precision information was available for Aroclors 1242, 1254, and 1260. The matrix spike for Aroclor 1254 was satisfactory for both sediment and water residue analyses and could be used to represent the whole PCB group. The matrix spike for Aroclor 1254 was unsatisfactory for the analyses of oil residue. In both sediment or residue analyses, no matrix spike information was available for Aroclors 1242, 1248, and 1260. The surrogate spike recoveries were satisfactory for PCB analyses in both sediments and residues.

In eight of sixteen PAH analyses of treated and untreated sediments, the accuracy objective was satisfactory. No accuracy information was available for six PAHs (naphthalene, acenaphthylene, acenaphthene, fluorene, chrysene, and dibenzo(a,h)anthracene) analyses in both treated and untreated sediments. The accuracy objective was not satisfactory for benzo(k) fluoranthene and benzo(a)pyrene in treated or untreated sediments. No accuracy information was available for any of the PAH analyses in water and oil residues. In treated and untreated sediments, and in water residues, PAH analyses satisfied ARCS specified QA/QC requirements for blank analyses. In all cases of oil residues, the blank analyses exceeded the MDL indicating potential contamination at the laboratory. Initial and ongoing calibration limits for PAH analyses met the ARCS QA/QC specifications for both treated and untreated sediments and water and oil residue analyses. Detection limit information was not available for PAH analyses in treated and untreated sediments, nor for water and oil residues. In untreated sediments and oil residues, the precision information was satisfactory for all PAH analyses, except for acenaphthene in untreated sediment, and naphthalene in oil residues where no precision information was available. In treated sediments, the precision information was satisfactory for fluorene, phenanthrene, and

anthracene but was unsatisfactory for the remaining PAH analyses. In water residues, no precision information was available for PAH analyses except for benzo(g,h,i)pyrene where precision was unsatisfactory. The matrix spike information was satisfactory for twelve of sixteen PAH analyses in treated sediment and for eight of the sixteen analyses in untreated sediment and in water and oil residues. Surrogate recoveries were not satisfactory for PAHs in either sediment and residue analyses.

## ZIMPRO

The ZIMPRO technology was evaluated by analyzing sediment samples, treated sediments, and water residues for metals, conventionals, PCBs, and PAHs. PCB and PAH analyses were performed for both sediment and water residues. The metals and conventional analyses were performed for the both sediment samples only.

In the majority of the cases studied, the accuracy objective was satisfactory for the metal analyses in treated and untreated sediments. Of the thirteen metals analyzed, accuracy information was not available for Ba, Se, and Ag. In both treated and untreated sediments, ten of the thirteen metal analyses (As, Cd, Cr, Cu, Fe, Pb, Mn, Hg, Ni, and Zn) satisfied ARCS specified QA/QC requirements for accuracy. Four of the thirteen metal analyses (Cd, Hg, Se, and Ag) satisfied QA/QC requirements for blank analyses, while the remaining nine metals (As, Ba, Cr, Cu, Fe, Mn, Ni, Pb, and Zn) were analyzed by XRF techniques. In all of the XRF analyses, blank sample analyses are not applicable. Both initial and ongoing calibration for Cd, Hg, Se, and Ag analyses met the ARCS QA/QC specifications for both treated and untreated sediments while for the remaining nine metals (As, Ba, Cr, Cu, Fe, Mn, Ni, Pb, and Zn), calibration information was not available. Detection limit information for metal analyses in treated and untreated sediments was not available for verification except for Cd, Hg, Se, and Ag where the detection limits were satisfactory. The precision for the metal analyses in treated sediment was not satisfactory for As, but was satisfactory for the remaining elements. The precision information for the metal analyses in untreated sediment was satisfactory for all elements. The matrix spike information for both treated and untreated sediment analyses were satisfactory for four (Cd, Hg, Se, and Ag) of the thirteen elements while the remaining nine metals (As, Ba, Cr, Cu, Fe, Mn, Ni, Pb, and Zn) were analyzed by XRF techniques. In all of the XRF analyses, results from matrix spike analyses were not applicable.

Of the seven conventional analyses, the accuracy information in both treated and untreated sediments was satisfactory for TOC and was not available for total cyanide, and total phosphorus. In the remaining four conventional analyses, accuracy was not applicable. In both sediments, three of the seven conventionals (TOC, total cyanide, and total phosphorus) satisfied QA/QC requirements for blank analyses. The blank information was unsatisfactory for oil and grease, was not available for %TVS, and the blank information was not applicable for moisture and pH. Both initial and ongoing calibration information was satisfactory for all conventional analyses in both treated and untreated sediments except for %moisture, pH, and TVS where calibration information was not available, and for TOC and oil and grease, where ongoing

calibration information was not available. Detection limits were satisfactory for three (TOC, total cyanide, and total phosphorus) of the seven conventional analyses in treated and untreated sediments. Detection limits were unsatisfactory for oil and grease analyses in treated and untreated sediments and were not applicable for %moisture, pH, and %TVS. The precision information was satisfactory for pH, %TVS, and oil and grease analyses in treated, and for %moisture, %TVS, and oil and grease analyses in untreated sediment. No precision information was available for %moisture, TOC, total cyanide, and total phosphorus analyses in treated sediment and for pH, TOC, total cyanide, and total phosphorus analyses in untreated sediments. The matrix spike information for both treated and untreated sediment analyses were satisfactory for total cyanide and total phosphorus, were unsatisfactory for oil and grease while for the remaining four conventional analyses the matrix spike information was not applicable.

The accuracy objective was unsatisfactory for the PCB analyses in treated and untreated sediments for Aroclor 1254. No accuracy information was available for the remaining three Aroclor analyses in treated and untreated sediments. In water residue, the accuracy objective for PCBs was satisfactory for Aroclor 1254 analyses only and could be used to represent the whole PCB group. No accuracy information was available for the remaining three Aroclor analyses in water residues. In water residues and in both treated and untreated sediments, the blank analyses exceeded the detection limits specified in the QAPP indicating potential contamination at the laboratory. Initial and ongoing calibration was satisfactory for all PCB analyses in both treated and untreated sediments as well as in water residues. Detection limits information were not available for PCB analyses in treated and untreated sediments, nor in the water residues. In untreated sediment analyses, most PCB observations were below the instrument detection limits, therefore it was not possible to calculate meaningful precision information for PCB Aroclors, with the exception of Aroclor 1248 analyses, where precision information satisfied QA/QC requirements. No precision information was available for PCB analyses in treated sediments, except for Aroclor 1254 in treated sediment where it did not satisfy QA/QC requirements. In the water residue, no PCB precision information was available. The matrix spike for Aroclor 1254 was satisfactory for both sediments, and the water residue analyses and could be used to represent the whole PCB group. The matrix spike information for sediments and water residue analyses for Aroclor 1242, 1248, and 1260 were not available for verification. The surrogate recoveries were satisfactory for PCB analyses in sediment and residue analyses.

In ten of the sixteen PAH analyses in treated sediment and nine of the sixteen PAH analyses in untreated sediments, the accuracy objective was satisfactory. No accuracy information was available for six PAHs (naphthalene, acenaphthylene, acenaphthene, fluorene, chrysene, and dibenzo(a,h)anthracene) analyses in treated and untreated sediment. The accuracy objective was not satisfactory for benzo(k)fluoranthene in untreated sediment. Accuracy information in water residue was unsatisfactory for naphthalene, acenaphthylene, acenaphthene, phenanthrene, and benzo(a)pyrene. Accuracy was satisfactory for the rest of the PAH analyses in water residues. In treated sediments and water residues, PAH analyses satisfied ARCS specified QA/QC requirements for blank analyses. In all cases of untreated sediment analyses, the blank analyses exceeded the detection limit specified in the QAPP. Calibration limits for

PAH analyses met the ARCS QA/QC specifications for both treated and untreated sediments, and also for water residue analyses. Detection limits information were not available for PAH analyses in treated and untreated sediments, nor for the water residues. The precision information was satisfactory for PAH analyses in both sediments except for naphthalene, acenaphthylene, acenaphthene, fluorene, and benzo(a)pyrene analyses in treated sediment and for naphthalene, acenaphthene, phenanthrene, and benzo(a)pyrene in water residue, where precision was unsatisfactory. The matrix spike information was satisfactory for fifteen of the sixteen PAH analyses in treated sediment, for five of the sixteen analyses in untreated sediment and for eleven of the sixteen analyses in water residues. Surrogate recoveries were not satisfactory for PAHs in the sediment and residue analyses.

### SOIL TECH

The Soil Tech technology was evaluated by analyzing sediment samples and their treated residues (treated sediments, water residues, and oil residues) for metals, conventionals, PCBs, and PAHs. PCB and PAH analyses were performed for sediment and residues. The metals and conventional analyses were performed for the sediment samples only.

In the majority of the cases studied, the accuracy objective was satisfactory for the metal analyses in treated and untreated sediments. Of the thirteen metals analyzed, accuracy information was not available for Ba, Se, and Ag. Four of the thirteen metal analyses (Cd, Hg, Se, and Ag) satisfied QA/QC requirements for blank analyses, while the remaining nine metals (As, Ba, Cr, Cu, Fe, Mn, Ni, Pb, and Zn) were analyzed by XRF techniques. In all of the XRF analyses, blank sample analyses are not applicable. Both initial and ongoing calibration for Cd, Hg, Se, and Ag analyses met the ARCS QA/QC specifications for both treated and untreated sediments while for the remaining nine metals (As, Ba, Cr, Cu, Fe, Mn, Ni, Pb, and Zn), calibration information was not available. Detection limits information for metal analyses in treated and untreated sediments were not available for verification except for Cd, Hg, Se, and Ag where detection limits were satisfactory. The precision information for the metal analyses in treated sediment was not available for Se and Hg but was satisfactory for the remaining elements with the exception of Cr, where precision information did not satisfy the QA/QC requirements. The precision information for the metal analyses in untreated sediment was satisfactory for all metal analyses. The matrix spike information were satisfactory for four (Cd, Hg, Se, and Ag) of the thirteen elements for treated sediments and two (Cd, Hg) of the thirteen elements for untreated sediments. The matrix spike information were unsatisfactory for Se and Ag analyses in untreated sediments. The remaining nine metals (As, Ba, Cr, Cu, Fe, Mn, Ni, Pb, and Zn) were analyzed by XRF techniques. In all of the XRF analyses, results from matrix spike analyses were not applicable.

Of the seven conventional analyses, the accuracy information in both treated and untreated sediments was satisfactory for TOC and was not available for total cyanide, and total phosphorus. In the remaining four conventional analyses, accuracy was not applicable. In both sediments, four of the seven conventionals (%TVS, TOC, total cyanide, and total phosphorus)

satisfied QA/QC requirements for blank analyses, and the blank information was not applicable for moisture and pH, while blank analyses was not satisfactory for oil and grease. Both initial and ongoing calibration information was satisfactory for all conventional analyses in both treated and untreated sediments, except for %moisture, pH, and %TVS where calibration information was not available. Ongoing calibration information was not available for TOC and oil and grease. Detection limits were satisfactory for three (TOC, total cyanide, and total phosphorus) of the seven conventional analyses in treated and untreated sediments. Detection limits were unsatisfactory for oil and grease and were not applicable for %moisture, pH, and %TVS. The precision information was satisfactory for %moisture, %TVS, and oil and grease in treated sediments. The precision information was satisfactory for %TVS, and oil and grease in treated sediments. No precision information was available for the remaining conventional analyses in treated or untreated sediments. The matrix spike information were satisfactory for oil and grease, total phosphorus, and total cyanide in treated sediment analyses and for total phosphorus in untreated sediment analyses. The matrix spike information were not available for oil and grease and total cyanide in untreated sediment analyses. While for the remaining four conventional analyses, the matrix spike information was not applicable.

The accuracy objective was satisfactory for the PCB analyses in treated sediments and in oil residue analyses for Aroclor 1254 only and could be used to represent the whole PCB group. The accuracy objective was unsatisfactory for the PCB analyses in untreated sediments and in water residue analyses for Aroclor 1254. No accuracy information was available for the remaining three Aroclor analyses in sediment or residue analyses. In both residues and in both treated and untreated sediments, the blank analyses exceeded the detection limits specified in the QAPP, except for Aroclor 1260 in oil residue. Initial and ongoing calibration was satisfactory for all PCB analyses in both treated and untreated sediments, as well as in both water and oil residues. Detection limit information was not available for PCB analyses in both sediments and residues. In untreated sediment analyses, most PCB observations were below the instrument detection limits, therefore, it was not possible to calculate meaningful precision information for PCB Aroclors, with the exception of Aroclor 1248 analyses, where precision information satisfied QA/QC requirements. No precision information was available for PCB analyses in treated sediment, except for Aroclor 1254, where it did not satisfy QA/QC requirements. No precision information was available for PCB analyses in oil and water residues, except for Aroclor 1248 in oil residue, where precision was satisfactory. The matrix spike for Aroclor 1254 was satisfactory for both sediments and the oil residue analyses and could be used to represent the whole PCB group. The matrix spike for Aroclor 1254 was unsatisfactory for the water residue analyses, and the matrix spike information for both sediment and residue analyses for Aroclor 1242, 1248, and 1260 were not available for verification. The surrogate recoveries were satisfactory for PCB analyses in sediment and residue analyses, except for water residue where surrogate information was not available.

In eight of sixteen PAH analyses in treated and untreated sediments, the accuracy objective was satisfactory. No accuracy information was available for six PAHs (naphthalene, acenaphthylene, acenaphthene, fluorene, chrysene, and dibenzo(a,h)anthracene) analyses in both treated and untreated sediments. The accuracy objective was not satisfactory for benzo(k)

fluoranthene in treated or untreated sediments nor for benzo(g,h,i)perylene in untreated sediment. Accuracy information was satisfactory for the PAH analyses in water and oil residues. In treated and untreated sediments and water residues, PAH analyses satisfied ARCS specified QA/QC requirements for blank analyses. In all cases of oil residues, the blank analyses exceeded the MDL. Calibration limits for PAH analyses met the ARCS QA/QC specifications for both treated and untreated sediments as well as water and oil residue analyses. Detection limit information was not available for PAH analyses in treated and untreated sediments nor for water and oil residues. In untreated sediment and oil residues, the precision information was satisfactory for all PAH analyses, except for acenaphthene and acenaphthene in untreated sediment, and naphthalene in oil residues, where no precision information was available. In treated sediments, the precision information was satisfactory for naphthalene, acenaphthylene acenaphthene, fluorene, phenanthrene, and anthracene, and was unsatisfactory for the remaining PAH analyses. In water residues, no precision information was available for any of the PAH analyses. The matrix spike information was satisfactory for twelve of sixteen PAH analyses in treated sediment, and for thirteen of the sixteen analyses in untreated sediment and ten of the sixteen analyses in water and all analyses in oil residues. Surrogate recoveries were unsatisfactory for PAHs in either sediment and oil residue analyses but were satisfactory in water residue.

## RETEC

The RETEC technology was evaluated by analyzing sediment samples and their treated residues (water residues and oil residues) for metals, conventionals, PCBs and PAHs. PCB and PAH analyses were performed for sediment and residues. The metals and conventional analyses were performed for both sediment samples and water residues.

In a majority of the cases studied, the accuracy objective was satisfactory for the metal analyses in treated and untreated sediments. Of thirteen metals analyzed, accuracy information was not available for Ba, Se, and Ag. In both treated and untreated sediments, ten of the thirteen metal analyses (As, Cd, Cr, Cu, Fe, Pb, Mn, Ni, Hg, and Zn) satisfied ARCS specified QA/QC requirements for accuracy. The accuracy objective was satisfactory for all metal analyses in water, except for Se, where accuracy did not satisfy QA/QC requirements. Four of the thirteen metal analyses (Cd, Hg, Se, and Ag) satisfied QA/QC requirements for blank analyses. The remaining nine metal analyses (As, Ba, Cr, Cu, Fe, Pb, Mn, Ni, and Zn) were analyzed by XRF techniques. In all of the XRF analyses, blank sample analyses are not applicable. In water residue, blank analyses were satisfactory for all metals except for Fe, Mn, and Se, where blank analyses exceeded the detection limits specified in the QAPP, and for Ba, where no information regarding blank analyses was available. Both initial and ongoing calibration met the ARCS QA/QC specifications for Cd, Hg, Se, and Ag for both treated and untreated sediments, and for all metals in water residue analyses. While in both treated and untreated sediments the remaining nine metals (As, Ba, Cr, Cu, Fe, Pb, Mn, Ni, and Zn), calibration information were not available. Detection limits information for metal analyses in treated and untreated sediments were not available for verification, except for Cd, Hg, Se, and

Ag, where detection limits were satisfactory. Detection limits for metal analyses in water residue were satisfactory, except for Mn, Se, and Zn, where detection limits exceeded the QA/QC requirements. The precision information for the metal analyses in treated and untreated sediments, and in water residue was satisfactory for all elements, except for Hg in treated sediment, and Se and Hg in water residue analyses, where precision information did not satisfy QA/QC requirements. The matrix spike information for treated sediment analyses were satisfactory for Cd, Hg, and Ag, and was not satisfactory for Se. The matrix spike information for untreated sediment analyses were satisfactory for Cd and Hg, and was not satisfactory for Se and Ag. The remaining nine metals (As, Ba, Cr, Cu, Fe, Pb, Mn, Ni, and Zn) were analyzed by XRF techniques for treated and untreated sediment. In all of the XRF analyses, matrix spike analyses are not applicable. The matrix spike information for water residue analyses was satisfactory for all metals except for Ag where matrix spike information did not satisfy QA/QC requirement.

Of the seven conventional analyses in both treated and untreated sediments, accuracy information was satisfactory for TOC, and was not available for total cyanide, or total phosphorus. In the remaining four conventional analyses accuracy was not applicable. Of ten conventional analyses in water residue, accuracy information was not available for TOC, total cyanide, total phosphorus, and conductivity. In the remaining seven conventional analyses accuracy was not applicable. In both treated and untreated sediments and in water residue analyses, %TVS, oil and grease, TOC, total cyanide, and total phosphorus satisfied QA/QC requirements for blanks. Also, the blank information was satisfactory for total solids and total suspended solids in water residue analyses. The blank information was not applicable for the remaining conventional analyses in sediment and water residue analyses. Both initial and ongoing calibration information was satisfactory for all conventional analyses in both sediment and water residue, except for %moisture (in sediment), pH, and TVS, TSS, TS where calibration information was not available, and for TOC and oil and grease, where ongoing calibration information was not available. Detection limit information was not available in both treated and untreated sediments and in water residue for oil and grease, TOC, total cyanide, and total phosphorus, and was not applicable for the remaining conventional analyses. In treated sediment, the precision information was not satisfactory for oil and grease and no precision information was available for total cyanide. In untreated sediment, the precision information was not satisfactory for total cyanide, and no precision information was available for TOC. The precision information was satisfactory for the remaining five conventional analyses in treated and untreated sediments. In water residue, the precision information was satisfactory for all the conventionals, except for moisture, where no precision information was available. The matrix spike information was not available for oil and grease, and was satisfactory for total cyanide and total phosphorus in treated sediment analyses. The matrix spike information was not available for oil and grease, total cyanide, and total phosphorus in untreated sediment analyses. The matrix spike information was satisfactory for oil and grease, total cyanide, and total phosphorus in water residue analyses. The matrix spike information for the remaining conventional analyses was not applicable for sediment and water residue analyses.

The accuracy objective was unsatisfactory for the PCB analyses in treated sediments, untreated sediments, and oil residue for Aroclor 1254 and could be used to represent the whole PCB group. No accuracy information was available for the remaining three Aroclor analyses in treated and untreated sediments. No accuracy information was available for PCB analyses in water residues. In both sediments and residues, the blank analyses exceeded the detection limits specified in the QAPP. Both initial and ongoing calibration for PCB analyses met the ARCS QA/QC specifications for both treated and untreated sediments, as well as for water and oil residues. Detection limit information was not available for PCB in either sediments or residue analyses. The precision information for the PCB analyses in treated and untreated sediment was satisfactory for Aroclor 1254. In all remaining analyses, precision information was not available. The matrix spike was satisfactory for Aroclor 1254 in treated sediment and in oil residue analyses, and could be used to represent the whole PCB group. The matrix spike information was not available for the remaining Aroclors in treated sediment and oil residues. The matrix spike information was not available for PCB analyses in untreated sediment and in water residues. The surrogate recoveries were satisfactory for PCB analyses in sediment and residue analyses.

In ten of the sixteen PAH analyses in treated sediments and in seven of the sixteen PAH analyses in untreated sediments, the accuracy objective was satisfactory. No accuracy information was available for six PAHs (naphthalene, acenaphthylene, acenaphthene, fluorene, chrysene, dibenzo(a,h)anthracene) analyses in treated and untreated sediment. The accuracy objective was not satisfactory for benzo(k)fluoranthene, benzo(a)pyrene, and benzo(g,h,i)perylene in untreated sediment. Accuracy information was satisfactory for fourteen of the sixteen PAH analytes in oil residue. Accuracy information was unsatisfactory for PAH analyses in water residue, except for benzo(k)fluoranthene, indeno(1,2,3,c,d)pyrene, dibenzo(a,h)anthracene. The blank analyses for the PAHs in treated and untreated sediment was satisfactory in all cases except for acenaphthylene, acenaphthene, fluorene, phenanthrene, and anthracene. In water residues, all PAH analyses satisfied ARCS specified QA/QC requirements for blank analyses. In all oil residues, the blank analyses exceeded the detection limit specified in the QAPP. Both initial and ongoing calibration information for PAH analyses met the ARCS QA/QC specifications for both treated and untreated sediments, and also for water and oil residue analyses. Detection limit information was not available for PAH analyses in either sediments or residues. The precision information was satisfactory for PAH analyses in treated sediments, except for benzo(k)fluoranthene, where precision did not satisfy QA/QC requirements. The precision information was satisfactory for PAH analyses in untreated sediments except for acenaphthylene and acenaphthene, where precision information was not available, and for benzo(k)fluoranthene, where precision did not satisfy QA/QC requirements. The precision information was satisfactory for PAH analyses in oil residue, except for benzo(k)fluoranthene, where precision information did not satisfy QA/QC requirements. In water residue, precision was unsatisfactory for PAH analyses except for benzo(k)fluoranthene, indeno(1,2,3,c,d)pyrene, and dibenzo(a,h)anthracene, where precision was satisfactory. The matrix spike information was satisfactory for ten of the sixteen PAH analytes in treated sediment, for fourteen of the analytes in untreated sediment, for thirteen of the analytes in oil residues, and for three of the analytes in water residues. Surrogate recoveries were satisfactory

for PAHs in both treated and untreated sediments as well as for oil and water residue analyses.

### **Summary**

Based on the compliance with the ARCS QA/QC requirements, SAIC was capable of supplying acceptable results for metals, conventionals, PCBs, and PAHs. The results received for all four technologies satisfied ARCS QA/QC requirements.

An examination of results of the bench scale technology demonstration data set indicates, that SAIC could have successfully provided acceptable data for all parameters. The data user should be aware that some QA/QC discrepancies were identified, as indicated by subscript 1 and 2 flags in Table 3.

**APPENDICES A and D**

**are not included in this report.**

**Copies are available from GLNPO upon request.**

**APPENDIX B**

**QA/QC Sample Rating Factors**

<u>CATEGORY</u>	<u>RATING FACTORS</u>	<u>SCORE</u>	<u>CATEGORY ACCEPTABILITY LEVEL</u>
<u>Accuracy</u>	Certified Reference Material	= 3	Acceptable = 3
<u>Precision</u>	Analytical Replicate	= 3	Acceptable = 3
<u>Spike Recovery</u>	Matrix Spike	= 3	Acceptable = 3
	Surrogate Spike (organics)	= 3	(organics) = 6
<u>Blanks</u>	Blanks	= 3	Acceptable = 3
<u>Miscellaneous</u>	Instrument Calibration (initial)	= 3	
	Instrument Calibration (on going)	= 2	
	Instrument Detection Limit	= 3	Acceptable = 3

## **APPENDIX C**

### **Data Verification Flags**

**A = Accuracy Problem**

**A<sub>0</sub> = no standard available/no information available**

**A<sub>1</sub> = accuracy limit for the reference materials exceeded**

**A<sub>9</sub> = accuracy is not applicable**

**B = Blank Problem**

**B<sub>0</sub> = no information available**

**B<sub>2</sub> = reagent blank value exceeded MDL**

**B<sub>9</sub> = blanks are not applicable**

**C = Calibration Problem**

**C<sub>0</sub> = no information available**

**C<sub>1</sub> = initial calibration problem**

**C<sub>2</sub> = on-going calibration problem**

**C<sub>3</sub> = no information on initial calibration**

**C<sub>6</sub> = no information on on-going calibration**

**C<sub>9</sub> = on-going calibration is not applicable**

**D = Detection Limit Problem**

**D<sub>0</sub> = no information available**

**D<sub>1</sub> = detection limit exceeded**

**D<sub>9</sub> = detection limit is not applicable**

**H = Holding Times Exceeded**

**P = Precision Problem**

**P<sub>0</sub> = no information available**

**P<sub>1</sub> = precision limit for analytical replicate exceeded the QA/QC requirements**

**P<sub>3</sub> = MSD exceeded the QA/QC requirement**

**P<sub>9</sub> = precision is not applicable**

**S = Spike Recovery Problem**

**S<sub>0</sub> = no information available on spike**

**S<sub>1</sub> = limit of matrix spike recovery exceeded**

**S<sub>2</sub> = limit of surrogate spike recovery exceeded**

**S<sub>3</sub> = no information available on matrix spike recovery**

**S<sub>6</sub> = no information available on surrogate spike recovery**

**S<sub>9</sub> = spike recovery not applicable**