



Assessment and Remediation Of Contaminated Sediments (ARCS) Program

BENCH-SCALE EVALUATION OF ReTEC'S THERMAL DESORPTION TECHNOLOGY ON CONTAMINATED SEDIMENTS FROM THE ASHTABULA RIVER



**Bench-Scale Evaluation of
ReTeC's Thermal Desorption Technology on
Contaminated Sediments from the Ashtabula River**

Prepared by

Michael Giordano and Evelyn Meagher-Hartzell
Science Applications International Corporation
Cincinnati, Ohio

for the
Assessment and Remediation of Contaminated Sediments (ARCS) Program
U.S. Environmental Protection Agency
Great Lakes National Program Office
Chicago, Illinois

U.S. Environmental Protection Agency
Region 5, Library (PL-12J)
77 West Jackson Boulevard, 12th Floor
Chicago, IL 60604-3590

DISCLAIMER

The information in this document has been funded wholly or in part by the U.S. Environmental Protection Agency (EPA) under Contract No. 68-C8-0062, Work Assignment No. 3-52, to Science Applications International Corporation (SAIC). It has been subjected to the Agency's peer and administrative review and it has been approved for publication as an EPA document. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

ACKNOWLEDGEMENTS

This report was prepared by the Engineering/Technology Work Group (ETWG) as part of the Assessment and Remediation of Contaminated Sediments (ARCS) program. Dr. Stephen Yaksich, U.S. Army Corps of Engineers (USACE) Buffalo District, was chairman of the Engineering/Technology Work Group.

The ARCS Program was managed by the U.S. Environmental Protection Agency (USEPA), Great Lakes National Program Office (GLNPO). Mr. David Cowgill and Dr. Marc Tuchman of GLNPO were the ARCS program managers. Mr. Dennis Timberlake of the USEPA Risk Reduction Engineering Laboratory was the technical project manager for this project. Mr. Stephen Garbaciak of USACE Chicago District and GLNPO was the project coordinator.

This report was drafted through Contract No. 68-C8-0062, Work Assignment No. 3-52, to Science Applications International Corporation (SAIC). Michael Giordano and Evelyn Meagher-Hartzell of SAIC were the principal authors of the report, with final editing and revisions made by Mr. Garbaciak prior to publication.

This report should be cited as follows:

U.S. Environmental Protection Agency. 1994. "Bench-Scale Evaluation of ReTeC's Thermal Desorption Technology on Contaminated Sediments from the Ashtabula River," EPA 905-R94-008, Great Lakes National Program Office, Chicago, IL.

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. A bench-scale study using the ReTeC Thermal Desorption 1000 lb/hr technology is the subject of this report. This study took place at Star Refinery in Delaware City, DE on September 25, 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 ReTeC Holo-Flite™ Screw Processor was tested using a sediment sample obtained from the Ashtabula River. The concentrations of the contaminants of concern in the sediment were 14.6 mg/kg PCBs and 6.1 mg/kg PAHs. The PCB and PAH concentrations of <0.6 and <2.4 mg/kg, respectively, were found in the treated solids. This corresponds to PCB and PAH removals of >96 and >60 percent, respectively. Since the concentrations of individual PAHs in the feed and treated solids are very close to detection limits, significant error is associated with the calculated PAH removals. The percent removal achieved for PAHs can be attributed to the method used to quantify the individual PAHs, making this result an unreliable reflection of the technology's ability to remove PAHs. Metals analyses were performed on the treated solids and untreated sediments. The data demonstrate that except for mercury, there is no indication that the ReTeC technology effectively removes metals. The feed and treated solids were analyzed for percent moisture, oil and grease, total organic carbon (TOC), volatile solids, and pH. Moderate reductions were experienced for oil and grease and total volatile solids (i.e., 56.6 percent and 44.4 percent, respectively). Because of the relatively small amount of material treated, accurate mass balances were not possible.

TABLE OF CONTENTS

<u>Section</u>	<u>Page</u>
Disclaimer	i
Acknowledgements	ii
Abstract	iii
List of Figures	v
List of Tables	vi
 1.0 Executive Summary	 1
 2.0 Introduction	 2
2.1 Background	3
2.2 Sediment Descriptions	3
2.3 Sediment Characterization	6
2.4 Technology Description	6
 3.0 Treatability Study Approach	 7
3.1 Test Objectives and Rationale	7
3.2 Experimental Design and Procedures	10
3.3 Sampling and Analysis	13
 4.0 Results and Discussion	 14
4.1 Summary of Phase I Results	14
4.2 Phase II Results	14
4.3 Summary of Vendor Results	25
4.4 Quality Assurance/Quality Control	25
 Appendix A - Thermal Treatability Testing, Ashtabula River Sediments, Great Lakes Program Office	 33
Appendix B - Quality Assurance/Quality Control	79
Appendix C - Quality Assurance Project Plan	105
Appendix D - Battelle Analytical Results	151

LIST OF FIGURES

<u>Number</u>		<u>Page</u>
1	ARCS Priority Areas of Concern	4
2	Ashtabula River Sampling Points	5
3	Process Flow Diagram	8

LIST OF TABLES

<u>Number</u>		<u>Page</u>
1	Summary of Total PCBs and PAHs	1
2	Mass Balance Summary	2
3	Characterization of the Ashtabula River Sediment	6
4	Parameters for Analysis of ARCS Program Technologies	10
5	Full-Scale Holo-Flite™ Screw Processor Specifications	12
6	Analytical Methods Used by ReTeC During Phase I Testing	13
7	ReTeC Analytical Matrix and Sample Identification-Ashtabula River Sediment	15
8	Optimal Operating Parameters	16
9	Total PCBs	16
10	Feed and Treated Solids PAH Concentrations	18
11	Metals Concentration in the Feed and Treated Solids	19
12	Removal Efficiencies for Other Parameters	20
13	PAH Concentrations in the Treated Solids, Water, and Oil	21
14	PCB Concentrations in the Treated Solids, Water, and Oil	21
15	Metals Concentration in the Residual Water	22
16	Residual Water Characterization Data	22
17	Solids Mass Balance	24
18	Water Mass Balance	24
19	Oil Mass Balance	25
20	PAH Mass Balance	26

1.0 EXECUTIVE SUMMARY

The ReTeC Holo-Flite™ Screw Processor was tested using sediment obtained from the Ashtabula River. The contaminants of concern in the sediment were PCBs and PAHs. Samples of the feed and the treated solids produced using the ReTeC technology were analyzed by Battelle Marine Sciences Laboratory for PCB and PAH contamination. The data from these analyses are presented in Table 1.

**Table 1. Summary of Total PCBs and PAHs
(mg/kg, dry)**

Parameter	Feed	Treated Solids	% Removal
Total PCBs	14.6	<0.6	>96
Total PAHs	6.1	<2.4	>60

The data in Table 1 indicate that PCB and PAH concentrations of <0.6 and <2.4 mg/kg, respectively, were found in the treated solids. This corresponds to PCB and PAH removal of >96 and >60 percent, respectively. Since the concentrations of individual PAHs in the feed and treated solids are very close to detection limits, significant error is associated with the calculated PAH removal. The percent removal achieved for PAHs can be attributed to the method used to quantify the individual PAHs, making this result an unreliable reflection of the technology's ability to remove PAHs.

Metal analyses were performed on the treated solids and untreated sediments. The data demonstrate that except for mercury, there is no indication that the ReTeC technology effectively removes metals. The feed and treated solids were analyzed for percent moisture, oil and grease, TOC, volatile solids, and pH. Moderate reductions were experienced for oil and grease and total volatile solids (i.e., 56.6 percent and 44.4 percent, respectively). The results of these analyses are discussed in more detail in Section 4.2.

Given the size of the unit employed during testing (1000 lb/hr), the relative small amount of material available for treatment (460 lbs), and the relatively large amount of material trapped within the system following treatment (approximately 107 lbs, assuming perfect recovery), accurate mass balances could not be calculated. To address the issue of equipment contamination fully, rough mass balances were performed for solids, water, oils, and PAHs. As shown in Table 2, the excessively high values obtained for oil and PAHs support suspicions of processor contamination.

Table 2. Mass Balance Summary (%)

Sample	Solids	Water	Oil	PAHs
Ashtabula River	39.6	88.5	3500	3170

Small vials of the residuals from the pilot-scale 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 the pilot-scale test were sent to the analytical laboratory, Battelle Marine Sciences Laboratory, for analysis. None of the residuals 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 was also utilized. The Engineering/Technology (ET) Work Group was charged with overseeing the development and application of the bench-scale and pilot-scale tests.

Science Applications International Corporation (SAIC) was contracted to provide technical support to the ET Work Group. As part of this effort, SAIC was charged with conducting bench-scale treatability studies on designated sediments to evaluate the removal of specific organic contaminants. The bench-scale study using the ReTeC Thermal Desorption Technology, which is the subject of this report, took place at Star Refinery in Delaware City, DE on September 25, 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 treatability tests for the ARCS Program on four different sediments using four treatment technologies: Thermal Desorption Technology (ReTeC), Anaerobic Thermal Process Technology (SoilTech), Wet Air Oxidation (Zimpro Passavant), and B.E.S.T.[™] Solvent Extraction Process (RCC). This report summarizes the approach used and results obtained during Phase I and Phase II testing of the ReTeC Thermal Desorption Technology. The sediment tested during this evaluation technology was obtained from the Ashtabula River.

The primary objective of this portion of the study was to determine the feasibility and cost-effectiveness of the ReTeC Thermal Desorption Technology for treating and removing PCBs and PAHs from the Ashtabula River sediment. Based upon previous tests performed by ReTeC, it is their experience that the data obtained from the bench-scale testing simulate full-scale operation. Thus, data generated by these tests may be used to estimate treatment costs for full-scale operations and to evaluate process feasibility.

2.2 Sediment Descriptions

The sediments used during the treatability studies conducted by SAIC are typical of sediments within the Great Lakes and their tributaries. The primary contaminants in the Ashtabula River sediment include PCBs.

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. A map is provided in Figure 1 which shows the ARCS Priority Areas of Concern. Specifics of the sample location for the Ashtabula River sediment is shown in Figure 2.

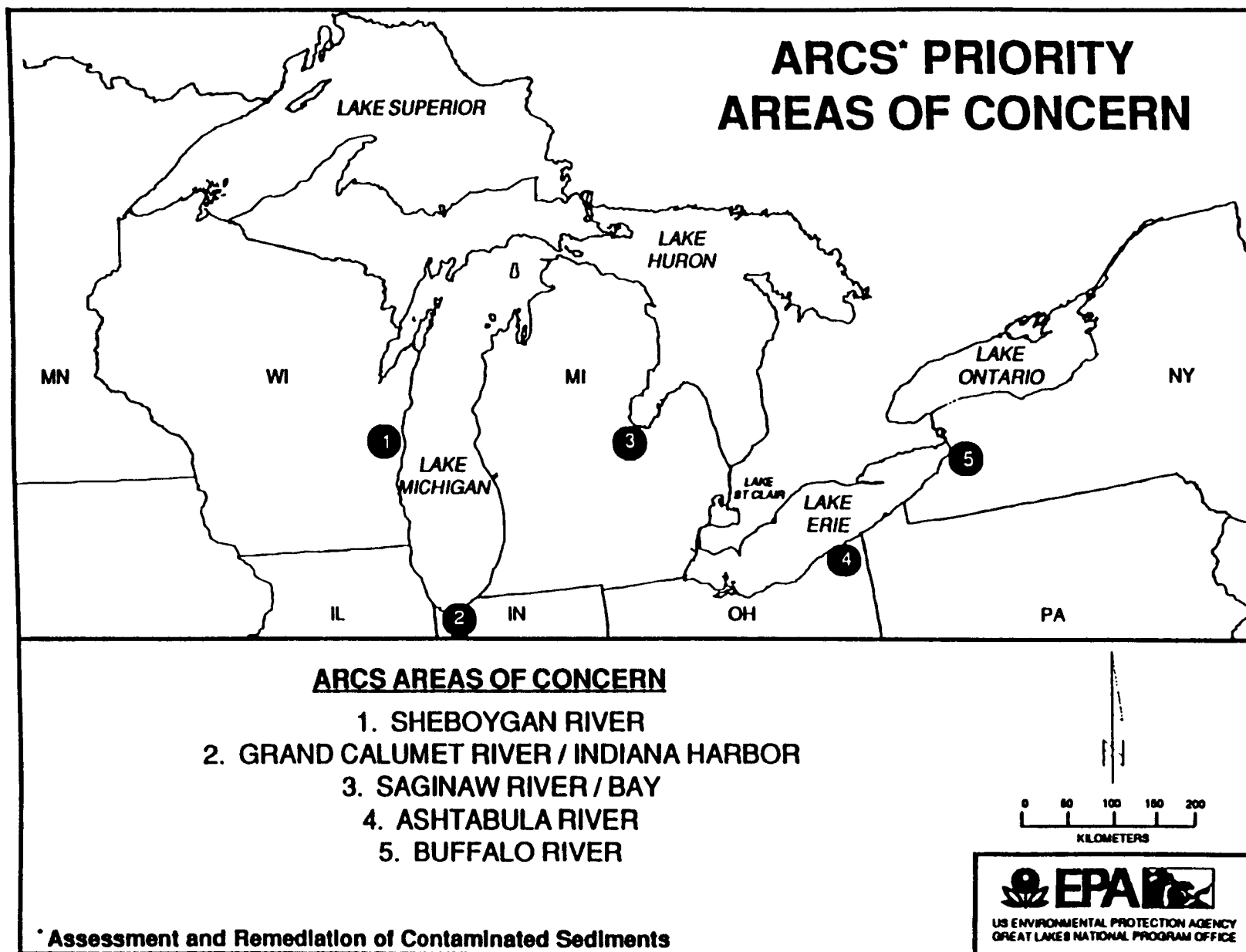


Figure 1. ARCS Priority Areas of Concern.



Ashtabula River

The sediment sample was a composite comprised of subsamples taken throughout the Ashtabula River system

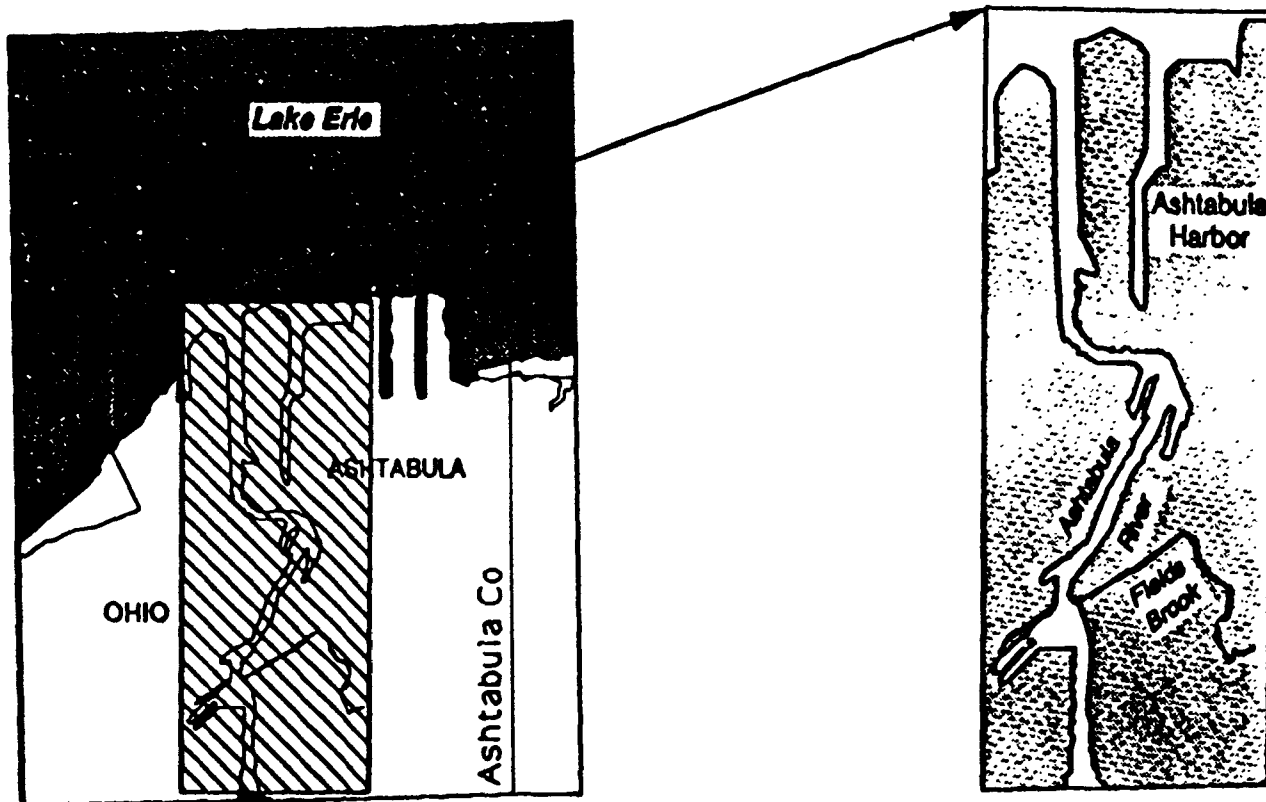


Figure 2. Ashtabula River Sampling Points

2.2.2 Sediment Acquisition and Homogenization

Prior to conducting the treatability study using the ReTeC technology, the sediment was homogenized and stored under refrigeration by the U.S. EPA Environmental Research Laboratory in Duluth, MN.

Samples of the homogenized sediment were sent to SAIC by the Duluth laboratory. Ten gallons of sediment were then transferred by SAIC to ReTeC. ReTeC used these samples to perform a series of standard tests to determine if the samples were compatible with their process and to determine optimum testing conditions and procedures for the treatability study (Phase I). Eleven 5-gallon pails of the sediment were later forwarded to ReTeC by SAIC for Phase II testing.

2.3 **Sediment Characterization**

SAIC was responsible for the physical and chemical characterization of the raw sediment used during the tests. Under SAIC's direction, the sediment and residuals were analyzed by Battelle Marine Sciences Laboratory in Sequim, WA. Table 3 provides characterization data pertaining to the sediment.

**Table 3. Characterization of the Ashtabula River Sediment
(mg/kg dry, unless specified)**

Parameter	Feed
Total PCBs	14.6
Total PAHs	6.1
Moisture, %, as received	35.6
Oil & Grease	1004
TOC, % weight	2.00
Total Volatile Solid, %	7.64
pH, S.U., as received	7.88

2.4 **Technology Description**

ReTeC claims to have developed a thermal desorption technology that is effective in processing solids contaminated with organic constituents. Thermal desorption refers to the separation of contaminants from a solid matrix through volatilization. The desorption process can be used in conjunction with other processes such as incineration or condensation for subsequent control of the

volatilized constituents. According to ReTeC, the technology has potential in low-temperature applications as a pre-treatment step for subsequent biological treatment or in higher-temperature applications as a final treatment option for waste materials. The resultant concentrated waste stream is treated or disposed of, as appropriate.

The primary component of this thermal technology is an indirectly heated thermal desorption/dryer system called the Holo-Flite™ Screw Processor. The Holo-Flite™ Screw Processor is an indirect heat exchanger commonly used to heat, cool, or dry bulk solids/slurries. It consists of a jacketed trough housing a double-screw mechanism. The rotation of the screws promotes the movement of the material forward through the processor. The augers are arranged so that the flights of the two screws mesh, facilitating the movement of material and improving heat transfer. Heated fluid continuously circulates through the hollow flights of the screw augers to elevate the temperature of the solids. This fluid travels the length of the screws and returns to the heater through the center of each shaft. To expand the surface area available for heat transfer, fluid is also circulated through the trough jacket.

Organic material present in the sediment is volatilized and removed from the treatment chamber by means of an induced draft fan to an off-gas control system. The atmosphere in the treatment chamber is controlled during treatment to ensure that oxidation of the volatilized materials does not occur. A three-stage approach is used to control the off-gas from the Holo-Flite™ Screw Processor. Initially, entrained particulate matter is collected using a series of cyclones. The volatilized moisture and organics are then removed using a water-cooled condenser. The remaining non-condensable gas is then passed through a canister containing activated carbon for volatile organic compound (VOC) control. During operation, the composition of the off-gas stream is monitored continuously to ensure the effective operation of the treatment system.

A process flow diagram of the 1000 lb/hr thermal desorption system which was used for Phase II is provided in Figure 3.

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 ReTeC

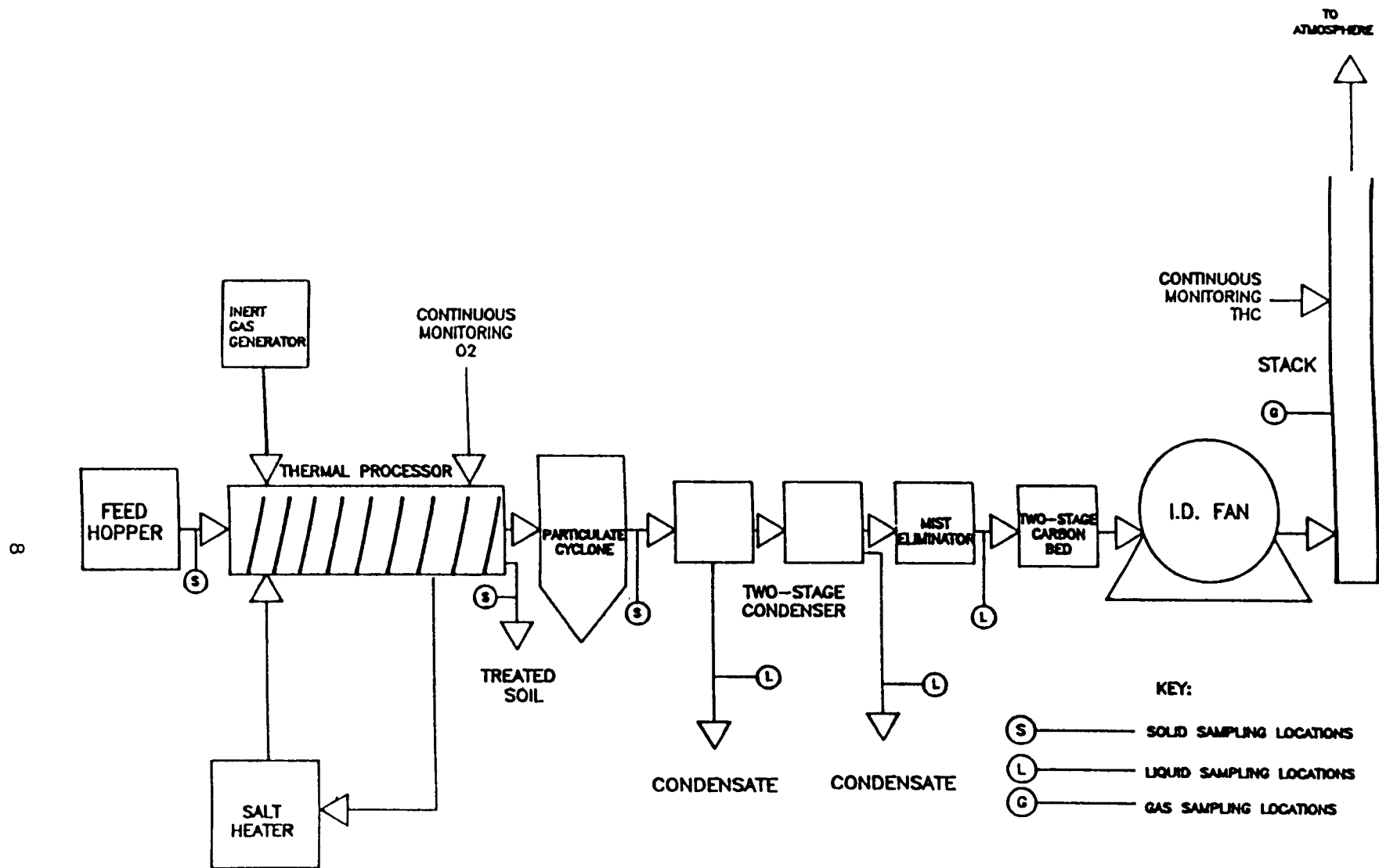


Figure 3. Process Flow Diagram
(Source: ReTeC, Inc.)

Thermal Desorption Technology for treating and removing PCBs and PAHs from the Ashtabula River sediment. The following objectives were critical to the success of the study:

- To record observations and data to predict full-scale performance of the ReTeC Thermal Desorption Technology utilizing their Holo-Flite™ Screw Processor
- To take samples during the desorption tests and conduct analyses sufficient to allow for calculation of mass balances for oil, water, solids, and other compounds of interest
- To calculate the desorption efficiency of target compounds
- To obtain treated solids (300 g dry basis), water, and oil for independent analysis

Based upon previous tests performed by ReTeC, it is their experience that the data obtained from the Phase II test simulate full-scale operation. Ultimately, this data may be used to estimate both the feasibility and treatment costs associated with full-scale application of the technology.

A two-phase approach was used during this study. During Phase I, SAIC sent samples of the raw (untreated) sediments to ReTeC. These samples underwent a series of initial tests in order to determine the optimum conditions to be used during the actual Phase II test. During Phase II, 11 5-gallon pails of untreated, Ashtabula River sediment were sent to ReTeC by SAIC. Untreated sediment and the various end products generated during the Phase II test were obtained and analyzed by SAIC. The data generated by SAIC were used to determine treatment desorption efficiencies. Vendor- or subcontractor- generated data are 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 Science Laboratory was subcontracted to perform all analyses for the different treatability studies performed by SAIC (seven treatability studies utilizing four technologies on four sediments). Assuming that the appropriate volumes of sediment and residuals were available, the same set of parameters listed in Table 4 was analyzed during the characterization of each of the raw sediments and end products from the different treatability tests. In addition, representatives from SAIC observed how all Phase II testing was conducted.

Table 4. Parameters for Analysis of ARCS Program 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

A bench-scale unit located at ReTeC's thermal treatability laboratory in Acton, MA was used during Phase I testing. During Phase I testing, 5-gallon sediment samples were processed using the ReTeC 100 lb/hr system in order to determine waste-specific processing conditions for Phase II. The process operates at temperatures ranging from 500 to 850°F with a solids content of 20 percent or greater required. Thirty-, 60-, and 90-minute residence times were employed during Phase I testing. Process data were collected at 10-minute intervals throughout Phase I. Using this data, ReTeC determined that a heat transfer media temperature of approximately 600°F, with an average solids residence time of 60 minutes, was employed during Phase I. Additionally, the carrier gas flow rate during Phase I averaged 5 scfm, with an average temperature of approximately 1,000°F. See Appendix A for complete data.

The data obtained by analyzing the raw sediments and treated solids for PCBs, PAHs, and moisture were used to determine optimum solids content, processing temperatures, and residence times to be employed during Phase II.

3.2.2 Phase II

Phase II testing is referred to as demonstration-scale testing in ReTeC's Technical Proposal of November 1990. Because of a scheduling conflict, Phase II testing was not performed at ReTeC's

thermal treatability laboratory unit in Acton, MA. In order to prevent additional delay, Phase II was conducted at the Star Refinery in Delaware City, DE. According to ReTeC, this unit had never treated PCB-contaminated waste; however, the processor had recently been used to treat refinery waste.

To prevent possible contamination, the unit was decontaminated prior to testing. Decontamination consisted of steam-cleaning by ReTeC. This procedure was done before the SAIC representative arrived at the site. The unit's feed hopper and screw conveyor were inspected and were found clean by the SAIC representative. A possible source of contamination was the hose for the condenser tank. Visual inspection was not made of this hose.

The Phase II Holo-Flite™ Screw Processor contains two 7-inch intermeshing screw conveyors and has the capacity to treat 1000 to 2000 lbs/hr of material. The system uses a molten eutectic salt as the heat transfer fluid. The salt has heat transfer characteristics similar to those of oils and provides the capability for achieving processing temperatures in excess of 850°F. A series of electric heaters provide 60 kw to heat the salt.

During operation, entrained particulate matter is collected and characterized using a heated cyclone with a "cut" size of 10 µm. The volatilized moisture and organics are subsequently condensed in a two-stage system consisting of a vertically-mounted shell and tube heat exchangers cooled by a closed-loop glycol chiller with a thermal capacity of 30 tons. A mist eliminator is used in-line after the condenser to minimize the carryover of entrained moisture and contaminants. The remaining non-condensable gas stream is passed through an activated charcoal unit to control VOCs prior to release to the atmosphere. A process flow diagram of the demonstration system is provided in Figure 3. Specifications for the system are provided in Table 5.

ReTeC conducted Phase II testing at processing conditions [temperature (975°F), screw rotation rate (0.75 rpm), and residence time (75 min.)] determined following Phase I. Approximately 500 lbs of Ashtabula River sediment were used during the single Phase II run. Because a significant amount of the feed would have been lost by utilizing the bucket elevator (i.e., relative to the total amount of material being processed), the sediment was hand-fed into the unit.

To prevent liquids present in the sediment from passing through the system with less than optimal retention times, decanted sediment solids were initially fed into the unit to produce a "dam" effect in the screw processor. During actual operation, dry sand may be used to create the dam effect in the screw processor. This will keep more liquid feeds from flowing too quickly through the screw conveyor. After the first three and a half pails of solids were added to the unit, operators began introducing water with each scoop of solids fed into the unit. All the water associated with the original sediment was fed through the unit. Approximately 180 lbs of dry treated solids were generated.

Table 5. Full-Scale Holo-Flite™ Screw Processor Specifications

Product Contact Parts:	316 STAINLESS STEEL
Design Pressures:	Screws = 150 PSIG
	Jacket = 30 PSIG
ASME CODE CONSTRUCTION AND STAMPED	
Screw Area:	43 Sq ft
Flight Thickness:	0.25 In
Jacket Area:	17 Sq ft
Trough Volume:	4.5 Cu ft
Screws Fluid Volume:	11 Gal
Jacket Fluid Volume:	15 Gal
Rotary Joint Size:	1.25 In
Design Fluid Flow:	38 Screws-GPM
	9 Jacket-GPM
Fluid Pressure Drop:	60 PSIG
Recommended Operating Pressures:	Screws = 75 PSIG
	Jacket = 30 PSIG MAX

The off-gases from the process were continuously monitored at the stack by ReTeC for total hydrocarbons. The monitoring program was designed to provide emissions data during operation. Although not covered within this report, these data may be used to characterize the airborne emissions from the system. The monitoring system was calibrated prior to and after the completion of the test run using commercially obtained standards. These data were not provided by ReTeC.

During Phase II, process data were collected at 15-minute intervals throughout the test run (see Appendix A). The data included:

- Material feed rate (lb/hr)
- Processor rotational rate (rpm)
- Transfer media temperatures in/out (°F)
- Solids residence time (min.)
- Solids temperature in/out (°F)
- Carrier gas flow rate (scfm)
- Off-gas temperature (°F)
- Mass rates of all process streams (lb/hr).

3.3 Sampling and Analysis

The Quality Assurance Project Plan is provided in Appendix B.

3.3.1 Phase I

During Phase I, the samples of the raw sediments and treated solids were collected for analysis. The procedures used by ReTeC to characterize these samples are listed in Table 6.

Table 6. Analytical Methods Used by ReTeC During Phase I Testing

Parameter	Analytical Method
PCBs (GC/ECD)	EPA 8080
PAHs (GC/MS)	EPA 8270
Moisture	Gravimetric difference after drying at 105°C

Analyses of the sediments were conducted for ReTeC by CEIMIC Laboratories in Narragansett, RI. CEIMIC has been contracted by the EPA for both organic and inorganic analysis in the Superfund program and is a CLP contractor. In addition, CEIMIC has been approved by the Department of Defense's NEESA and HAZWRAP programs.

3.3.2 Phase II

3.3.2.1 *Test Sample Preparation*

The contaminated sample from the Ashtabula River was gray-colored and contained limited debris. The sample contained free-standing water. Since it was very difficult to homogenize the samples with the free-standing water present, the water was decanted prior to conducting the pilot-scale tests and was proportionally recombined with the portion used for the Phase II testing.

3.3.2.2 *Sampling*

At the beginning of the Phase II treatability test, SAIC personnel observing Phase II packed and shipped a sample of the untreated Ashtabula River sediment to SAIC's subcontract laboratory, Battelle, in accordance with written detailed instructions supplied to the SAIC on-site representative. The sample was representative of the material treated by the ReTeC Holo-Flite™ Screw Processor system. This sample was obtained by decanting the standing water from the 11 pails of sediment and compositing an equal volume of the sediment from each of the 11 5-gallon pails with a proportional volume of the decanted water.

Residuals from the ReTeC system consisted of an organic condensate, aqueous condensate, treated solids, and gaseous by-products. After treatment, samples of these residuals were distributed to SAIC. As specified in the Quality Assurance Project Plan (QAPP), a minimum of 300 g (dry basis) of solid material were required in order for Battelle to be able to complete the necessary analyses. Since approximately 177 lbs of solids were produced, this requirement was easily met.

When aqueous condensate was drained from the unit, the initial flow contained 1 to 2 gallons of an oily, black liquid. A sample of the first portion of this oily liquid (sample A-OR-RE-3) was analyzed so that comparisons could be made between this sample and a sample taken after all the water and oil had drained from the unit into a collection drum (sample A-OR-RE). Sample A-OR-RE was obtained from the oil which collected on the surface of the water contained in the collection drum. PAH concentrations within these samples may provide information regarding possible contamination within the unit.

3.3.2.3 Analysis

Analyses were conducted by SAIC's subcontracted laboratory, Battelle, on the raw sediment and the process by-products during Phase II. The number of analyses conducted on these sediments and their residuals are listed in Table 7. Descriptions of the analytical methods employed can be found in Appendix C.

4.0 RESULTS AND DISCUSSION

4.1 Summary of Phase I Results

ReTeC performed a series of initial tests on the raw Ashtabula River sediment to determine specific operating parameters which would optimize the performance of the Holo-Flite™ Screw Processor unit during Phase II testing. Following analyses of the raw sediment and residuals produced during Phase I testing, the following parameters were evaluated relative to their effect on performance: heat transfer media temperature, solids residence time, carrier gas flow rate, and carrier gas temperature. Table 8 briefly summarizes the operating conditions for the Phase I test.

4.2 Phase II Results

As stated previously, the concentration 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. The following sections briefly address the analytical results pertaining to contaminant concentrations in the raw sediment and the process residuals (i.e., treated solids, water, and oil), as well as applicable removal efficiencies. The discussion of Phase II results

**Table 7. ReTeC Analytical Matrix and Sample Identification
Ashtabula River Sediment**

<i>Parameters</i>	<i>QC Sample () and Method Blank</i>	<i>Untreated Sediment</i>	<i>MS</i>	<i>Tripli- cate</i>	<i>Treated Solids</i>	<i>MS</i>	<i>MSD</i>	<i>Tripli- cate</i>	<i>Water</i>	<i>MS</i>	<i>MSD</i>	<i>Tripli- cate</i>	<i>Oil</i>	<i>MS</i>	<i>Tripli- cate</i>
Total Solids (Moisture)	(1) YES	(1) A		(2) A	(1) A			(2) A							
Volatile Solids	(2) YES	(1) A		(2) A	(1) A			(2) A	(1) A			(2) A			
O & G	(2) YES	(1) A		(2) A	(1) A			(2) A	(1) A			(2) A			
Metals	(0) YES	(1) A	(1) A	(2) A	(1) A	(1) A		(2) A	(1) A	(1) A		(2) A			
PCBs	(0) YES	(1) A	(1) A	(2) A	(1) A	(1) A	(1) A		(1) A	(1) A	(1) A		(1) A	(1) A	(2) A
PAHs	(0) YES	(1) A	(1) A	(2) A	(1) A	(1) A	(1) A		(1) A	(1) A	(1) A		(1) A	(1) A	(2) A
TOC	(0) YES	(1) A	(1) A	(2) A	(1) A	(1) A	(1) A		(1) A	(1) A		(2) A			
Total Cyanide	(0) YES	(1) A	(1) A	(2) A	(1) A	(1) A		(2) A	(1) A	(1) A		(2) A			
Total Phosphorous	(0) YES	(1) A	(1) A	(2) A	(1) A	(1) A		(2) A	(1) A	(1) A		(2) A			
pH	(2) YES	(1) A		(2) A	(1) A			(2) A	(1) A			(2) A			
BOD	(1) YES								(1) A			(2) A			
Total Suspended Solids	(1) YES								(1) A			(2) A			
Conductivity	(1) YES								(1) A			(2) A			

(1) = Number of Analyses
A = Ashtabula River Sediment
MS = Matrix Spike
MSD = Matrix Spike Duplicate

Table 8. Optimal Operating Parameters

Operating Parameter	Setting
Heat Transfer Media Temperature (°F)	600
Solids Residence Time (Min.)	60
Carrier Gas Flow Rate (acFm)	9
Carrier Gas Temperature (°F)	960

concludes with an analysis of the mass balance of the media and contaminants. A complete copy of the data generated by Battelle for the Phase II study can be found in Appendix D.

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 the detected values, with one exception: when a compound was detected in the feed sediment sample but not detected in the treated solids, the treated solids total was presented as less than the sum of the detected values plus the detection limit of those undetected compounds that were found in the feed material.

4.2.1 Sediments/Treated Solids

The following sections address the quality of the sediments before and after treatment. Each section focuses upon a different contaminant type and the reductions experienced following treatment.

4.2.1.1 *PCBs*

Samples of the feed material and the treated solids produced using the ReTeC technology were analyzed for PCB contamination. The data from these analyses are presented in Table 9. Total PCBs were identified primarily as Aroclor 1248.

**Table 9. Total PCBs
(mg/kg, dry)**

	Feed	Treated Solids	% Removal
Total PCBs	14.6	<0.6	>96

As demonstrated by these data, a PCB concentration of <0.6 mg/kg was found in the treated solids generated from the Ashtabula River sediment. This corresponds to a PCB removal efficiency of >96 percent.

4.2.1.2 PAHs

Feed material and treated solids were also analyzed for PAHs. As shown in Table 10, a total PAH concentration of <2.4 mg/kg was found in the treated solids. This value corresponds to a removal efficiency of >60 percent. The treated solids contained a relatively large amount of naphthalene, a common constituent of refining wastes, which was not detected in the raw sediments. The presence of naphthalene is most likely the result of trace contamination from previous testing of the ReTeC unit at the refinery, and not a breakdown product from other, higher-molecular weight PAHs that were detected in the feed sediment. Therefore the naphthalene concentration was not included in the calculation of the total PAHs in the treated solids.

Generally, the low removal efficiencies obtained for the individual PAHs in the sediment can be attributed to the low concentration of PAHs initially present in the sediment and the large errors associated with evaluating contaminant concentrations close to analytical detection limits. The higher removal efficiency obtained for the system (i.e., for total PAHs) may be attributed to the method used to quantify the individual PAHs. When making comparisons between individual PAH and total PAH removals it must be realized that since the concentrations of individual PAHs in the feed and treated sediment are very close to analytical detection limits, it is impossible to assess accurately the percent removal achieved by the ReTeC technology.

4.2.1.3 Total Metals

The data in Table 11 highlight the recoveries achieved for the metal contaminants present in the untreated feed and the treated solid. As demonstrated by the percent removal listed in Table 11, with the exception of mercury, there is no indication that the ReTeC technology effectively removes metals. It was assumed that the metals in the solids left in the screw conveyor did not differ in contaminant concentration from those solids that passed through the unit. There is no logical explanation for the increase in metal concentration for copper, nickel, lead, or selenium.

4.2.1.4 Other Analyses

The feed sediment and treated solids were analyzed for percent moisture, oil and grease, TOC, volatile organic solids, and pH as shown in Table 12. The apparent negative percent removal (-13 percent) for TOC is within the range of acceptable precision for this analysis. Four determinations (two

**Table 10. Feed and Treated Solids PAH Concentrations
(mg/kg, dry)**

	Feed	Treated Solids	Percent Removal
Naphthalene	<0.3	0.49	NC ¹
Acenaphthylene	<0.3	<0.3	NC
Acenaphthene	<0.4	<0.5	NC
Fluorene	<0.4	<0.4	NC
Phenanthrene	1.4	<0.3	>78
Anthracene	<0.3	<0.3	NC
Fluoranthene	1.0	<0.3	>70
Pyrene	0.95	<0.3	>68
Benzo(a)anthracene	0.36	<0.3	>16
Chrysene	0.56	<0.3	>46
Benzo(b)fluoranthene	0.45	<0.2	>55
Benzo(k)fluoranthene	0.35	<0.2	>42
Benzo(a)pyrene	0.34	<0.2	>41
Indeno(1,2,3-cd)pyrene	0.34	<0.2	>41
Dibenzo(a,h)anthracene	<0.2	<0.2	NC
Benzo(g,h,i)perylene	0.30	0.10	67
Total PAHs	6.1	<2.4	>60

1 = Naphthalene in treated solids result of cross-contamination; therefore percent removal not calculated.
NC = Not Calculated

moisture and two TOC) are necessary for determining their value. There is no reason to attribute an increase in TOC to the technology; therefore, the best interpretation of the data is that within the limitation of the analytical procedures, there is no change in the TOC context before and after treatment. Moderate removals of 56.6 and 44.4 percent were achieved for oil and grease and total volatile solids, respectively, with these removal rates corresponding to total PAH removals rates but not with total PCB removal rates; therefore the use of either oil and grease or total volatile solids as a surrogate parameter for assessing the performance of the ReTeC process is limited.

Table 11. Metals Concentration in the Feed and Treated Solid (mg/kg, dry)

	Feed	Treated Solids	Percent Removal
Silver	0.19	0.19	0.0
Arsenic	20.8	16.5	20.7
Barium	903	792	12.3
Cadmium	3.06	2.69	12.1
Chromium	591	520	12.0
Copper	33.7	48.1	-42.7
Iron	4.26	3.91	8.2
Mercury	1.361	0.005	99.6
Manganese	559	530	5.2
Nickel	53.0	77.1	-45.5
Lead	58.5	77.0	-31.6
Selenium	0.91	1.53	-68.1
Zinc	234	231	1.3

4.2.2 Oil

The concentration of PAHs and PCBs in the oil separated from the sediment can be found in Tables 13 and 14. Final concentrations in the treated solids and water have been included for comparison. As mentioned previously, two separate samples of the oil were submitted for analysis. Sample A-OR-RE was collected after all the condensates had been drained from the system, while sample A-OR-RE-3 was taken from the initial flow of organics drained from the unit. Comparisons between the data obtained for these two samples clearly indicate that a higher degree of contamination was present in the initial organic flow, supporting the possibility of treatment unit contamination before processing of the ARCS sample began.

4.2.3 Water

The concentration of PAHs and PCBs in the water extracted from the sediment can also be found in Tables 13 and 14. Metal concentrations in the water extract are listed in Table 15, while data characterizing the treated water according to more general parameters are listed in Table 16.

**Table 12. Removal Efficiencies for Other Parameters
(mg/kg, dry, unless specified otherwise)**

Contaminant	Feed	Treated Solids	Percent Removal
Total PCBs	14.6	<0.6	>96
Total PAHs	6.1	<2.4	>60
Moisture, % (as received)	35.6	0.05	
Oil & Grease	1004	436	56.6
TOC, % weight	2.00	2.27	-13.5
Total Volatile Solids, %	7.64	4.25	44.4
pH, S.U. (as received)	7.88	8.09	

4.2.4 Mass Balance

As previously stated, Phase II testing was performed using a 1000 lb/hr Holo-Flite™ Screw Processor. During Phase II, only 353 pounds of treated residuals (177 pounds of treated solids, 145 pounds of aqueous condensates, and 31 pounds of organic condensates), of the 460 pounds of raw sediment introduced to the processor were collected from the system. ReTeC estimates that of the 107 lbs of material lost, 75 lbs were probably caught under the flights of the processor while the remaining 32 pounds of material were most likely undrained condensates. It is apparent that the size of the unit, and the amount of material available for treatment preclude the calculation of a accurate mass balance. Further study involving a significantly larger volume of sediment is needed to evaluate a mass balance for the unit appropriately.

In order to fully address the issue of equipment contamination, rough mass balances were performed for solids, water, oil, and PAHs. Because of the limitations associated with the residual recoveries obtained during testing, these mass balances lack the level of detail found in the mass balances presented in other treatability reports produced by SAIC under the ARCS Program. Furthermore, since PCBs were not found within the residuals and were not suspected of contaminating the thermal processor used during Phase II testing, a mass balance was not performed for this parameter. It is possible the PCBs volatilized and were captured in the activated carbon unit. ReTeC did not analyze the carbon.

Table 13. PAH Concentrations in the Treated Solids, Water, and Oil

Contaminant	Solids (ug/kg)	Residual Water (ug/L)	Residual Oil (ug/kg)	
			Sample A-OR-RE	Sample A-OR-RE-3
Naphthalene	490	609	1070000	2670000
Acenaphthylene	<300	4.16	6860	11600
Acenaphthene	<500	53.0	79500	158000
Fluorene	<400	82.0	118000	21700
Phenanthrene	<300	200	264000	430000
Anthracene	<300	26.7	38600	61100
Fluoranthene	<300	13.0	14900	18200
Pyrene	<300	72.0	86700	115000
Benzo(a)anthracene	<300	18.0	22000	26600
Chrysene	<300	43.5	50000	56100
Benzo(b)fluoranthene	<200	7.91	9190	7890
Benzo(k)fluoranthene	<200	<1	1220	2540
Benzo(a)pyrene	<200	10.2	12700	14600
Indeno(1,2,3-cd)pyrene	<200	1.22	1450	2000
Dibenzo(a,h)anthracene	<200	1.85	2220	2220
Benzo(g,h,i)perylene	100	5.51	7080	8550
Total PAH	<2400	<1150	1780000	3800000

Table 14. PCB Concentrations in the Treated Solids, Water and Oil

Contaminant	Solids (ug/kg)	Residual Water (ug/L)	Residual Oil (ug/kg)	
			Sample A-OR-RE	Sample A-OR-RE-3
Total PCBs	<600	<20	<7000	<7000

Table 15. Metals Concentration in the Residual Water (ug/L)

Metals	Water
Silver	0.002
Arsenic	7.37
Barium	55.6
Cadmium	0.61
Chromium	17.3
Copper	46.5
Iron	1800
Mercury	34.2
Manganese	477
Nickel	147
Lead	43.8
Selenium	<2
Zinc	202

**Table 16. Residual Water Characterization Data
(mg/L, unless specified)**

Contaminant	Water
Total PCBs	<0.020
Total PAHs	<1.15
Moisture	NA
Oil & Grease	564
TOC	446
Total Volatile Solids	81
Total Solids	1600
Total Suspended Solids	1400
pH, S.U., as received	8.20

NA = Not Analyzed

The following sections address the different mass balances and those factors that influence their closures. Tables 17 through 20 contain the data used to calculate the mass balances.

4.2.4.1 Solids

A closure of 39.6 percent was obtained for the solids initially present in the Ashtabula sediment (see Table 17). As stated previously, a large amount of the solids (75 pounds) was most likely caught under the screws of the processor. Given the relatively large nature of these losses, the impact of solids suspended in either the aqueous or organic condensates is minimal and has not been accounted for in the mass balance.

4.2.4.2 Water

The water closure obtained for the Ashtabula River sediment was comparatively good; approximately 88.5 percent (see Table 18). Water adhering to the condensing system did not contribute to the output water recovered. The impact of any residual water present in the treated solids is considered negligible and has not been accounted for in the mass balance.

4.2.4.3 Oil

A closure of 3500 percent was obtained for the oil initially present in the Ashtabula River sediment (see Table 19). This excessively high value does not take into account any oil adhering to the interior of the condensing unit or present in air emissions. The impact of any residual oil present in the treated solids or aqueous condensate was also considered negligible. Apparently residual contamination, possibly from recent tests using the unit to treat refinery wastes, was present within the processor used during the Phase II study.

4.2.4.4 PAHs

An excessively high closure of 3170 percent was realized for the PAHs introduced to the ReTeC system. As shown in Table 20, the vast majority of the PAHs were found in the organic condensate, further substantiating suspicions of equipment contamination. When considering the worst case scenario for raw contaminant concentration (i.e., by adding the detection limits obtained for individual PAHs which were not found above detection limit when determining a value for total PAHs), a closure of 2380 was obtained. Thus, attributions justifying the excessively high closure obtained to an underestimation of raw sediment contamination is precluded. Furthermore, closure does not take into account oil adhering to the interior of the condensing unit or present in air emissions. The impact of any residual oil present in the treated solids was also considered negligible.

Table 17. Solids Mass Balance

Ashtabula River	
<u>Input</u>	
Sediment, lbs	460
H ₂ O, %	35.6
Dry Sediment, lbs (dry)	296.2
Oil & Grease, % dry wt.	0.100
Oil, lbs	0.3
Total Sediment Solids, lbs (dry)	295.9
<u>Output</u>	
Treated Solids, lbs	117
Aqueous Condensate, lbs	145
Total Solids, %	0.16
Solids, lbs	0.2
Total Sediment Solids, lbs (dry)	117.2
<u>Recovery, %</u>	39.6

Table 18. Water Mass Balance

Ashtabula River	
<u>Input</u>	
Sediment, lbs	460
H ₂ O, %	35.6
Total Input Water, lbs	163.8
<u>Output</u>	
Aqueous Condensate, lbs	145
Oil, %	0.0565
Oil, lbs	0.1
Total Input Water, lbs	144.9
<u>Recovery, %</u>	88.5

Table 19. Oil Mass Balance

Ashtabula River	
<u>Input</u>	
Sediment, lbs	460
H ₂ O, %	35.6
Dry Sediment, lbs (dry)	296.2
Oil & Grease, % dry wt.	0.100
Total Oil, lbs	0.3
<u>Output</u>	
Organic Condensate, lbs	31
Oil & Grease, %	34.0
Total Oil, lbs	10.5
<u>Recovery, %</u>	3500

4.3 Summary of Vendor Results

ReTeC did not contract to provide any analyses from this test.

4.4 Quality Assurance/Quality Control

The conclusions and the limitations of data obtained during the evaluations of ReTeC's Thermal Desorption technology are summarized in following paragraphs.

Upon review of all sample data and associated QC results, the data generated for the ReTeC 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.

pH analysis for the sediments was performed using a 1:10 soil-to-water ratio rather than the required 1:1. These data should be used with caution.

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.

Table 20. PAH Mass Balance

Ashtabula River	
<u>Input</u>	
Sediment, lbs	460
H ₂ O, %	35.6
Dry Sediment, lbs (dry)	296.2
Oil & Grease, % dry wt.	0.100
Oil, lbs	0.3
Total Sediment Solids, lbs (dry)	295.9
PAH Conc., % dry wt.	6.05×10^{-4}
Total PAHs, lbs	0.0018
<u>Output</u>	
Organic Condensate, lbs	31
Conc. Total PAHs, %	0.178
PAHs, lbs	0.055
Aqueous Condensate, lbs	145
Conc. Total PAHs, %	1.15×10^{-4}
PAHs, lbs	0.002
Total PAHs Recovered, lbs	0.057
<u>Recovery, %</u>	3170

Large unidentified peaks were observed in the PCB analyses of the untreated sediment, water residual, and oil residual samples. Due to the high concentration of PCBs present in the untreated sediment, the necessary dilutions eliminated any effect on data quality. For the water and oil samples, detection limits had to be increased significantly because of these peaks. While removal efficiencies are not affected, mass balance closures may be difficult.

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

APPENDIX A

FINAL REPORT

**THERMAL TREATABILITY TESTING
ASHTABULA RIVER SEDIMENTS
GREAT LAKES NATIONAL PROGRAM OFFICE**

Prepared for:

SAIC
635 West Seventh Street
Cincinnati, Ohio 45203

Prepared by:

Remediation Technologies, Inc.
9 Pond Lane
Concord, Massachusetts 01742

RETEC Project # 8-0755

Prepared by:

Reviewed by:

September 1992

TABLE OF CONTENTS

NO.	DESCRIPTION	PAGE NO.
1.0	Introduction	1-1
2.0	Technology and Equipment Description	2-1
2.1	Bench-Scale System	2-2
2.2	Pilot-Scale System	2-2
2.2.1	Material Handling	2-4
2.2.2	Thermal Processor	2-4
2.2.3	Media Heater	2-8
2.2.4	Off-Gas Control	2-10
3.0	Scope of Work	3-1
3.1	Phase I Tests	3-1
3.2	Phase II Tests	3-2
3.2.1	Process Monitoring	3-2
3.2.2	Process Stream Sampling	3-7
4.0	Presentation and Discussion of Results	4-1
4.1	Phase I Results	4-1
4.2	Phase II Results	4-2
4.2.1	Feed Material	4-2
4.2.2	Treated Material	4-2
4.2.3	Liquid Condensates	4-6
4.2.4	Mass Balance	4-10
4.2.5	Conclusions	4-10
Appendix A Phase I and II Data Sheets		

LIST OF FIGURES

NO.	DESCRIPTION	PAGE NO.
2-1	Holo-Flite Processor	2-3
2-2	Field-Scale Thermal Processor	2-5
2-3	Process Flow Diagram - Demonstration System	2-6

LIST OF TABLES

NO.	DESCRIPTION	PAGE NO.
2-1	Processor Specifications	2-7
2-2	Solids Cooler Specifications	2-9
3-1	Process Parameters of Interest	3-4
4-1	Phase I Operating Summary	4-1
4-2	Removal Efficiencies for Other Parameters	4-3
4-3	Feed and Treated Sediment PAH Concentrations	4-4
4-4	Metals Concentration in the Feed and Treated Sediment	4-4
4-5	PAH Concentrations in the Treated Sediment, Water and Oil	4-7
4-6	PCB Concentrations in the Treated Sediment, Water and Oil	4-7
4-7	Metals Concentration in the Aqueous Condensate	4-8
4-8	Aqueous Condensate Characterization Data	4-9

1.0 INTRODUCTION

Remediation Technologies, Inc. (RETEC) was contacted to evaluate its thermal desorption system as a treatment technology for contaminated sediments in support of the program being conducted by the Great Lakes National Program Office (GLNPO).

The demonstration program, conducted in two phases, had the following objectives:

- Determine if thermal desorption, using RETEC's system, could effectively treat the contaminated sediments of concern;
- Determine the material processing and handling requirements for the sediments prior to and after the thermal treatment process;
- Determine organic contaminant removal efficiencies for each constituent of interest (PCBs and PAHs); and
- Determine the by-product waste stream characteristics.

Phase I bench-scale testing and Phase II demonstration-scale testing, conducted from August 1 through September 25, 1991, provided specific information related to the thermal desorption of organic species such as polyaromatic hydrocarbons (PAHs) and polychlorinated biphenyls (PCBs) from the contaminated sediments.

Phase I testing involved the use of RETEC's 100 lb/hr bench-scale thermal desorption system. Results of this phase established optimum operating parameters involving treatment temperatures, residence times, and material handling requirements for the next phase of testing using RETEC's 1,000 lb/hr thermal desorption system to determine the effectiveness of treatment at a meaningful scale.

This report provides a description of RETEC's thermal desorption technology capabilities and summarizes the results of key operational data collected during Phase I and Phase II of the program.

2.0 TECHNOLOGY AND EQUIPMENT DESCRIPTION

RETEC has developed a thermal desorption technology that has been used effectively in processing solids contaminated with organic constituents. The technology has potential, in low-temperature applications, as a pretreatment step for subsequent biological treatment, or when used at higher temperatures, as a final disposal option for waste materials.

Thermal desorption refers to the separation of contaminants from a solid matrix through volatilization. The desorption process can be used in conjunction with separate processes, such as incineration or condensation for subsequent control of the volatilized constituents.

The fact that, for some contaminants, efficient removals can be achieved at relatively low treatment temperatures makes thermal desorption a cost-effective approach for the remediation of solids contaminated with hazardous organic constituents.

The desorption process can be accomplished using various types of direct-fired, incineration- or indirect-fired equipment. Applications using indirectly fired methods are preferred in many cases since they generate a significantly smaller volume of off-gas than do traditional incineration systems. As a result, the capital and operating costs for the system can be reduced significantly.

RETEC's application of the technology provides for its use in a condensing mode, i.e., volatilized organics are condensed into a concentrated liquid stream which can subsequently be managed on-site using biological treatment systems, or off-site at a permitted disposal facility.

The benefits of the system include capital costs that are two to three times less expensive than more traditional thermal technologies, and permitting requirements that are significantly less stringent than those for incineration systems.

RETEC's system is based upon the use of an established, indirectly-heated thermal desorption/dryer system, the Holo-Flite® Screw Processor, manufactured by the Denver Equipment Company, Colorado Springs, Colorado.

The Holo-Flite® processor is an indirect heat exchanger commonly used to heat, cool, or dry bulk solids/slurries. The treatment system consists of a jacketed trough which houses a double screw mechanism. The rotation of the screws promotes the forward movement of the

material through the processor. The augers are arranged in the trough so that the flights of the two screws mesh, facilitating the movement of material and improving heat transfer.

The processor uses a contained, non-contact circulating heat transfer fluid to elevate the temperature of the soils. As indicated in Figure 2-1, the heated media continuously circulates through the hollow flights of the screw augers, travels the full length of the screws, and returns through the center of each shaft to the heater.

2.1 BENCH-SCALE SYSTEM

RETEC's bench-scale system uses a Fin-Flite® thermal processor manufactured by the Denver Equipment Company of Colorado Springs, CO. The system is designed to heat the material to temperatures appropriate to volatilize the organic contaminants from the original matrix, leaving a "clean" soil for disposal.

The processor consists of two three-inch diameter hollow augers to convey the material and provide the principal heat transfer surface. The system circulates a synthetic oil as the heat transfer media through the augers on a continuous basis (1.5 gpm) to achieve appropriate solids processing temperatures. The temperature of the media is maintained using a separate 6 Kw heater. The heat transfer media, THERMALANE 600, has a maximum operating temperature of approximately 650°F, resulting in solids temperatures in the range of 550°F.

The system uses a proprietary inert gas handling system to improve the removal efficiency for higher boiling organic species. Off-gases from the processor are collected in a two-stage direct contact condenser/carbon bed assembly.

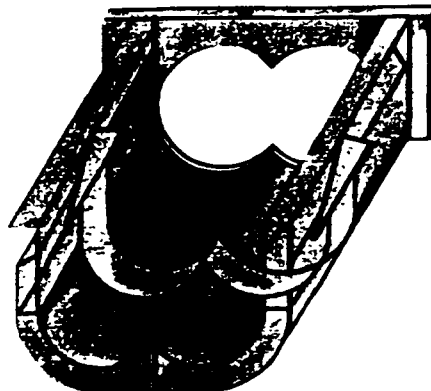
2.2 PILOT-SCALE SYSTEM

RETEC utilized its transportable demonstration system for the performance of the Phase II testing. The system, as contained on a single 8' x 45' flatbed trailer, consists of material feed equipment, thermal processor, indirect condensing system, and an activated carbon unit for the control of volatile organic constituents. The system has been designed to meet Class 1 Division 2 electrical code by means of Type Z Purging of enclosures for electrical equipment and use of

RELTEC

DENVER

Holo-Flite Screw®



Trough Jacket

Color change denotes change in
temperature of heat exchange agent
(Red = Hot Blue = Cool)

Holo-Flite Processor

**FIGURE
2-1**

TEFC motors. RETEC's transportable demonstration system is presented in Figure 2-2. A process flow diagram for the system is provided in Figure 2-3.

The processor uses a contained, non-contact circulating heat transfer fluid to elevate the temperature of the solids. The heated media continuously circulates through the hollow flights of the screw augers, travels the full length of the screws, and returns through the center of each shaft to the heater. RETEC's pilot-scale system employs a unique heat transfer medium, a molten salt eutectic consisting of 53% potassium nitrate, 40% sodium nitrite, and 7% sodium nitrate. The use of this media provides the ability to achieve processing temperatures up to 850°F to affect appropriate removals of heavier organic species and increase the efficiency in treating more complex solid matrices. In addition to the enhanced thermal properties, the salt eutectic provides significant aesthetic benefits; the salt melt is non-combustible; it provides no risk of explosion; and potential vapors are non-toxic.

2.2.1 Material Handling

Generally, material to be processed is placed in a live bottom feed storage hopper (capacity of 1.5 cubic yards). The material is sized and conveyed to a bucket elevator using twin six-inch diameter screws with ribbon flights. The bucket elevator raises the material to a height of 17 feet to a feed conveyor. The feed conveyor then uses a single six-inch ribbon flight auger to convey the material to a double slide gate (air lock) to prevent the leakage of ambient air into the processor.

The sediments dredged for the program were not amenable to the use of this equipment because of the amount of free liquids present. The feed material had a high moisture content and exhibited the adhesive characteristics of fine-grained material. Therefore, sediments were fed manually into the thermal processor using the original five-gallon shipping containers.

2.2.2 Thermal Processor

The Holo-Flite® thermal processor, Model D7-10, contains two, seven-inch intermeshing screw conveyors and has the nominal capacity to treat 0.5 ton per hour of material. Specifications for the system are provided in Table 2-1.

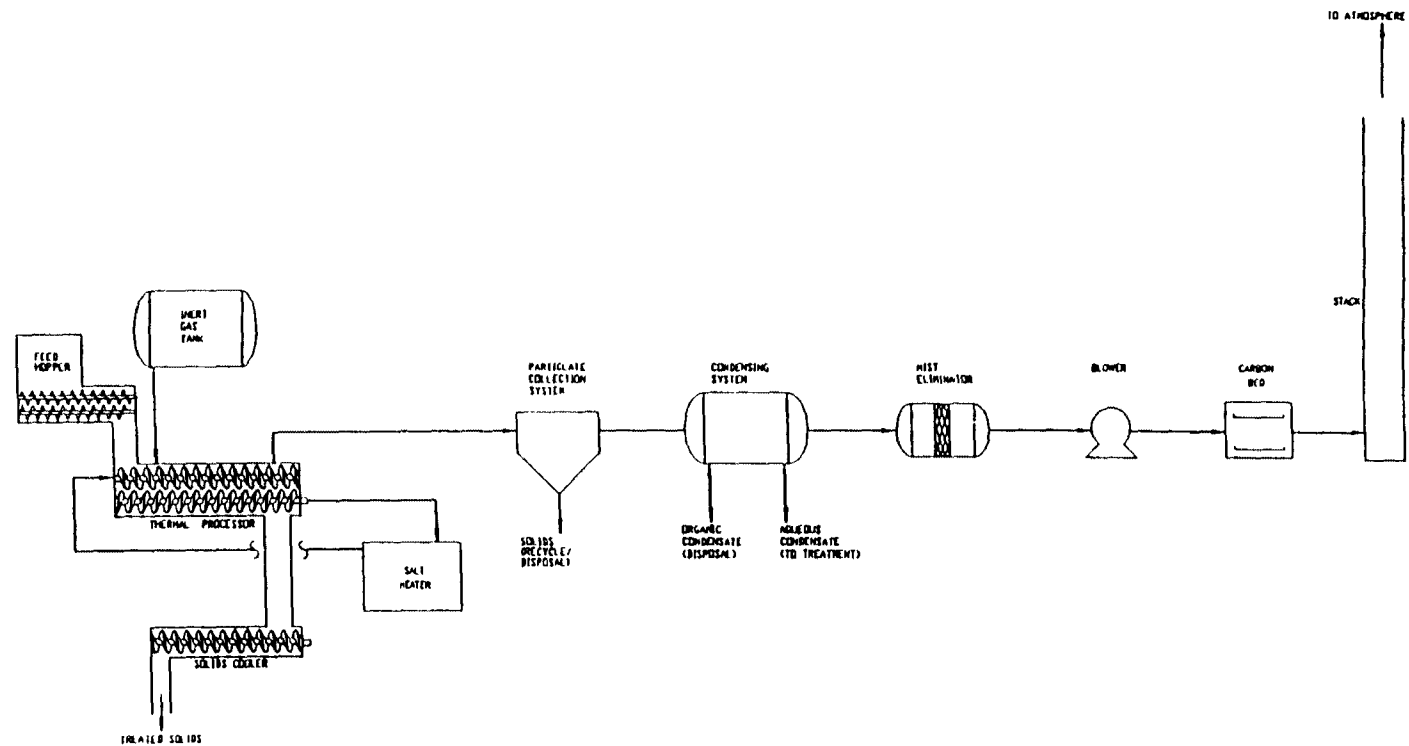
RETEC



FIELD-SCALE THERMAL PROCESSOR

RELEC

42



FLOW DIAGRAM OF THERMAL DESORPTION SYSTEM

FIGURE
2-3

TABLE 2-1

Processor Specifications

Product Contact Parts: 316 STAINLESS STEEL

Design Pressures:

Screws = 150 PSIG

Jacket = 30 PSIG

****ASME CODE CONSTRUCTION AND STAMPED****

Screw Area: 43 Sq. Ft.

Flight Thickness: 1/4 Inches

Jacket Area: 17 Sq. Ft.

Trough Volume: 4.5 Cu. Ft.

Screws Fluid Volume: 11 Gallons

Jacket Fluid Volume: 15 Gallons

Rotary Joint Size: 1 1/2 Inches

Design Fluid Flow: 38 GPM Screws

9 GPM Jacket

Fluid Pressure Drop: 60 PSIG

Recommended Operating Pressures:

Screws = 75 PSIG

Jacket = 30 PSIG MAX.

The system was operated to achieve solids temperatures in the range of 500-675°F. At these temperatures, organic constituents and moisture present in the waste material were volatilized and drawn away under negative pressure to the off-gas control system. The pressure inside the processor was maintained at -0.1 to -0.5 inch of water column (W.C.) to minimize both fugitive emissions and the leakage of ambient air into the processor. Solids residence times in the processor were set at 90 minutes.

The atmosphere in the treatment chamber was controlled during all treatment activities to ensure that oxidation of the volatilized materials did not occur. An "inert" atmosphere was maintained in the treatment chamber through the controlled introduction of nitrogen. RETEC used a tube tank (commercially provided) as the inert gas source. The nitrogen was delivered at a flow rate of 5 to 30 cfm (gas). The oxygen content in the off-gas was monitored continuously during the operation of the treatment system using a Beckman Model 255 oxygen analyzer.

Treated solids were fed by gravity to a second processor designed to cool the solids prior to release to the atmosphere. The "cooling screw" was also of the Holo-Flite design and used a single auger with chilled water as the cooling media. Specifications for this system component are provided in Table 2-2. The cooling screw required approximately 12 gpm of water (<90°F) to cool the solids to a temperature of approximately 140°F. The temperature of the water was maintained using a closed-loop chiller system. The treated solids were discharged from the cooler through a rotary air lock into a 55-gallon storage drum.

2.2.3 Media Heater

The salt eutectic was stored/heated in an enclosed, insulated stainless steel vessel having a capacity of approximately 600 gallons. The tank system was equipped with a continuous containment area. The eutectic was heated electrically using 27 immersion heaters capable of providing 1 MMBTU/hr of heating capacity to the unit and media temperatures of approximately 1,000°F. The media was delivered to the thermal processor by means of a vertical cantilever pump with a submersible head. The pump has the capability to deliver up to 50 gpm of media to the processor.

TABLE 2-2

Solids Cooler Specifications

Product Contact Parts: 316 STAINLESS STEEL
Design Pressures:
Screws = 150 PSIG
Jacket = 30 PSIG

****ASME CODE CONSTRUCTION AND STAMPED****

Screw Area: 22 Sq. Ft.
Flight Thickness: 1/4 Inches
Jacket Area: 12 Sq. Ft.
Trough Volume: 2.6 Cu. Ft.
Screws Fluid Volume: 5.5 Gallons
Jacket Fluid Volume: 7.5 Gallons
Rotary Joint Size: 1 1/2 Inches
Design Fluid Flow: 8 GPM Screws
4 GPM Jacket

Fluid Pressure Drop: 60 PSIG
Recommended Operating Pressures:
Screws = 75 PSIG
Jacket = 30 PSIG MAX.

2.2.4 Off-Gas Control

The off-gas control system was designed to accommodate an off-gas flow rate of ~ 150 scfm and a "worst case" moisture and organic loading of 400 lbs/hr and 150 lbs/hr, respectively. Two particulate cyclones were used to remove any fine solid particles ($> 10 \mu\text{m}$) which may have been entrained with the off-gases. Two indirect-heat exchangers, having a combined surface area of 200 sq ft., were used to reduce the temperature of the gas leaving the processor to approximately 120°F and condense the majority of the entrained moisture/organics. An after-cooler (condenser #3) was placed in-line to remove the remaining moisture and volatile organics from the off-gas stream. The exchanger was designed to achieve an exit gas temperature of 50°F. Cooling water was recirculated in a closed loop through a chiller having a capacity of 240,000 BTU/hr. Condensates were collected in two separate vessels prior to transfer from the system. The system was driven by a variable speed rotary blower capable of developing 300 scfm of flow at a vacuum of 3 inches of Hg.

The thermal system was equipped with an activated carbon system to control non-condensable organics prior to release to the atmosphere. The carbon system was charged with 1,500 lbs of carbon. Volatile organic emissions from the system were monitored in the stack on a continuous basis using the equipment described in Section 3.0 of this document.

3.0 SCOPE OF WORK

RETEC conducted a series of treatability tests to evaluate the effectiveness of its thermal desorption technology in processing contaminated sediments from the Ashtabula River as part of the GLNPO study.

The demonstration tests were designed to characterize the influent and effluent process streams from RETEC's thermal systems under several process conditions to determine the most cost-effective and efficient method of operation. The results from the program were used to validate current estimates of treatment costs.

All treatability activities, including shipment storage and disposal of samples, were conducted in accordance with appropriate regulations. The transportation of all samples complied with applicable shipping requirements including those of the Department of Transportation (DOT) and the U.S. Postal Service.

RETEC provided the capability to test materials with both bench- (Phase I) and pilot-scale (Phase II) treatment systems. In this manner, RETEC obtained meaningful data related to the effectiveness of the technology and the composition of the effluent process streams.

3.1 PHASE I TESTS

Prior to the performance of the demonstration test program, appropriate, waste-specific processing conditions were selected through an initial screening test conducted using RETEC's bench-scale system. The test was conducted on a representative five-gallon sample of material provided by SAIC. Three solids processing conditions (30, 60 and 90 minutes residence time) were evaluated for the sample at the maximum operating temperature of the bench system, approximately 650°F. Multiple residence times were achieved by successive 30-minute passes of the material through the processor. Appropriate process data was collected at 10-minute intervals throughout the tests. The recorded data included:

- material feed rate (lb/hr);
- processor rpms;
- transfer media temperatures in/out (°F);
- solids residence time (min.);

- solids temperatures in/out (°F);
- carrier gas flow rate (scfm);
- carrier gas inlet temperature (°F);
- off-gas temperature (°F); and
- mass rates of all process streams (lb/hr).

Data sheets for the Phase I testing are provided in Appendix A.

Samples of the feed and treated solids for each residence time were collected for analysis as discussed in Section 4.1. The results from the screening tests were appropriate to define the system configuration, processing temperature, and approximate residence time for subsequent testing for the Phase II program.

3.2 PHASE II TESTS

Pilot-scale testing was conducted using approximately 500 lbs. (one 55-gallon drum) of material. RETEC conducted this test at processing conditions defined during the Phase I testing program. Process data was collected at 15-minute intervals throughout the test run. Treated solids temperatures averaged 570°F, for a residence times of 90 minutes.

3.2.1 Process Monitoring

RETEC monitored all pertinent process parameters at routine intervals during the program. Such an approach was imperative to develop appropriate data for the subsequent design of installed equipment. The recorded data included:

- material feed rate (lb/hr);
- processor rpms;
- transfer media temperatures in/out (°F);
- solids residence time (min.);
- solids temperatures in/out (°F);
- carrier gas flow rate (scfm);
- carrier gas inlet temperature (°F);
- off-gas temperature (°F); and
- mass rates of all process streams (lb/hr).

A complete list of the parameters monitored is provided in Table 3-1. Off-gas concentrations of oxygen and hydrocarbons were recorded continuously with a strip chart recorder. Discussions of the principal parameters monitored during Phase II testing are provided below.

Temperature

Process temperatures were monitored at 21 locations using Type-K thermocouples manufactured by Omega Engineering, inc. Temperature signals were transmitted to a panel-mounted Model 115 readout, using wire insulated to withstand temperatures up to 1,000°F.

Pressure

Atmospheric pressures were monitored at seven locations within the processing system using magnahelic gauges manufactured by the Dwyer Co. Pressures were monitored within the headspace of the processor and across all of the principal components of the off-gas system to ensure proper operation of the system and to help anticipate maintenance problems, such as poor heat transfer due to particulate "fouling."

Gas Flow Rates

The off-gas flow rate from the thermal system was monitored within the stack gas using a hot-wire anemometer. The hot-wire measured the off-gas velocity, in feet per minute. The actual flow rate was calculated by using the area of the stack, multiplied by the velocity to give a flow rate of cubic feet per minute. The flow rate of inert gas into the processor dome was monitored by use of a standard flow meter manufactured by the Dwyer Company.

Solids Feed Rate

The solids feed rate (lb/hr) to the processor was monitored by recording the known volume of sample material entering the unit. The untreated sediment was distributed into five-gallon pails to ease loading of the system. The feeding was performed on a batch basis by emptying a five-gallon pail into the feed system every ten minutes.

TABLE 3-1

Process Parameters of Interest

Process Temperatures (°F)

Thermal Processor

Waste Feed_(in)
Treated Solids _(out)
Transfer Media Tank
Transfer Media _(in)
Transfer Media _(out)
Inert Gas_(in)

Solids Cooler

Treated Solids _(out)
Transfer Media_(in)
Transfer Media_(out)

Off-Gas Treatment

Gas from Processor
Gas Exiting Cyclone
Gas Exiting Condenser
Condenser Cooling Media_(in)
Condenser Cooling Media_(out)
Fin Fan Cooler Setpoint
Gas Entering After-cooler
Gas Exiting After-cooler
After-cooler Media_(in)
After-cooler Media_(out)
Chiller Setpoint

TABLE 3-1
Process Parameters of Interest (Continued)

Off-Gas Composition

Oxygen (exit from particulate cyclone)
Total Hydrocarbons (stack)

Process Pressures (in. W.C.)

Inert Gas Delivery
Processor Headspace
Solids Cooler Headspace
Exit from Particulate Cyclone
Exit from Condenser
Inlet to After-cooler
Inlet to Carbon Bed

Gas Flow Rates (acfm)

Inert Gas
Exit from Particulate Cyclone
Discharge Stack

Liquid Flow Rates (gpm)

Condenser Cooling Media
After-cooler media
Solids Cooler Transfer Media
Aqueous Condensate
Organic Condensate

Solids Processing Rate (lb/hr)

Waste Feed
- feed auger rpms
- processor rpms
Treated Solids
- solids cooler rpms

Continuous Emissions Monitoring

The continuous emissions monitoring (CEM) system, a self-contained, nitrogen-purged cabinet on the thermal system, was designed to meet Class 1, Division 2 electrical codes. Samples were collected from two principal locations: the exit point from the particulate cyclone (oxygen); and the discharge stack (total hydrocarbons). Heated sample lines carried the gas samples to the cabinet through sample conditioning systems prior to analysis. The sample stream conditioning systems consisted of:

- refrigerated condensers with an automatic drainage system;
- coalescing high-efficiency filters to remove oil mists, particulates, and acid vapors; and
- membrane dryers for selective drying of the gas sample based on permeation distillation.

Oxygen Analyzer

The oxygen monitoring system used a Beckman Model 755 oxygen analyzer to provide continuous data related to the oxygen content of the off-gas stream. The analyzer makes measurements based upon the determination of magnetic susceptibility of the sample gas, oxygen being very paramagnetic, and other gases being weakly diamagnetic. The instrument provided direct readout of oxygen concentration on a front panel meter.

The meter has a range of 0% to 100% oxygen concentration, a reproducibility of $\pm 0.01\%$, and a zero drift of $\pm 1\%$ of full scale per 24 hours. Maximum sample temperature and pressure are 150°F and 10 psig, respectively. The sample flow rate is 250 cubic centimeters per hour.

Total Hydrocarbon Analyzer

Total hydrocarbons were monitored in the exhaust gas from the system using a Beckman Model 400A hydrocarbon analyzer. The analyzer continuously measured the concentration of hydrocarbons in the gas stream using a flame ionization detector (FID).

Electronic stability at maximum sensitivity is $\pm 1\%$ of full scale through a sample temperature of 30 to 110°F. Sensitivity is adjustable from 1 ppm to 2% calibrated to methane.

Recorded process data sheets for the Phase II tests are presented in Appendix A of this document.

3.2.2 Process Stream Sampling

The field demonstration program was designed to incorporate a comprehensive sampling and analytical program to characterize all of the influent and effluent process streams associated with the system. Five sample streams are associated with the demonstration test equipment:

- Waste Feed;
- Treated Soil;
- Aqueous Condensate;
- Organic Condensate; and
- Process Off-Gas.

Composite samples of each of the solid and liquid streams were collected as a part of the test program as detailed below. Appropriate aliquots of each sample were collected into precleaned containers by SAIC and submitted for subsequent analysis.

Solid Samples

As-Received Material

RETEC pretreated the feed material by pouring off free standing liquid and stored it in lined, covered containers before treatment. Samples of the material were obtained during the preparation step using a grab sampling technique, S000 (scoop). Approximately ten grab samples were collected from the drum of material into precleaned containers and submitted to SAIC personnel for analysis.

Treated Material

Samples of the final treated material were obtained from the exit point of the solids cooler at 15-minute intervals throughout each test and composited to form a single sample for each test condition. The entire sample from the test run was collected into a lined 55-gallon drum.

Liquid Samples

The aqueous and organic condensates from the test were also collected into lined 55-gallon drums. Samples of the condensate streams were collected from a tap located in the line leading to the sample drums. The samples were collected at 15-minute intervals and composited into samples for analysis.

At the conclusion of the test, the collected process streams were weighed for subsequent mass balance determinations.

The off-gases from the process stream were continuously monitored at the stack location for total hydrocarbons. The monitoring program was designed to provide emissions data during the operation of the thermal desorption system, and to generate information that might further characterize the airborne emissions from the system. The monitoring system was calibrated prior to and at the completion of each of the test runs using commercially obtained standards.

4.0 PRESENTATION AND DISCUSSION OF RESULTS

RETEC performed bench- (Phase I) and pilot- (Phase II) scale testing of its thermal desorption technology on contaminated sediments from the Ashtabula River. Results of the program are presented in detail in the following sections.

4.1 PHASE I RESULTS

RETEC performed a series of bench-scale tests on the raw Ashtabula River sediment to determine specific operating parameters which would optimize the performance of the thermal desorption technology during pilot-scale testing (Phase II). Process parameters during Phase I testing were analyzed and evaluated relative to their effect on treatment performance. Table 4-1 briefly summarizes the operating conditions for the bench-scale test.

TABLE 4-1

Phase I Operating Summary

OPERATING PARAMETERS	
Heat transfer media temperature (°F)	600
Solids residence time (min.)	60
Carrier gas flow rate (acfm)	5
Carrier gas temperature (°F)	1,000

Waste feed for the bench-scale tests had PCB concentrations of 11.6 mg/kg and a moisture content of 48%. The analysis of residuals associated with a residence time of 60 minutes indicated that PCB concentrations had been reduced to < 0.5 mg/kg. The moisture content of the material had been reduced to less than 1%. Concentrations of PAHs in the waste feed were not detected. Data sheets for the Phase I tests are included in Appendix A.

4.2 PHASE II RESULTS

RETEC's pilot-scale demonstration system was used to evaluate further the effectiveness of thermal desorption on the contaminated river sediments. The test provided the opportunity to process sediments with a treatment technology which resembles a design and operating condition typical of larger full-scale treatment systems.

The pilot test was performed on September 25, 1991. Residence time for the pilot-scale test was set at 90 minutes in order to ensure appropriate removal rates. The following sections address the quality of the sediments before and after treatment.

4.2.1 Feed Material

Approximately 500 lbs. of Ashtabula River sediments was received. A composite sample was collected and analyzed for PCBs, PAHs, metals, oil and grease, total organic carbon, total volatile solids, moisture content and pH. Data from these analyses are presented in Tables 4-2 through 4-4. Percent moisture of the feed material was 35.6%. Total PCB and PAH concentrations were 14.6 and 6.05 mg/kg, respectively. Oil and grease content was measured at 1,000 mg/kg dry weight.

4.2.2 Treated Material

A single composite sample of the treated material was analyzed for the same parameters as the initial feed material. Results of the analyses calculated removal efficiencies summarized in Tables 4-2 through 4-4. PCBs detected in the initial feed of 14.6 mg/kg were reduced to less than 0.6 mg/kg. This corresponds to a removal efficiency of >96%.

As shown in Table 4-3, a total PAH concentration of 0.58 mg/kg was found in the treated sediment. This value corresponds to a removal efficiency of 90.4 percent. The principal contaminant in the treated solids, naphthalene, was not detected in the as-received sediments, but is a common constituent of petroleum refining wastes. Therefore, the presence of naphthalene may be the result of trace contamination from previous testing at a petroleum refinery.

Generally, the low removal efficiencies obtained for the individual PAHs in the sediment can be attributed to the low concentration of PAHs initially present in the sediment and the large errors associated with evaluating contaminant concentrations close to analytical detection limits. The high removal efficiency obtained for the system (i.e., for total PAHs) may be attributed to the method used to quantify the individual PAHs. When making comparisons between individual PAH and total PAH removals, it must be realized that, since the concentrations of individual PAHs in the feed and treated sediments are very close to analytical detection limits, it is impossible to accurately assess the removal efficiencies effectively.

The data in Table 4-4 highlight the recoveries achieved for the metal contaminants present in the untreated feed and the treated sediment. As demonstrated by the percent removals listed in Table 4-4, with the exception of mercury, there is no indication that this technology is effective at removing metals.

The feed sediment and treated solids were analyzed for percent moisture, oil and grease, TOC, volatile organic solids, and pH as shown in Table 4-2. Although moderate removals of 56.6% (oil and grease) and 44.4% (volatilize organic solids) were achieved, these removals do not closely correspond to total PAH or PCB removal efficiencies and is believed to be from the contamination of residuals from past treatment demonstrations at petroleum refineries. Percent moisture in the sediments was removed at a rate of 97.2%. The TOC concentration increased from 2.00 to 2.27% because of the weight reduction of the treated material versus the feed.

TABLE 4-2
Removal Efficiencies for Other Parameters
(mg/kg, dry, unless specified differently)

CONTAMINANT	FEED	TREATED SEDIMENT	% REMOVAL
Total PCBs*	14.8	<0.6	>98
Total PAHs	6.05	0.58	90.4
Moisture, % (as received)	35.6	<1	97.2
Oil & Grease	1004	436	56.6
TOC, % weight	2.00	2.27	-13.5
Total Volatile Solids, %	7.64	4.25	44.4
pH, S.U. (as received)	7.88	8.09	

* Identified primarily as Aroclor 1248

TABLE 4-3**Feed and Treated Sediment PAH Concentrations
(mg/kg, dry)**

	FEED	TREATED SEDIMENT	% REMOVAL
Naphthalene	<0.3	0.485*	NC
Acenaphthylene	<0.3	<0.3	NC
Acenaphthene	<0.4	<0.5	NC
Fluorene	<0.4	<0.4	NC
Phenanthrene	1.378	<0.3	>78.1
Anthracene	<0.3	<0.3	NC
Fluoranthene	1.037	<0.3	>70.9
Pyrene	0.949	<0.3	>68.3
Benzo(a)anthracene	0.358	<0.3	>16.1
Chrysene	0.559	<0.3	>46.3
Benzo(b)fluoranthene	0.448	<0.2	>55.1
Benzo(k)fluoranthene	0.348	<0.2	>42.3
Benzo(a)pyrene	0.337	<0.2	>40.3
Indeno(1,2,3-cd)pyrene	0.335	<0.2	>39.7
Dibenzo(a,h)anthracene	<0.2	<0.2	NC
Benzo(g,h,i)perylene	0.303	0.097	68.7
Total PAHs	6.05	0.58	90.4

NC = Not Calculated.

* Potentially the result of residual contamination

TABLE 4-4

**Metals Concentration in the Feed and Treated Sediment
(mg/kg, dry)**

	FEED	TREATED SEDIMENT	% REMOVAL
Silver	0.19	0.19	0.0
Arsenic	20.8	16.6	20.7
Barium	903	792	12.3
Cadmium	3.06	2.69	12.1
Chromium	591	520	-12.0
Copper	33.7	48.1	-42.7
Iron	4.26	3.91	8.2
Mercury	1.361	0.005	99.7
Manganese	559	530	5.2
Nickel	53.0	77.1	-45.5
Lead	58.6	77.0	-31.6
Selenium	0.91	1.53	-68.1
Zinc	234	231	1.3

4.2.3 Liquid Condensates

Liquid condensates were collected from the off-gas condensing system during the treatment process. A two-phase condensate of water and oil was drained from the system.

The concentration of organics in the soil separated from the sediment can be found in Tables 4-5 and 4-6. Final concentrations in the treated sediment and water have been included for comparison. There are two columns for constituent levels in the oil phase. Oil₁ was a sample collected of the first oil condensate from the off-gas condensing system. This first "sludge" was very viscous and dark in color. The second oil, Oil₂, was a much lighter color oil which exited the condensing system after Oil₁. The analyzed results indicate that Oil₁ has a much higher contaminant concentration than Oil₂. Upon further investigation, Oil₁ is reminiscent of condensates collected during the thermal treatment of oily petroleum refinery wastes. It is believed that constituent concentrations in Oil₁ condensate are residues from past treatment demonstrations at petroleum refineries and, in fact, was the last program conducted before the Ashtabula River program.

Oil₂ is more indicative of a lighter condensate from a material that contains low oil and grease and high moisture content, such as the sediments from the Ashtabula River.

Water

The concentration of PAHs and PCBs in the water extracted from the sediment can also be found in Tables 4-5 and 4-6. Metal concentrations in the water extract may be found in Table 4-7, while data characterizing the treated water according to more general parameters can be found in Table 4-8.

TABLE 4-5**PAH Concentrations in the Treated Sediment, Water, and Oil**

CONTAMINANT	SEDIMENT (ug/kg)	WATER (ug/L)	OIL₂ (ug/kg)	OIL₁ (ug/kg)
Naphthalene	485	609	1,070,000	2,670,000
Acenaphthylene	< 300	4.16	6,860	11,600
Acenaphthene	< 500	53.0	79,500	158,000
Fluorene	< 400	82.0	118,000	21,700
Phenanthrene	< 300	200	264,000	430,000
Anthracene	< 300	26.7	38,600	61,100
Fluoranthene	< 300	13.0	14,900	18,200
Pyrene	< 300	72.0	86,700	115,000
Benzo(a)anthracene	< 300	18.0	22,000	26,600
Chrysene	< 300	43.5	50,000	56,100
Benzo(b)fluoranthene	< 200	7.92	9,190	7,890
Benzo(k)fluoranthene	< 200	< 1	1,220	2,540
Benzo(a)pyrene	< 200	10.2	12,700	14,600
Indeno(1,2,3-cd)pyrene	< 200	1.22	1,450	2,000
Dibenzo(a,h)anthracene	< 200	1.85	2,220	2,220
Benzo(g,h,i)perylene	97	5.51	7,080	8,500
Total PAH	582	1150	1,780,000	3,800,000

TABLE 4-6**PCB Concentrations in the Treated Sediment, Water and Oil**

CONTAMINANT	SEDIMENT (ug/kg)	WATER (ug/L)	OIL₂ (ug/kg)	OIL₁ (ug/kg)
Total PCBs	< 600	< 20	< 7,000	< 7,000

TABLE 4-7

Metals Concentration in the Aqueous Condensate (ug/L)

	WATER
Silver	0.002
Arsenic	7.37
Barium	55.6
Cadmium	0.61
Chromium	17.3
Copper	46.5
Iron	1,800
Mercury	34.2
Manganese	477
Nickel	147
Lead	43.8
Selenium	< 2
Zinc	202

TABLE 4-8

**Aqueous Condensate Characterization Data
(mg/L, unless specified differently)**

CONTAMINANT	WATER
Total PCBs	<0.020
Total PAHs	1.15
Moisture	NA
Oil & Grease	564
TOC	446
Total Volatile Solids	81
Total Solids	1,600
Total Suspended Solids	1,400
pH, S.U. as received	8.20

4.2.4 Mass Balance

As previously stated, Phase II testing was performed using a full-scale, 1,000 lb/hr Holo-Flite® thermal processor. During Phase II, only 353 pounds of treated residuals (177 pounds of treated solids, 145 pounds of aqueous condensates, and 31 pounds of organic condensates), out of the 460 pounds of raw sediment introduced to the processor, were collected from the system. This calculates into a mass balance of 76.7%. RETEC estimates, of the 107 lbs of material not accounted for, 75 lbs. of solids were retained in the void spaces of the processor, and approximately 32 pounds (four gallons) of water were most likely retained in the condensate receiving vessels. It is apparent that the size of the unit, as compared to the amount of material available for treatment, precludes the calculation of a meaningful mass balance. Further study, involving a significantly larger volume of sediment, is needed in order to appropriately evaluate a mass balance for the unit.

4.2.5 Conclusions

A review of the results provide for the following conclusions related to material composition, material handling, effectiveness of treatment and process conditions.

Material Composition

The as-received material had an average moisture content of 35.6%. Solids were primarily silts and clays and were very cohesive. The material contained relatively low levels of organic contamination. The oil and grease concentration was 0.1%. PCBs and total PAHs were present at 14.8 and 6.05 mg/kg, respectively.

Material Handling

Due to the high moisture content, the sediments could not be fed through the existing material handling system. Feeding was performed on a batch basis by emptying a five-gallon pail of sediment into the feed chute every ten minutes. The sediment was generally free of debris or oversized material which would adversely affect the processing of the sediment during full-scale operations. RETEC recommends investigating alternative methods to feed the processor, such as a positive displacement pump designed to handle high solids content.

Performance of the Technology

RETEC's process was successful in treating the Ashtabula River sediments for both waste minimization and organic contaminant removal.

The most dramatic effect of treatment is the mass/volume reduction of the treated solids. The mass reduction was 23.2%, primarily due to the removal of moisture. Moisture was reduced by 97% in the treated solids, demonstrating that the technology can provide an effective means of dewatering sediment.

Total PCB and PAH concentrations in the feed material were removed by greater than 98 and 90%, respectively. Oil and grease, as well as total volatile solids concentrations were also reduced. It is believed that residual contamination from previous testing at petroleum refineries were the cause of the lower removal rates.

Due to the limited scale of this test program (500 lbs. of feed) and the size of RETEC's demonstration unit (1,000 lb/hr), it is difficult to define processing parameters with much detail. Further, with the low concentration of contaminants in the feed, a single batch-type test is not representative of the technology's full capabilities. RETEC recommends the performance of a larger scale test program designed to evaluate processing data under steady state operating conditions and varying treatment parameters.

APPENDIX A

Phase I and II Data Sheets

ReTeCS MOBILE SALT SYSTEM

SALC, Atchafalaya River Sediments

PROJECT #: H80-755-400

DATE:	9/25
SHIFT START TIME:	
SHIFT END TIME:	

AIR MONITORING													AVG.
O2 %	10	10.4	8.6	10.8	11.8	10.2	12	11.3	12.3	11.4	11	11.4	10.93
THC PPM	219	214	200	198	180	172	183	204	293	470	317	293	245.2

MOISTURE CONTENT % TOTAL		
WASTE FEED		
TREATED SOIL		

LEVEL OF PRODUCT		
OIL		
WATER		

SALT DRIP RATE													
PROCESSOR dpm	60	60	60	50	45	45	50	40	40	30	20	20	43.33

SAMPLE COLLECTION				
TIME OF SAMPLING				
TYPE OF SAMPLE				
# OF SAMPLES				
AMOUNT OF EACH SAMPLE				
DESTINATION				

ReTeC'S MOBILE SALT SYSTEM
 SAIC, Atitula River Sediments
 PROJECT # H80-755-400

DATE: 9/25
 SHIFT START TIME:
 SHIFT END TIME:

blower											
off											
AVG.											
OFF GAS FLOW RATES											
STACK GAS VELOCITY (afpm)	650.0	550.0	850.0	750.0	850.0	750.0	900.0	850.0	900.0	650.0	600.0
STACK GAS FLOW RATE (afcm)											

SYSTEM PRESSURES	AVG.												
PROCESSOR HEAD SPACE (IN H ₂ O)	-0.4	-0.3	-0.1	-0.4	-0.4	-0.1	-0.4	-0.5	-0.5	-0.5	-0.5	0	-0.34
COOLING HEAD SPACE (IN H ₂ O)	0	0	0	0	0	0	0	0	0	0	0	0	0
PRE - CYCLONE #1	0.5	0.5	0.5	0.5	1.5	1.7	2.5	2	0	0.5	0.5	0	0.891
PRE - CYCLONE #2	0	0	0	0.5	1	1	1.5	1	1	0	0	0	0.5
POST - CYCLONE #1													
POST - CYCLONE #2													
PRE - HX1													
POST - HX1 Pres drop	0	0	0	0	0	0	0	0	0	0	0	0	0
PRE - HX2													
POST - HX2 Pres drop	0	0	0	0	0	0	1	1	0	0	0	0	0.166
PRE - HX3 Pres drop	0	0	0	0	0	0	0	0	0	0	0	0	0
POST - HX3													
BL O W E R (in Hg)	0	0	0	0	1	1	2	1.5	0	0	0	0	0.458
STACK (in H ₂ O)													
BL O W E R S P H Z	6	6	6.5	8.2	10	10	12.4	11.9	10.4	7.7	7.7	0	8.066

DATE:	9/25
SHIFT START TIME:	
SHIFT END TIME:	

[illegible][illegible]

ReTeC'S MOBILE SALT SYSTEMSAIC, Atitula River SedimentsPROJECT #: 1180-755-400

DATE:	9/25
START FEED	
FINISH FEED	

GAS TEMPERATURES												AVG.	
N2 TEMP OUT OF HEATERS	1026	1026	1028	1031	1029	1030	1027	1030	1031	1033	1032	989	1026
PRE - CYCLONE TEMP #1	699	729	716	714	606	467	340	326	474	580	632	551	569.5
PRE - CYCLONE TEMP #2	616	622	665	632	629	624	556	522	527	540	545	512	582.5
POST - CYCLONE #1	468	507	520	520	481	385	288	271	346	410	442	397	419.5
POST - CYCLONE #2	435	455	505	518	542	544	509	468	452	432	421	375	471.3
INLET HX1 GAS TEMP	468	507	520	520	481	385	288	271	346	410	442	397	419.5
OUTLET HX1 GAS TEMP	65	66	64	66	70	67	73	71	71	69	67	65	67.83
INLET HX2 GAS TEMP	435	455	505	518	542	544	509	468	452	432	421	375	471.3
OUTLET HX2 GAS TEMP	65	64	64	65	69	69	74	72	64	64	63	64	66.41
INLET HX3 GAS TEMP	75	75	75	73	73	73	74	74	73	74	74	76	74.08
OUTLET HX3 GAS TEMP	63	62	62	62	63	61	64	62	60	62	61	64	62.16
STACK GAS TEMP	76	77	77	78	81	80	80	79	79	78	78	78	78.41

COOLING WATER TEMPS												AVG.	
CONDENSER HX3 IN	60	60	59	60	63	61	63	62	60	61	61	62	61
CONDENSER HX3 OUT/HX1 IN	60	61	60	61	63	61	64	62	61	62	61	62	61.5
CONDENSER HX1 OUT/HX2 IN	61	61	60	62	65	62	65	63	60	62	60	62	61.91
CONDENSER HX2 OUT/COOLING SCW IN	60	60	60	61	64	64	65	63	60	61	62	61	61.75
COOLING SCREW OUT													
CHILLER SET POINT													

ReTeC'S MOBILE SALT SYSTEM
DATA SHEET FOR SAIC, ASTIBUOLA RIVER SEDIMENTS
Project #: H80-755-400

DATE:	9/25
START FEED	
FINISH FEED	

CREW		TREATMENT GOAL	
		EST. SALT TEMP	975
		EST. SOIL TEMP	700

PROCESSOR DATA:

WASTE FEED CONVEYOR SPEED	NA
PROCESSOR RPM	0 9
RESIDENCE TIME	90 min
COOLING SCREW RPM	
RESIDENCE TIME	ERR

Start Feed @ 11.13 am
 Flush Feed @ 12.40 pm

SYSTEM TEMPERATURES

SOLIDS TEMPERATURES												AVG.	
TIME	1105	1120	1135	1150	1205	1220	1235	1250	1315	1330	1345	1400	1234
TEMP IN													
TEMP OUT RIGHT	676	570	567	534	528	532	560	546	542	589	599	675	576.5
TEMP OUT LEFT	614	529	511	565	542	561	573	572	564	613	632	668	578.6
COOLING SCREW OUT	69	69	69	70	70	75	84	80	132	147	154	163	98.5

SALT TEMPERATURES													AVG.
BATH TEMP - SALT TANK	977	976	974	975	970	968	970	969	972	974	970	970	972.0
TEMP IN RIGHT	975	974	973	972	967	964	961	960	967	973	975	975	969.6
TEMP IN LEFT	973	972	969	969	964	961	959	958	965	970	973	971	967
TEMP OUT RIGHT	960	956	952	949	934	926	919	924	942	953	958	953	943.8
TEMP OUT LEFT	962	957	952	950	934	925	915	921	942	954	959	960	944.2
SET POINT TEMP	976	976	976	976	976	976	976	976	976	976	976	976	976

DATA SHEET
RETEC'S BENCH-SCALE THERMAL DESORPTION SYSTEM

DATE: 8/1/91

PROJECT #: H80-755

CLIENT: SAIC

WASTE TYPE: Sediment - PCB

TREATMENT GOAL: Hi - Temperature Treatment

TEST RESULTS

MIN. TO HOPPER EMPTY: 20

MATERIAL IN (lbs): 3.125

MATERIAL OUT (lbs): 2.875

DENSITY (lb/cu.ft.):

FEED RATE (lb/hr): 9.37

MASS REDUCTION %: 0.876

DATA SHEET

RETEC'S BENCH-SCALE THERMAL DESORPTION SYSTEM

DATE: 8/1/91

PROJECT #: 1180-755

CLIENT: SAIC

WASTE TYPE: Sediment

TREATMENT GOAL: III - Temperature Treatment

PROPOSED TEST CONDITIONS

RESIDANCE TIME: 30 minutes (90 Total) TEST START TIME: 4:10

MATERIAL TEMPERATURE: 550 HOPPER EMPTY TIME: 4:30

CARRIER GAS TEMPERATURE: 1000 TEST END TIME: 5:00

MEDIA TEMPERATURE: 650 ENGINEER: Mike Gardner

TIME	4:10	4:20	4:30	4:40	4:50	5:00			AVG.
OIL TEMP IN	607	666	611	613	612	612			620.1666
OIL TEMP OUT	530	530	532	533	532	532			531.5
HEAD SPACE TEMP	499	483	477	462	467	471			476.5
CARRIER GAS TEMP	976	982	986	981	982	987			982.3333
OFF GAS TEMP	312	296	286	287	302	296			296.5
SOIL TEMP IN	300								300
SOIL TEMP OUT		290	325	410	450	489			392.8

DATA SHEET
RETEC'S BENCH-SCALE THERMAL DESORPTION SYSTEM

DATE: 8/1/91
PROJECT #: H80-755
CLIENT: SAIC
WASTE TYPE: Sediment - PCB
TREATMENT GOAL: Hi - Temperature Treatment

TEST RESULTS

MIN. TO HOPPER EMPTY: 15
MATERIAL IN (lbs): 4.125
MATERIAL OUT (lbs): 3.12
DENSITY (lb/cu.ft.):
FEED RATE (lb/hr): 16.5
MASS REDUCTION %: ~~0.757575~~ 24.476

DATA SHEET

RETEC'S BENCH-SCALE THERMAL DESORPTION SYSTEM

DATE: 8/1/91

PROJECT #: H80-755

CLIENT: SAIC

WASTE TYPE: Sediment

TREATMENT GOAL: Hi - Temperature Treatment

PROPOSED TEST CONDITIONS

RESIDANCE TIME: 30 (60 minutes total) TEST START TIME: 3:20

MATERIAL TEMPERATURE: 500 HOPPER EMPTY TIME: 3:35

CARRIER GAS TEMPERATURE: 1000 TEST END TIME: 4:05

MEDIA TEMPERATURE: 600 ENGINEER: Mike Gardner

TIME	3:20	3:30	3:40	3:50	3:55	4:05		AVG.
OIL TEMP IN	606	599	601	607	601	603		602.8333
OIL TEMP OUT	526	526	525	531	532	534		529
HEAD SPACE TEMP	509	519	516	490	501	494		504.8333
CARRIER GAS TEMP	964	971	967	972	976	976		971
OFF GAS TEMP	299	300	303	308	308	310		304.6666
SOIL TEMP IN	160	160						160
SOIL TEMP OUT			347	439	500	496		445.5

DATA SHEET

RETEC'S BENCH-SCALE THERMAL DESORPTION SYSTEMDATE: 8/1/91PROJECT #: 1180-755CLIENT: SAICWASTE TYPE: SedimentTREATMENT GOAL: Hi Temp TreatmentPROPOSED TEST CONDITIONSRESIDANCE TIME: 30 minutesMATERIAL TEMPERATURE: 500CARRIER GAS TEMPERATURE: 1000MEDIA TEMPERATURE: 650TEST START TIME: 2:15HOPPER EMPTY TIME: 2:50TEST END TIME: 3:15ENGINEER: Mike Gardner

TIME	2:15	2:25	2:35	2:45	2:55	2:05	3:10		AVG.
OIL TEMP IN	608	593	595	596	597	606	607		600.2857
OIL TEMP OUT	530	519	509	509	510	523	530		518.5714
HEAD SPACE TEMP	547	518	455	406	285	440	476		446.7142
CARRIER GAS TEMP	958	965	966	968	964	969	964		964.8571
OFF GAS TEMP	169	280	288	281	269	273	281		263
SOIL TEMP IN	75	75	75	75					75
SOIL TEMP OUT			383	421	460	440	450		430.8

DATA SHEET
RETEC'S BENCH-SCALE THERMAL DESORPTION SYSTEM

DATE: 8/1/91

PROJECT #: H80-755

CLIENT: SAIC

WASTE TYPE: Sediment - PCB

TREATMENT GOAL: Hi - Temperature Treatment

TEST RESULTS

MIN. TO HOPPER EMPTY: 40

MATERIAL IN (lbs): 10.9

MATERIAL OUT (lbs): 4.125

DENSITY (lb/cu.ft.):

FEED RATE (lb/hr): 16.35

MASS REDUCTION %: ~~0.378440~~ 62.170

Feed Note: Gray - Fine grained saturated sediment with slight organic and sulfide odor, free liquid.

APPENDIX B

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, oil and grease and volatile solids 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 polynuclear aromatic hydrocarbons (PAHs) and polychlorinated biphenyls (PCBs) were determined as a check on their fate resulting from treating the sediments. In addition, for the ReTec water residual sample, total suspended solids and conductivity analysis were performed. 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 QC results, modifications and deviations from the QAPP, and the results of a laboratory audit performed. Any possible effects of deviations or audit findings on data quality are 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 and precision are 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:

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

where: C_i = 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 a measure of the amount of valid data produced compared to the total amount of data planned for the project. For the ReTec treatability studies, one of two samples collected as contamination checks of the system was broken during sample shipment. Though all guidelines for QA objectives were not met, all data generated was deemed useable.

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. Samples of untreated and treated sediment and residuals were taken by SAIC personnel during Phase II of these tests. Samples were shipped under chain-of-custody to Battelle Marine Sciences Laboratory in Sequim, Washington. Therefore, the data is representative of material actually treated.

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. However, because specialized procedures were used in some instances, the data may not be directly comparable to projects outside 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 for the ReTec technology evaluation.

PAHs

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 isotopically-labelled 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 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 for each PAH. 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 ReTec 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. (This criteria was not applied by Battelle to method blanks.) All samples were acceptable with respect to the guidelines used for surrogate recoveries.

As required by the QAPP, triplicate analyses of the Ashtabula River untreated sediment (A-US-RE) 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 RSDs with the exception of benzo(k)fluoranthene fell within the control limits specified. The lack of precision for benzo(k)fluoranthene can be attributed to concentrations near analytical detection limits. All matrix spike recoveries fell within specified control limits.

As required by the QAPP, a matrix spike and a matrix spike duplicate (MS/MD) analysis was performed for the treated Ashtabula River sediment (A-TS-RE). These results are presented in Table QA-3. Recoveries for benzo(g,h,i)perylene were slightly outside the specified accuracy control limits. Due to the minimal quantity of this compound found in the sample, the total PAH concentration is minimally affected.

TABLE QA-1. PAH SURROGATE RECOVERIES

Sample	d8-Naphthalene (%)	d10-Acenaphthalene (%)	d12-Perylene (%)	Control Limits (%)
A-US-RE, Rep. 1	58	70	93	40 - 120
A-US-RE, Rep. 2	72	76	95	
A-US-RE, Rep. 3	74	80	100	
Method Blank	97	91	71	
A-TS-RE	74	78	61	40 - 120
Method Blank	77	80	94	
A-WR-RE	29 *	61	51	40 - 120
Method Blank	79	77	66	
A-OR-RE, Rep. 1	72	81	94	40 - 120
A-OR-RE, Rep. 2	61	68	80	
A-OR-RE, Rep. 3	72	80	89	
A-OR-RE3	71	109	90	
Method Blank	23 *	26 *	72	

* Outside Control Limits

(1) Insufficient sample remained for reanalysis of water residuals

As required by the QAPP, a matrix spike and a matrix spike duplicate (MS/MSD) analysis was performed on the Ashtabula River water residual (A-WR-RE). These results are presented in Table QA-4. Due to the high concentrations of PAHs found in this water samples, many of the spiking levels were too low. Only those recoveries obtained from spike greater than half of the sample concentration are presented. The recovery problem observed for benzo(b)fluoranthene could not be identified. Due to the high concentrations of the other 15 PAHs analyzed, total PAH concentration should be minimally affected. As this matrix is noncritical, removal efficiencies are not affected.

The QAPP specified that triplicate analyses and a matrix spike be performed on the Ashtabula River oil residual (A-OR-RE). These results are summarized in Table QA-5. Due to the high concentrations of naphthalene and phenanthrene found, spiking levels were too low for accurate recovery determinations.

One certified National Institute of Science and Technology (NIST) standard reference material (SRM) was extracted and analyzed with the sediment samples. The recoveries for this standard are summarized

TABLE QA-2. PAH REPLICATE AND SPIKE RESULTS FOR B-US-ST

Compound	Replicate 1 dry ppb	Replicate 2 dry ppb	Replicate 3 dry ppb	Mean	RSD (%)	Precision Control Limits (%)	Recovery (%)	Accuracy Control Limits (%)
Naphthalene	300U	222	243	NC	NC	20	58	40 - 120
Acenaphthylene	300U	300U	200U	NC	NC		71	
Acenaphthene	400U	400U	300U	NC	NC		69	
Fluorene	400U	300U	247	NC	NC		76	
Phenanthrene	1420	1340	1380	1380	2.8		78	
Anthracene	300U	300U	171	NC	NC		82	
Fluoranthene	1120	971	1020	1040	7.6		88	
Pyrene	1010	905	927	949	6.1		87	
Benzo(a)anthracene	393	324	356	358	10		91	
Chrysene	585	520	571	559	6.1		86	
Benzo(b)fluoranthene	437	385	522	448	15		85	
Benzo(k)fluoranthene	368	265	410	348	21*		84	
Benzo(a)pyrene	384	300	326	337	13		89	
Indeno(1,2,3,c,d)pyrene	333	285	387	335	15		89	
Dibenzo(a,h)anthracene	200U	200U	88	NC	NC		92	
Benzo(g,h,i)perylene	348	283	277	303	13		61	

NC = Not Calculated

U = Undetected

TABLE QA-3. PAH MS/MSD RESULTS FOR A-TS-RE

Compound	MS Recovery (%)	MSD Recovery (%)	RPD	Accuracy Control Limits (%)	Precision Control Limits (%)
Naphthalene	52	46	12	40 - 120	20
Acenaphthylene	60	53	12		
Acenaphthene	73	68	7.1		
Fluorene	73	69	5.6		
Phenanthrene	79	76	3.9		
Anthracene	75	71	5.5		
Fluoranthene	82	79	3.7		
Pyrene	82	78	5.0		
Benzo(a)anthracene	78	73	6.6		
Chrysene	77	75	2.6		
Benzo(b)fluoranthene	74	70	5.6		
Benzo(k)fluoranthene	74	70	5.6		
Benzo(a)pyrene	68	60	12		
Indeno(1,2,3,c,d)pyrene	57	47	19		
Dibenzo(a,h)anthracene	66	56	16		
Benzo(g,h,i)perylene	38 *	35 *	8.2		

* Outside Control Limits

in Table QA-6. No recovery was obtained for anthracene as the certified value was less than the analytical detection limit achieved.

Method blanks were extracted and analyzed with each set of samples extracted. Insignificant quantities of benzo(g,h,i)perylene were detected in the blanks analyzed with the sediment and water samples. No corrections were performed for method blanks as no consistent significant contamination problems were observed.

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*. Oils were diluted 1:10 in hexane. One surrogate, tetrachloro-m-xylene, was 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;

TABLE QA-4. PAH MS RESULTS FOR A-WR-RE

Compound	MS Recovery (%)	MSD Recovery (%)	RPD	Accuracy Control Limits (%)	Precision Control Limits (%)
Naphthalene	IS	IS	NC	Not Specified	Not Specified
Acenaphthylene	61	48	24		
Acenaphthene	IS	IS	NC		
Fluorene	IS	IS	NC		
Phenanthrene	IS	IS	NC		
Anthracene	IS	IS	NC		
Fluoranthene	IS	IS	NC		
Pyrene	IS	IS	NC		
Benzo(a)anthracene	IS	IS	NC		
Chrysene	IS	IS	NC		
Benzo(b)fluoranthene	10	-8	NC		
Benzo(k)fluoranthene	73	67	7.1		
Benzo(a)pyrene	IS	IS	NC		
Indeno(1,2,3,c,d)pyrene	53	52	1.9		
Dibenzo(a,h)anthracene	64	62	3.2		
Benzo(g,h,i)perylene	38	25	41		

IS = Inappropriate Spiking Concentration
NC = Not Calculated

criteria for percent 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.

PCB QC Results and Discussion

Surrogate recoveries for all PCB samples for the ReTec demonstration are summarized in Table QA-7. Water sample could not be quantified due to unidentified coeluting peaks. All samples were acceptable with respect to the surrogate criteria used with the exception of the water residual. The surrogate recovery for the presence of these peaks also resulted in significantly increased Aroclor detection limits (approximately 25 to 50 times higher than the method blank).

TABLE QA-5. PAH REPLICATE RESULTS FOR A-OR-RE

Compound	Replicate 1 ppb	Replicate 2 ppb	Replicate 3 ppb	Mean	RSD (%)	Precision Control Limits (%)	Recovery (%)	Accuracy Control Limits (%)
Napthalene	964000	1010000	1130000	1040000	8.4	Not Specified	IS	Not Specified
Acenaphthylene	6300	6670	7140	6700	6.2		83	
Acenaphthene	73300	76000	83500	77600	6.8		96	
Fluorene	112000	111000	122000	115000	5.5		104	
Phenanthrene	241000	254000	278000	258000	7.2		IS	
Anthracene	35400	37500	40200	37700	6.4		99	
Flouranthene	13700	14400	15600	14500	6.6		106	
Pyrene	79300	84000	90000	84600	6.4		110	
Benzo(a)anthracene	20200	21400	23100	21500	6.8		100	
Chrysene	46400	48100	52000	48800	5.9		102	
Benzo(b)fluoranthene	8830	8660	9430	8970	4.5		84	
Benzo(k)fluoranthene	1000U	1000U	2000U	NC	NC		92	
Benzo(a)pyrene	12000	11900	13300	12400	6.3		93	
Indeno(1,2,3,c,d)pyrene	1340	1500	2000U	NC	NC		93	
Dibenzo(a,h)anthracene	1940	2060	2510	2170	14		98	
Benzo(g,h,i)perylene	6290	6860	7570	6910	9.3		66	

IS = Inappropriate Spiking Concentration

TABLE QA-6. PAH SRM RESULTS

Compound	Recovery (%)	Control Limits (%)
Naphthalene	NC	80 - 120
Acenaphthylene	NC	
Acenaphthene	NC	
Fluorene	NC	
Phenanthrene	90	
Anthracene	0 *	
Fluoranthene	87	
Pyrene	92	
Benzo(a)anthracene	82	
Chrysene	NC	
Benzo(b)fluoranthene	99	
Benzo(k)fluoranthene	142 *	
Benzo(a)pyrene	78 *	
Indeno(1,2,3,c,d)pyrene	98	
Dibenzo(a,h)anthracene	NC	
Benzo(g,h,i)perylene	75 *	

NC = Not Certified

* Outside Control Limits

As required by the QAPP, triplicate analyses of the Ashtabula River untreated sediment (A-US-RE) were performed to assess precision. These results fell within specific guidelines and are summarized in Table QA-8. A matrix spike using Aroclor 1254 was performed on the same sample to assess accuracy; no recovery could be determined due to residual Aroclor 1248 peaks.

As required by the QAPP, a matrix spike and a matrix spike duplicate (MS/MSD) analysis was performed for the treated Ashtabula River sediment (A-TS-RE). These results are presented in Table QA-9. Both the recoveries and RSD were acceptable.

As required by the QAPP, a matrix spike and a matrix spike duplicate (MS/MSD) analysis was performed on the Ashtabula River water residual (A-WR-RE). Due to the presence of unidentified coeluting peaks, recoveries could not be determined.

The QAPP specified that triplicate analyses and a matrix spike be performed on the Ashtabula River oil residual (A-OR-RE). These results are summarized in Table QA-10.

TABLE QA-7. PCB SURROGATE RECOVERIES

Sample	Tetrachloro-m-xylene (%)	Control Limits (%)
A-US-RE, Rep. 1	101	40 - 120
A-US-RE, Rep. 2	109	
A-US-RE, Rep. 3	113	
Method Blank	102	
A-TS-RE	91	40 - 120
Method Blank	102	
A-WR-RE	NQ	40 - 120
Method Blank	20 *	
A-OR-RE, Rep. 1	108	
A-OR-RE, Rep. 2	93	
A-OR-RE, Rep. 3	100	
A-OR-RE3	96	
Method Blank	35 *	

* Outside Control Limits

NQ = Not quantifiable due to unidentified coeluting peaks

One standard reference material (SRM) certified by the National Research Council of Canada (NRCC) for Aroclor 1254 was extracted and analyzed with the sediment samples. a recovery of 190% was obtained. This can be attributed to a certified value near the analytical detection limit.

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

Early eluting large peaks were present in the untreated sediment, water residual, and oil residual. These peaks did not correspond to any Aroclor pattern and were therefore not quantified. Their presence, however, did create some QA/QC problems as have been discussed.

TABLE QA-8. PCB REPLICATE RESULTS FOR A-TS-RE

Aroclor	Replicate 1 ppb dry		Replicate 2 ppb dry		Replicate 3 ppb dry		Mean		RSD (%)	Precision Control Limits (%)
1242	200	U	200	U	200	U	200	U	NC	20
1248	14400		13900		15600				6.0	20
1254	100	U	100	U	100	U	100	U	NC	20
1260	100	U	100	U	100	U	100	U	NC	20

U = Undetected
NC = Not Calculated

TABLE QA-9. PCB MS/MSD RESULTS FOR A-TS-RE

PCB	MS Recovery (%)	MSD Recovery (%)	RPD	Accuracy Control Limits (%)	Precision Guideline Limits (%)
Aroclor 1254	78	76	2.6	40 - 120	20

U = Undetected
NC = Not Calculated
NS = Not Spiked

* Outside Control Limits

TABLE QA-10. PCB REPLICATE AND SPIKE RESULTS FOR A-OR-RE

Aroclor	Replicate 1 (ppb)		Replicate 2 (ppb)		Replicate 3 (ppb)		Mean		RSD (%)	Precision Control Limits (%)	Recovery (%)	Accuracy Control Limits
1242	2000	U	2000	U	2000	U	2000	U	NC	Not Specified	NS	Not Specified
1248	2000	U	2000	U	2000		2000	U	NC		NS	
1254	1000	U	1000	U	1000	U	1000	U	NC		73	
1260	1000	U	1000	U	1000	U	1000	U	NC		NS	

U = Undetected
NC = Not Calculated
NS = Not Spiked

METALS

Metals Procedures - Sediments

Sediments were prepared for metals analysis by freeze-drying, blending, and grinding. Sediments for Ag, Cd, Hg, and Se were digested using nitric and hydrofluoric acids. The digestates were analyzed for Ag, 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 0.5 g aliquot of dried, ground sediment pressed into a pellet with a diameter of 2 cm.

Metals Procedures - Water

The water sample was analyzed for all metals but Ba and Hg using direct injection flame atomic absorption (FLAA). Barium was analyzed using ICP/MS; Hg was analyzed using cold vapor AA (CVAA).

Metals QC Results and Discussion

Triplicate analyses of the Ashtabula River untreated sediment (A-US-RE), treated sediment (A-TS-RE), and water residual (A-WR-RE) were performed to assess precision. Matrix spikes were analyzed for the same samples to assess accuracy. Results are summarized in Tables QA-11, QA-12 and QA 13. It should be noted that the sediments were not spiked for XRF analysis as spiking is not appropriate for that analysis.

Accuracy and precision results for metals were acceptable with only a few minor exceptions, as shown in Tables QA 11, QA-12, and QA-13. An RSD result for mercury was outside limits due to concentrations near the analytical detection limits. Some recoveries for silver and selenium were outside limits, but due to the low concentrations found, data should be minimally affected.

One solid NIST certified standard reference material (SRM) was digested and analyzed with the sediment samples for XRF, GFAA, and CVAA analyses. These results are presented in Table QA-14. One aqueous NIST SRM was digested and analyzed with the water samples; results are also presented in Table QA-14. No reason for zero recovery for Se was identified; matrix spike recovery was good.

Method blanks were digested and analyzed for the metals analyzed by GFAA, CVAA, and FLAA (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.

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). Water samples were extracted with freon and analyzed gravimetrically as described in Method 413.1 from the same reference above.

Oil and Grease QC Results and Discussion

The untreated Ashtabula River sediment, (A-US-RE) and treated sediment (A-TS-RE), and water residual (A-W-RE) were analyzed for oil and grease in triplicate. Results are presented in Table QA-15. RPD results for A-TS-RE fell slightly outside specified guidelines; data is not significantly impacted.

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. Waters were analyzed using Method 160.4; a volume of sample was dried and then ignited at 550°C. The loss of weight on ignition was then determined.

TABLE QA-11. METALS REPLICATE AND SPIKE RESULTS FOR A-US-RE

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	0.18	0.19	0.20	0.19	5.3	20	124*	85 - 115
As	XRF	20.9	20.0	21.4	20.8	3.4		NS	---
Ba	XRF	892	906	910	903	1.0		NS	---
Cd	GFAA	3.02	3.04	3.12	3.06	1.7		97	85 - 115
Cr	XRF	588	624	561	591	5.3		NS	---
Cu	XRF	33.9	34.6	32.6	33.7	3.0		NS	---
Fe(1)	XRF	4.14	4.32	4.32	4.26	2.4		NS	---
Hg	CVAA	1.362	1.337	1.387	1.361	1.9		95	85 - 115
Mn	XRF	537	573	566	559	3.4		NS	---
Ni	XRF	49.5	54.9	54.7	53.0	5.8		NS	---
Pb	XRF	56.7	57.9	60.8	58.5	3.6		NS	---
Se	GFAA	0.87	0.99	0.87	0.91	7.6		175*	85 - 115
Zn	XRF	228	237	238	234	2.0		NS	---

NS = Not Spiked

* Outside Control Limits

(1) Results in Percent for Fe

TABLE QA-12. METALS REPLICATE AND SPIKE RESULTS FOR A-TS-RE

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	0.19	0.18	0.19	0.19	3.1	20	118*	85 - 115
As	XRF	15.3	16.5	17.6	16.5	7.0		NS	—
Ba	XRF	789	811	775	792	2.3		NS	—
Cd	GFAA	2.74	2.67	2.67	2.69	1.5		100	85 - 115
Cr	XRF	494	514	552	520	5.7		NS	—
Cu	XRF	47.7	44.6	52.0	48.1	7.7		NS	—
Fe(1)	XRF	3.85	3.92	3.97	3.91	1.5		NS	—
Hg	CVAA	0.005	0.006	0.003	0.005	33*		97	85 - 115
Mn	XRF	520	533	538	530	1.8		NS	—
Ni	XRF	73.3	80.7	77.7	77.1	4.8		NS	—
Pb	XRF	75.2	76.8	78.9	77.0	2.4		NS	—
Se	GFAA	1.60	1.49	1.49	1.53	4.2		172*	85 - 115
Zn	XRF	223	227	243	231	4.6		NS	—

NS = Not Spiked

* Outside Control Limits

U = Undetected

(1) Result in Percent for Fe

TABLE QA-13. METALS REPLICATE AND SPIKE RESULTS FOR A-WR-RE

Metal	Method	Replicate 1, ppb	Replicate 2, ppb	Replicate 3, ppb	Mean	RSD (%)	Precision Control Limits (%)	Recovery (%)	Accuracy Control Limits (%)
Ag	FLAA	0.003	0.001U	0.001U	NC	NC	20	56*	85 - 115
As	FLAA	7.37	7.53	7.22	7.37	2.1		87	
Ba	ICP/MS	55.2	56.0	55.6	55.6	0.7		90	
Cd	FLAA	0.72	0.56	0.54	0.61	16		84*	
Cr	FLAA	17.9	17.9	16.2	17.3	5.7		84*	
Cu	FLAA	47.6	45.9	45.9	46.5	2.1		95	
Fe	FLAA	1790	1760	1860	1800	2.8		90	
Hg	CVAA	35.7	34.6	32.3	34.2	5.1		106	
Mn	FLAA	482	489	459	477	1.2		91	
Ni	FLAA	152	152	136	147	6.3		86	
Pb	FLAA	43.8	43.8	43.8	43.8	0		87	
Se	FLAA	2U	2U	2U	2U	NC		102	
Zn	FLAA	202	195	208	202	3.2		104	

NS = Not Spiked
U = Undetected

* Outside Control Limits

TABLE QA-14. METALS SRM RECOVERIES

Metal	Sediment SRM (%)	Water SRM (%)	Control Limits (%)
Ag	NC	93.7	80 - 120%
As	96.6	103	
Ba	NC	99.2	
Cd	111	105	
Cr	93.4	100	
Cu	92.2*	87.0	
Fe	99.4	99.5	
Hg	103	96.2	
Mn	93.1	106	
Ni	97.2	85.1	
Pb	105	104	
Se	NC	0	
Zn	89.9	93.4	

* Outside Control Limits
 NC = Not Certified

TABLE QA-15. OIL AND GREASE REPLICATES AND SPIKE RESULTS FOR A-US-RE, A-TS-RE, AND A-WR-RE

Sample	Replicate 1	Replicate 2	Replicate 3	Mean	RSD (%)	Precision Control Limits (%)
A-US-RE, ppm dry	919	1080	1010	1000	8.0	20
A-TS-RE, ppm dry	563	357	389	436	25*	20
A-WR-RE, ppm	560	560	574	564	1.4	

NS = Not Spiked

Total Volatile Solid QC Results and Discussion

The Ashtabula River untreated sediment (A-US-RE), treated sediment (A-TS-RE), and water residual A-WR-RE) were analyzed for TVS in triplicate. Results are summarized in Table QA-16. All RSDs fell within specified control limits.

TABLE QA-16. TVS REPLICATES FOR A-US-RE, A-TS-RE, AND A-WR-RE

Sample	Replicate 1	Replicate 2	Replicate 3	Mean	RSD (%)	Control Limits (%)
A-US-RE, %	7.54	7.39	7.99	7.64	4.1	20
dry	4.30	4.33	4.13	4.25	2.5	20
A-TS-RE, %	1400	1400	1400	1400	0	
dry						
A-WR-RE, ppm						

OTHER ANALYSESpH

Sediment samples were analyzed for pH using SW-846 Method 9045. Sediment and water were combined in a 1:10 ratio rather than the required 1:1 ratio and mixed prior to pH determination. These results should be used with caution. Water samples were analyzed for pH using SW-846 Method 9040. Replicate pH results are presented in Table QA-17.

TABLE QA-17. pH REPLICATE RESULTS

Sample	Replicate 1	Replicate 2	Replicate 3	Mean	RSD (%)	Control Limits (%)
A-US-RE, SU	7.84	7.91	7.88	7.88	0.4	Not Specified
A-TS-RE, SU	8.07	8.07	8.14	8.09	0.5	
A-WR-RE, SU	8.15	8.24	8.20	8.20	0.6	

Total Organic Carbon (TOC)

Sediment and water samples were analyzed for TOC using SW-846 Method 9060. Two SRMs were analyzed with the sediments, yielding recoveries of 95.6 percent and 100 percent. Replicate TOC results are presented in Table QA-18.

TABLE QA-18. TOC REPLICATE RESULTS

Sample	Replicate 1	Replicate 2	Replicate 3	Mean	RSD (%)	Control Limits (%)
A-TS-RE, %	2.33	2.27	2.21	2.27	2.6	Not Specified
dry	428	452	458	446	3.6	
A-WR-RE, ppm						

Total Cyanide

Sediment and water samples were analyzed for cyanide by SW-846 Method 9010. For the sediments, approximately 5 g of sediment was distilled; the distillate was analyzed spectrophotometrically. Replicate and spike results for cyanide are presented in Table QA-19.

Total Phosphorus

Sediment and water samples were analyzed for phosphorus by EPA Method 365.2. For sediments, Approximately 1 g of sediment was digested; the digestate was analyzed spectrophotometrically. Replicate and spike results for phosphorus are presented in Table QA-20.

Total Solids

Sediment and water samples were analyzed for total solids using EPA Method 160.3. Replicate results are presented in Table QA-21.

Total Suspended Solids

Water samples were analyzed for total suspended solids using EPA Method 160.2. Replicate results are presented in Table QA-22.

Conductivity

Water samples were analyzed for conductivity using SW-846 Method 9050. Replicate results are presented in Table QA-23.

BOD

BOD was requested for the water sample using EPA Method 405.1. The holding time was exceeded at the laboratory and the analysis was not performed.

TABLE QA-19. CYANIDE REPLICATE AND SPIKE RESULTS

Sample	Replicate 1	Replicate 2	Replicate 3	Mean	RSD (%)	Precision Control Limits (%)	Recovery (%)	Accuracy Control Limits
A-US-RE, ppm dry	1.1	2.0	1.5	1.5	29	Not Specified	NS	Not Specified
A-TS-RE, ppm dry	2.1	2.1	NA	2.1	NC		93	
A-WR-RE, ppm	0.004	0.006	0.004 U	NC	NC		96	

NA = Not Analyzed
U = Undetected
NC = Not Calculated
NS = Not Spiked

TABLE QA-20. PHOSPHORUS REPLICATE AND SPIKE RESULTS

Sample	Replicate 1	Replicate 2	Replicate 3	Mean	RSD (%)	Precision Control Limits (%)	Recovery (%)	Accuracy Control Limits
A-US-RE, ppm dry	1200	1440	1220	1280	11	Not Specified	NS	Not Specified
A-TS-RE, ppm dry	2290	2070	2060	2140	6.1		106	
A-WR-RE, ppm	0.439	0.488	0.401	0.443	9.9		97	

NS = Not Spiked

TABLE QA-21. TOTAL SOLIDS REPLICATE RESULTS

Sample	Replicate 1	Replicate 2	Replicate 3	Mean	RSD (%)	Precision Control Limits (%)
A-US-RE, %	38.2	37.8	31.0	35.6	11	20
A-TS-RE, %	20.5	25.5	25.5	23.8	12	20
A-WR-RE, ppm	1600	1500	1700	1600	6.3	Not Specified

TABLE QA-22. TOTAL SUSPENDED SOLIDS REPLICATE RESULTS

Sample	Replicate 1	Replicate 2	Replicate 3	Mean	RSD (%)	Precision Control Limits (%)
A-WR-RE, ppm	70	85	88	81	12	Not Specified

TABLE QA-23. CONDUCTIVITY REPLICATE RESULTS

Sample	Replicate 1	Replicate 2	Replicate 3	Mean	RSD (%)	Precision Control Limits (%)
A-WR-RE, μ ho/cm	2.49	2.37	2.31	2.39	3.8	Not Specified

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 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 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.

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 isotopically-labelled 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. Peaks were considered valid if the peak shape was good, if there was no tailing, and if there was little or no coelution with other peaks. A definite Aroclor pattern was necessary for quantification of PCBs.
- 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.

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 guideline QA objectives and internal QC checks criteria guidelines 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), some 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 if these limits were exceeded.
- 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).

SAMPLE HOLDING TIMES

Water Samples

The QAPP specified holding times for water samples only. All water extractions for the critical organic parameters were performed within these holding times (from the time of sample receipt). PAH analysis of the water extract was performed approximately 12 days past the 40 day holding time. PCB analysis of the water extract was performed approximately 40 days past the 40 day holding time. Due to the noncritical nature of the water sample, removal efficiencies are not affected. Holding times for solids, TOC, cyanide, and phosphorus were exceeded slightly.

Sediment/Oil Samples

Though holding times for organics in sediment and oil samples were not specified in the QAPP, the referenced SW-846 methods do require that extractions be done within 14 days and that the analysis of the extracts be performed within 40 days after extraction. Any analyses exceeding these criteria for the critical parameters will be discussed below.

PAHs/PCBs

Analyses of PAH and PCB extracts for the sediments were performed approximately 12 and 40 days past the 40 day holding time, respectively. As both untreated and treated sediment extracts were analyzed similarly, relative removal efficiencies should not be affected.

CONCLUSIONS AND LIMITATIONS OF DATA

Upon review of all sample data and associated QC results, the data generated for the ReTec treatability study has 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.

pH analyses for the sediments were performed using a 1:10 soil:water ration rather than the required 1:1. This data should be used with caution.

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.

Large unidentified peaks were observed in the PCB analyses of the untreated sediment, water residual, and oil residual samples. Due to the high concentration of PCBs present in the untreated sediment, the necessary dilutions eliminated any affect on data quality. For the water and oil samples, detection limits had to be increased significantly because of these peaks. While removal efficiencies are not affected, mass balance closures may be difficult.

As discussed previously, the analytical laboratory used several specialized methods when analyzing samples from the ReTec treatability study. These same methods, however, have been used in analyzing all samples generated to date in support of the ARCS Program. Therefore, while the data generated for the Soil Tech treatability study may not be comparable to data generated by standard EPA methods, it is comparable to data generated within the ARCS Program.

The following Data Verification Report is an attachment to Appendix B. Page numbering resumes with Appendix C at page 105.

Data Verification Report For Assessment and Remediation of Contaminated Sediment Program

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

By

M. J. Miah, M. T. Dillon, and N. F. D. O'Leary

**Lockheed Environmental Systems and Technologies Company
980 Kelly Johnson Drive
Las Vegas, Nevada 89119**

Version 1.0

**Work Assignment Manager
Brian A. Schumacher
Exposure Assessment Research Division
Environmental Monitoring Systems Laboratory
Las Vegas, Nevada 89193**

**Environmental Monitoring Systems Laboratory
Office of Research and Development
U. S. Environmental Protection Agency
Las Vegas, Nevada 89193**

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 ≥ 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 A₁. 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 A₁ 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 S₉ 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-B₀ C₀ D₀ S₀, 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 ₀ D ₀	12-C ₀ D ₀	12-C ₀ D ₀	12-C ₀ D ₀
% Moisture	0-A ₀ B ₀ C ₀ D ₀ P ₀ S ₀	3-A ₀ B ₀ C ₀ D ₀ S ₀	0-A ₀ B ₀ C ₀ D ₀ P ₀ S ₀	3-A ₀ B ₀ C ₀ D ₀ S ₀
pH	0-A ₀ B ₀ C ₀ D ₀ P ₀ S ₀	0-A ₀ B ₀ C ₀ D ₀ P ₀ S ₀	0-A ₀ B ₀ C ₀ D ₀ P ₀ S ₀	3-A ₀ B ₀ C ₀ D ₀ S ₀
%TVS	6-A ₀ C ₀ D ₀ S ₀	3-A ₀ B ₀ C ₀ D ₀ S ₀	6-A ₀ C ₀ D ₀ S ₀	6-A ₀ C ₀ D ₀ S ₀
Oil and grease	15-A ₀ C ₀	6-A ₀ B ₀ C ₀ D ₀ S ₀	6-A ₀ B ₀ C ₀ D ₀ S ₀	9-A ₀ D ₀ C ₀ S ₀
TOC	12-C ₀ P ₀ S ₀	12-C ₀ P ₀ S ₀	12-C ₀ P ₀ S ₀	9-C ₀ D ₀ P ₀ S ₀
Total cyanide	14-A ₀ P ₀	14-A ₀ P ₀	11-A ₀ P ₀ S ₀	8-A ₀ D ₀ P ₀ S ₀
Total phosphorus	14-A ₀ P ₀	14-A ₀ P ₀	14-A ₀ P ₀	11-A ₀ D ₀ S ₀
PCBs	17-B ₀ D ₀	14-A ₀ B ₀ D ₀	14-A ₀ B ₀ D ₀	11-A ₀ B ₀ D ₀ S ₀
PAHs	17-D ₀ S ₀	11-B ₀ D ₀ S ₀ S ₀	17-D ₀ S ₀	20-D ₀
Treated Sediments				
Metals	12-C ₀ D ₀	12-C ₀ D ₀	12-C ₀ D ₀	12-C ₀ D ₀
% Moisture	0-A ₀ B ₀ C ₀ D ₀ P ₀ S ₀	0-A ₀ B ₀ C ₀ D ₀ P ₀ S ₀	3-A ₀ B ₀ C ₀ D ₀ S ₀	3-A ₀ B ₀ C ₀ D ₀ S ₀
pH	0-A ₀ B ₀ C ₀ D ₀ P ₀ S ₀	3-A ₀ B ₀ C ₀ D ₀ S ₀	0-A ₀ B ₀ C ₀ D ₀ P ₀ S ₀	3-A ₀ B ₀ C ₀ D ₀ S ₀
%TVS	6-A ₀ C ₀ D ₀ S ₀	3-A ₀ B ₀ C ₀ D ₀ S ₀	6-A ₀ C ₀ D ₀ S ₀	6-A ₀ C ₀ D ₀ S ₀
Oil and grease	15-A ₀ C ₀	6-A ₀ B ₀ C ₀ D ₀ S ₀	9-A ₀ B ₀ C ₀ D ₀	6-A ₀ C ₀ D ₀ P ₀ S ₀
TOC	12-C ₀ P ₀ S ₀	12-C ₀ P ₀ S ₀	12-C ₀ P ₀ S ₀	12-C ₀ D ₀ S ₀
Total cyanide	14-A ₀ P ₀	14-A ₀ P ₀	14-A ₀ P ₀	11-A ₀ D ₀ P ₀
Total phosphorus	14-A ₀ P ₀	14-A ₀ P ₀	14-A ₀ P ₀	14-A ₀ D ₀
PCBs	14-B ₀ D ₀ P ₀	11-A ₀ B ₀ D ₀ P ₀	14-B ₀ D ₀ P ₀	14-A ₀ B ₀ D ₀
PAHs	14-D ₀ P ₀ S ₀	17-D ₀ S ₀	14-D ₀ P ₀ S ₀	20-D ₀

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

Water residue				
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, D, P,	14-B, D, P,	5-A, B, D, P, S, S ₆	5-A, B, D, P, S, S ₆
PAHs	11-A, D, P, S,	17-D, S,	17-D, P,	11-A, D, P, S,
Oil residue				
PCBs	11-A, B, D, S,	*	17-B, D,	11-B, D, P, S,
PAHs	11-A, B, D, S,	*	14-B, D, S,	17-B, D,

* 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

Untreated Sediments	B.E.S.T.	ZIMPRO	Soil Tech	RETEC
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 ₂ D ₁ S ₁	11-B ₂ D ₁	17
TOC	17	17	17	17
Total cyanide	20	20	20	17-P ₁
Total phosphorus	20	20	20	20
PCBs	20-B ₂	17-A ₁ B ₂	17-A ₁ B ₂	17-A ₁ B ₂
PAHs	20-S ₂	14-B ₂ S ₁ S ₂	20-S ₂	23
Treated Sediments				
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 ₂ D ₁ S ₁	11-B ₂ D ₁	9-P ₁
TOC	17	17	17	17
Total cyanide	20	20	20	20
Total phosphorus	20	20	20	20
PCBs	17-B ₂ P ₁	14-A ₁ B ₂ P ₁	17-B ₂ P ₁	17-A ₁ B ₂
PAHs	17-P ₁ S ₂	20- S ₂	20-S ₂	23

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

Water residue				
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 ₂	20-B ₂	14-A ₁ B ₂ S ₁	20-B ₂
PAHs	17-P ₁ S ₂	20-S ₂	23	14-A ₁ P ₁ S ₁
Oil residue				
PCBs	14-A ₁ B ₂ S ₁	*	20-B ₂	20-B ₂
PAHs	17-B ₂ S ₂	*	17-B ₂ S ₂	20-B ₂

* 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.

NOTE

Appendix A - Laboratory Submitted Data Summary Sheets

and

Appendix D - ARCS Data Verification Templates by Parameter

are not included with 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

A₀ = no standard available/no information available

A₁ = accuracy limit for the reference materials exceeded

A₉ = accuracy is not applicable

B = Blank Problem

B₀ = no information available

B₂ = reagent blank value exceeded MDL

B₉ = blanks are not applicable

C = Calibration Problem

C₀ = no information available

C₁ = initial calibration problem

C₂ = on-going calibration problem

C₃ = no information on initial calibration

C₆ = no information on on-going calibration

C₉ = on-going calibration is not applicable

D = Detection Limit Problem

D₀ = no information available

D₁ = detection limit exceeded

D₉ = detection limit is not applicable

H = Holding Times Exceeded

P = Precision Problem

P₀ = no information available

P₁ = precision limit for analytical replicate exceeded the QA/QC requirements

P₃ = MSD exceeded the QA/QC requirement

P₉ = precision is not applicable

S = Spike Recovery Problem

S₀ = no information available on spike

S₁ = limit of matrix spike recovery exceeded

S₂ = limit of surrogate spike recovery exceeded

S₃ = no information available on matrix spike recovery

S₆ = no information available on surrogate spike recovery

S₉ = spike recovery not applicable

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

TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGES</u>	<u>REVISION</u>	<u>DATE</u>
1.0 INTRODUCTION	2	1	1/9/91
2.0 PROJECT DESCRIPTION	12	2	2/15/91
3.0 QUALITY ASSURANCE OBJECTIVES	2	2	2/15/91
4.0 SAMPLE TRANSFER AND PREPARATION PROCEDURES	4	1	1/9/91
5.0 ANALYTICAL PROCEDURES AND CALIBRATION	3	2	2/15/91
6.0 DATA REDUCTION, VALIDATION AND REPORTING	1	1	1/9/91
7.0 INTERNAL QUALITY CONTROL CHECKS	7	2	2/15/91
8.0 PERFORMANCE SYSTEMS AUDITS	1	2	2/15/91
9.0 CALCULATION OF DATA QUALITY IMPLICATORS	3	1	1/9/91
10.0 CORRECTIVE ACTION	2	1	1/9/91
11.0 QA/QC REPORTS TO MANAGEMENT	1	1	1/9/91
APPENDIX A - TECHNOLOGY SUMMARIES	3	1	1/9/91

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

Thomas J. Wagner
SAIC's Work Assignment Manager (print)

Thomas J. Wagner
Signature

2/5/91
Date

Clyde J. Dial
SAIC's QA Manager (print)

Clyde J. Dial
Signature

4/6/91
Date

Steve Yaksich
GLNPO Work Group Chair (print)

Signature

Date

Brian Schumacher
ARCS QA Officer (print)

Signature

Date

Gene Easterly
EPA, EMSL-LV, NRD QA Officer (print)

Signature

Date

Ralph Christensen
EPA Technical Project Manager (print)

Signature

Date

Dave Cowgill
ARCS Program Manager (print)

Signature

Date

DISTRIBUTION LIST:

Gene Easterly	U.S. EPA, EMSL (Las Vegas)
Brian Schumacher	LOCKHEED (Las Vegas)
Tony Kizlauskas	SAIC (Chicago)
Thomas Wagner	SAIC (Cincinnati)
Clyde Dial	SAIC (Cincinnati)
Steve Garbaciak	U.S. COE (Chicago)
Dennis Timberlake	U.S. EPA, RREL (Cincinnati)
Steve Yaksich	U.S. COE (Buffalo)
David Cowgill	U.S. EPA, GLNPO (Chicago)
Gary Baker	SAIC (Cincinnati)
Vic Engleman	SAIC (San Diego)

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.TM 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

<i>Description</i>	<i>Concentration (Mg/kgm)(a)</i>									<i>Concentration (%) (a)</i>		
	<i>Total PCB</i>	<i>Total PAH</i>	<i>Cu</i>	<i>Cd</i>	<i>Ni</i>	<i>Fe (%)</i>	<i>Cr</i>	<i>Zn</i>	<i>Pb</i>	<i>TOC</i>	<i>O & G</i>	<i>Moisture (b)</i>
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 (%)

<i>Description</i>	<i>Particle Size (a)</i>							<i>Median Diameter, u</i>
	<i>> 50 u</i>	<i>50-20 u</i>	<i>20-5 u</i>	<i>5-2 u</i>	<i>2-0.2 u</i>	<i>0.2-0.08 u</i>	<i>< 0.08 u</i>	
Buffalo River	19.8	12.1	29.0	11.8	24.3	2.4	0.6	9.3

(a) u micarons

GLNPO - QAPJP
 Section No.: 2
 Revision No.: 2
 Date: Feb. 15, 1991
 Page: 2 of 12

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

NOTES:

¹ 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.

² 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.

³ Detection limits for oil are ppm (mg/l).

⁴ Parameters tentatively identified for QC analyses.

⁵ 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 ¹
Total + Volatile Solids	5	10	15	Triplicate/Control
Oil & Grease	20	40	60	Triplicate/Control
Total Cyanide	10	--	10	None ²
Total Phosphorous	5	--	5	None ²
Metals (except Hg)	5	15	20	MS/Triplicate
Hg	1	3	4	MS/Triplicate
PCBs + PAHs	30	90(60) ³	90	(3)
pH	20	--	20	None ⁴
Subtotals	111	158(128)	269(239)	
Reserve	--	--	31(61)	
TOTAL	--	--	300	

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

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

³ Quality control for untreated solids is Triplicate and spike and for treated solids matrix spike and matrix spike duplicate.

⁴ 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

<i>Parameter</i>	<i>Priority</i>	<i>Analysis Volume, ml</i>	<i>QC Volume, ml</i>	<i>QC Approach</i>
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.

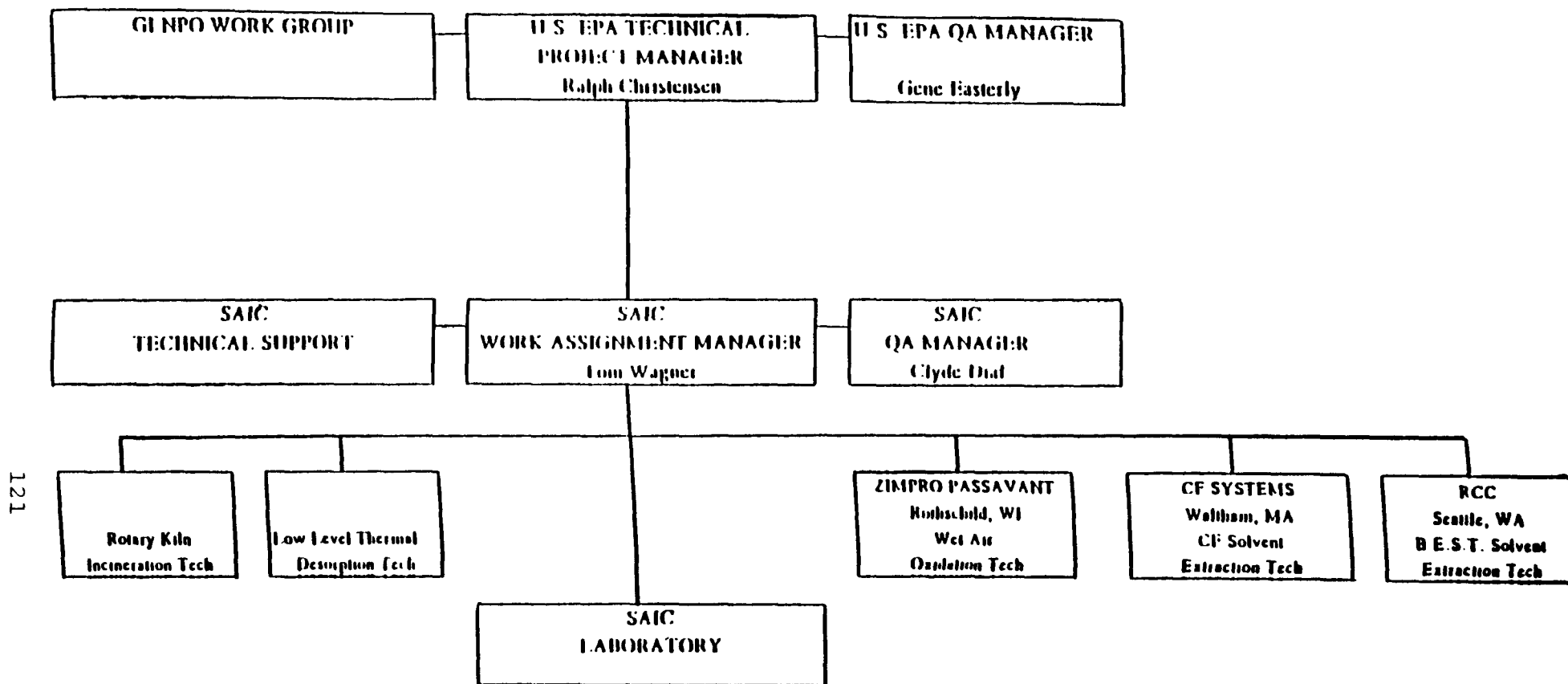


Figure 2-1. Project Organization

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)**

<i>Parameter</i>	<i>Method (a)</i>	<i>Accuracy (b) (as % recovery)</i>	<i>Precision (c) %</i>	<i>Method Detection Limit (d) (mg/kgm)</i>	<i>Completeness %</i>
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 (e))	3540 or 3550/8080	70-130	20	0.02	90
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.

GLNPO - QAPJP
 Section No.: 3
 Revision No.: 2
 Date: Feb. 15, 1991
 Page: 2 of 2

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 ₂ SO ₄ to pH < 2	28 days
Solids (Total, Volatile & Suspended)	P,G	Cool 4° C	7 days
Oil and Grease	G	Cool 4° C, H ₂ SO ₄ 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 ₂ SO ₄ to pH < 2	28 days
Metals (except Cr VI)	P,G	HNO ₃ 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 ₅	P,G	Cool 4° C	48 hours
pH	P,G		Performed immediately
Conductivity	P,G	Cool 4° C	28 days



Date _____ Page _____ of _____

Shipment No

[illegible]

Science Applications International Corporation ■ 635 West 7th Street Suite 403 Cincinnati OH 45203

White Laboratory Pink Project Manager

Yellow Paper 740

Goldenrod Field Project Manager

GLNPO - OAPJP
Section No.: 4
Revision No.: 1
Date: Jan. 9, 1991
Page: 3 of 4

Figure 4-1. Chain of Custody Record



635 W. 7th Street, Suite 403, Cincinnati, OH 45203

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^a</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 ^b	3510 or 3520/ 8270 or 8100 ^b	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^a

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

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

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

<i>Parameter</i>	<i>Method (a)</i>	<i>Initial Calibration</i>	<i>Calibration Checks</i>	<i>Method Blank</i>	<i>MS/MSD</i>	<i>Triplicate Sample Analysis</i>	<i>QC Sample</i>	<i>Surrogate Spikes</i>
Solids (Total & Volatile)	160.3 160.4	Balance (Yearly)	Balance Each Day	Yes	NA	Yes	Yes	NA
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 (untreated)	Yes	Yes
PAHs	8270 or 8100	5 points	Every 12 Hours	Yes	Yes (treated) MS only (untreated)	NA (treated) Yes (untreated)	Yes	Yes

(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)

<i>Parameter</i>	<i>Method (a)</i>	<i>Initial Calibration</i>	<i>Calibration Checks</i>	<i>Method Blank</i>	<i>MS/MSD</i>	<i>Triplicate Sample Analysis</i>	<i>QC Sample</i>	<i>Surrogate Spikes</i>
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	1		X	1			X							
Volatile Solids	Yes	1		X	1			X							
O & G	Yes	1		X	1			X							
Metals	Yes	1	X	X	1	X		X							
PCBs	Yes	1	X	X	1	X	X		1				1	X	X
PAHs	Yes	1	X	X	1	X	X		1				1	X	X
TOC	Yes	1			1										
Total Cyanide	Yes	1			1										
Total Phosphorous	Yes	1			1										
pH	Yes	1			1										
BOD															
Total Suspended Solids															
Conductivity															

138

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	-	-	-	-	-	-	-
Treated S.	3	-	3	2	3	2	3	2	3	-	3	-	3	3	3	2	3	2	3	-	-	-	-	-	-	-
Water	-	-	-	-	-	-	-	-	-	-	-	-	-	-	3	1	3	1	-	-	-	-	-	-	-	-
Oil	-	-	-	-	-	-	-	-	-	-	-	-	-	-	3	3	3	3	-	-	-	-	-	-	-	-
SET IV																										
Untreated S.	2	-	2	3	2	3	2	3	2	-	2	-	2	3	2	3	2	3	2	-	-	-	-	-	-	-
Treated S.	4	-	4	2	4	2	4	2	4	-	4	-	4	3	4	2	4	2	4	-	-	-	-	-	-	-
Water	-	-	-	-	-	-	-	-	-	-	-	-	-	-	2	1	2	1	-	-	-	-	-	-	-	-
Oil	-	-	-	-	-	-	-	-	-	-	-	-	-	-	2	3	2	3	-	-	-	-	-	-	-	-
SET II																										
Untreated S.	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	-	-	-	-	-	-
Water	1	3	-	-	1	3	1	3	1	3	1	3	1	3	1	2	1	2	1	3	1	3	1	3	1	3
Oil	-	-	-	-	-	-	-	-	-	-	-	-	-	-	1	3	1	3	-	-	-	-	-	-	-	-
SET III																										
Untreated S.	1	-	1	3	1	3	1	3	1	-	1	-	1	3	1	3	1	3	1	-	-	-	-	-	-	-
Treated S.	1	-	1	2	1	2	1	2	1	-	1	-	1	3	1	2	1	2	1	-	-	-	-	-	-	-
Water	-	-	-	-	-	-	-	-	-	-	-	-	-	-	1	2	1	2	-	-	-	-	-	-	-	-
TOTALS																										
Solids	16	5	16	20	16	20	16	20	16	6	16	6	16	24	16	20	16	20	16	5	-	-	-	-	-	-
Water	1	3	-	-	1	3	1	3	1	3	1	3	1	3	7	6	7	6	1	3	1	3	1	3	1	3
Oil	-	-	-	-	-	-	-	-	-	-	-	-	-	-	6	9	6	9	-	-	-	-	-	-	-	-

(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:

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

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_1 = The larger of two observed values
 C_2 = 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_i = 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

\bar{S} = standard deviation

\bar{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.

GLNPO - QAPjP
Section No.: Appendix A
Revision No.: 1
Date: Jan. 9, 1991
Page: 1 of 3

APPENDIX A
TECHNOLOGY SUMMARIES

B.E.S.T.TM Process Description

The B.E.S.T.TM 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.TM 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.TM 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.TM 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

BATTELLE DATA

SAIC-GLNPO (CF #361)
CONVENTIONALS IN UNTREATED SEDIMENT

RETEC

REVISED
2/14/92

MSL Code	Sponsor ID	% Moisture	pH	% Total Volatile Solid	Oil & Grease (mg/kg)	TOC % weight	Total Cyanide (mg/kg)	Total Phosphorus (mg P/kg)
361-37, Rep 1	A-US-RE, Rep 1	38.20%	7.84	7.54	919	2.00%	1.1	1196
361-37, Rep 2	A-US-RE, Rep 2	37.78%	7.91	7.39	1083	NA	2.0	1443
361-37, Rep 3	A-US-RE, Rep 3	30.96%	7.88	7.99	1011	NA	1.5	1217
Method Blank		NA	6.06	0%	1.1	0.009%	0.004 U	0.036

STANDARD REFERENCE MATERIAL

MESS-1 SRM	NA	NA	NA	NA	2.2	NA	NA
In-house Consensus Value *	NA	NA	NA	NA	2.3	NA	NA

REPLICATE ANALYSES

361-37, Rep 1	A-US-RE, Rep 1	38.20%	7.84	7.54	919	2.00%	1.1	1196
361-37, Rep 2	A-US-RE, Rep 2	37.78%	7.91	7.39	1083	NA	2.0	1443
361-37, Rep 3	A-US-RE, Rep 3	30.96%	7.88	7.99	1011	NA	1.5	1217
	RSD%	11%	0%	4%	8%	NA	29%	11%

NA = Not analyzed

U = Below detection limit

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

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

CONVENTIONALS IN TREATED SEDIMENT

MSL Code MDL	Sponsor ID	% Moisture	pH	% Total Volatile Solid	Oil & Grease (mg/kg)	TOC % weight	Total Cyanide (mg/kg)	Total Phosphorus (mg P/kg)
361-41, Rep 1	A-TS-RE, Rep 1	20.46%	8.07	4.30%	563	2.33%	2.1	2293
361-41, Rep 2	A-TS-RE, Rep 2	25.50%	8.07	4.33%	357	2.27%	NA	2071
361-41, Rep 3	A-TS-RE, Rep 3	25.50%	8.14	4.13%	389	2.21%	2.1	2058
Method Blank		NA	6.06	0%	1.1	0.008	0.004 U	0.036

STANDARD REFERENCE MATERIAL

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

MATRIX SPIKE RESULTS

Amount Spiked	NA	NA	NA	NA	NA	88.6	3993
Sample 361-41#	NA	NA	NA	NA	NA	2.1	2141
Sample + Spike	NA	NA	NA	NA	NA	84.3	6368
Amount Recovered	NA	NA	NA	NA	NA	82.2	4227
% Recovery	NA	NA	NA	NA	NA	93%	106%

REPLICATE ANALYSES

361-41, Rep 1		20.46%	8.07	4.30%	563	2.33%	2.1	2293
361-41, Rep 2		25.50%	8.07	4.33%	357	2.27%	NA	2071
361-41, Rep 3		25.50%	8.14	4.13%	389	2.21%	NA	2058
	RSD%	12%	0.5%	3%	25%	3%	NA	6%

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.

SAIC-GLNPO (CF #361)
CONVENTIONALS IN WATER

RETEC

REVISED
3/6/92

MSL Code	Sponsor ID	% Moisture	pH	Total Solids (mg/L)	Total Volatile Solids (mg/L)	Total Suspended Solids (mg/L)	Oil & Grease (mg/L)	TOC (mg/L)	Total Cyanide (mg/L)	Total Phosphorus (mg P/L)	Conductivity (umho/cm)
361-33, Rep 1	A-WR-RE, Rep 1	NA	NA	NA	NA	NA	NA	NA	0.004	NA	NA
361-33, Rep 2	A-WR-RE, Rep 2	NA	NA	NA	NA	NA	NA	NA	0.006	NA	NA
361-33, Rep 3	A-WR-RE, Rep 3	NA	NA	NA	NA	NA	NA	NA	0.004 U	NA	NA
361-35, Rep 1	A-WR-RE, Rep 1	NA	NA	NA	NA	NA	560	427.7	NA	0.439	NA
361-35, Rep 2	A-WR-RE, Rep 2	NA	NA	NA	NA	NA	560	452.5	NA	0.488	NA
361-35, Rep 3	A-WR-RE, Rep 3	NA	NA	NA	NA	NA	574	457.9	NA	0.401	NA
361-36, Rep 1	A-WR-RE, Rep 1	NA	8.15	1600	1400	70	NA	NA	NA	NA	2.49
361-36, Rep 2	A-WR-RE, Rep 2	NA	8.24	1500	1400	85	NA	NA	NA	NA	2.37
361-36, Rep 3	A-WR-RE, Rep 3	NA	8.20	1700	1400	88	NA	NA	NA	NA	2.31
Method Blank		NA	6.06	10 U	10 U	2.0 U	1.0 U	0.84	0.004 U	0.012	0.34

MATRIX SPIKE RESULTS

Amount Spiked	NS	NS	NS	NS	NS	NS	Blank Spike	68.5	40.0	0.207	0.4	NS
Sample A WR-RE #	NS	NS	NS	NS	NS	NS	1.0 U	446.0	0.005	0.443	NS	NS
Sample + Spike	NS	NS	NS	NS	NS	NS	62	511.8	0.203	0.830	NS	NS
Amount Recovered	NS	NS	NS	NS	NS	NS	62	65.8	0.198	0.387	NS	NS
% Recovery	NS	NS	NS	NS	NS	NS	90.5%	164.4%	95.7%	96.8%	NS	NS

REPLICATE ANALYSES

361-33, Rep 1	A-WR-RE, Rep 1	NA	NA	1600	NA	NA	NA	NA	0.004	NA	NA
361-33, Rep 2	A-WR-RE, Rep 2	NA	NA	1500	NA	NA	NA	NA	0.006	NA	NA
361-33, Rep 3	A-WR-RE, Rep 3	NA	NA	1700	NA	NA	NA	NA	0.004 U	NA	NA
	RSD%	NA	NA	6.3%	NA	NA	NA	NA	24.7%	NA	NA
361-35, Rep 1	A-WR-RE, Rep 1	NA	NA	NA	NA	NA	560	427.7	NA	0.439	NA
361-35, Rep 2	A-WR-RE, Rep 2	NA	NA	NA	NA	NA	560	452.5	NA	0.488	NA
361-35, Rep 3	A-WR-RE, Rep 3	NA	NA	NA	NA	NA	574	457.9	NA	0.401	NA
	RSD%	NA	NA	NA	NA	NA	1.4%	3.6%	NA	9.9%	NA
361-35, Rep 1	A-WR-RE, Rep 1	NA	8.15	NA	1400	70	NA	NA	NA	NA	2.49
361-35, Rep 2	A-WR-RE, Rep 2	NA	8.24	NA	1400	85	NA	NA	NA	NA	2.37
361-35, Rep 3	A-WR-RE, Rep 3	NA	8.20	NA	1400	88	NA	NA	NA	NA	2.31
	RSD%	NA	0.6%	NA	0.0%	11.9%	NA	NA	NA	NA	3.8%

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

METALS IN UNTREATED SEDIMENT

(Concentrations in ug/g dry weight)

MSL Code	Sponsor ID	Ag AA	As MF	Ba MF	Cd AA	Cr MF	Cu MF	%Fe MF	Hg CVAA	Mn MF	Ni MF	Pb MF	Se AA	Zn MF
MDL		0.007	2.5	43	0.006	33	5.5	0.26	0.0003	56	7.5	6.2	0.22	7.8
361-37, Rep 1	A-US-RE, Rep 1	0.18	20.9	892	3.02	588	33.9	4.14	1.362	537	49.5	56.7	0.87	228
361-37, Rep 2	A-US-RE, Rep 2	0.19	20.0	906	3.04	624	34.6	4.32	1.337	573	54.9	57.9	0.99	237
361-37, Rep 3	A-US-RE, Rep 3	0.20	21.4	910	3.12	561	32.6	4.32	1.383	566	54.7	60.8	0.87	238
Method Blank		0.020	NA	NA	0.006 U	NA	NA	NA	0.00133	NA	NA	NA	0.22 U	NA

STANDARD REFERENCE MATERIAL

1646 SRM		0.114	11.2	425	0.401	71	16.6	3.33	0.065	349	31.1	29.5	0.87	124
	certified	NC	11.6	NC	0.36	76	18	3.35	0.063	375	32	28.2	NC	138
	value	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	2	NS	NS	NS	NS	1.979	NS	NS	NS	2.73	NS
361-37 #	0.19	NS	NS	3.06	NS	NS	NS	NS	1.361	NS	NS	NS	0.91	NS
361-37 + Spike	2.67	NS	NS	5.00	NS	NS	NS	NS	3.246	NS	NS	NS	5.70	NS
Amount Recovered	2.48	NS	NS	1.94	NS	NS	NS	NS	1.89	NS	NS	NS	4.79	NS
Percent Recovery	124%	NS	NS	97%	NS	NS	NS	NS	95%	NS	NS	NS	175%	NS

REPLICATE ANALYSES

361-37, Rep 1	A-US-RE, Rep 1	0.18	20.9	892	3.02	588	33.9	4.14	1.362	537	49.5	56.7	0.87	228
361-37, Rep 2	A-US-RE, Rep 2	0.19	20.0	906	3.04	624	34.6	4.32	1.337	573	54.9	57.9	0.99	237
361-37, Rep 3	A-US-RE, Rep 3	0.20	21.4	910	3.12	561	32.6	4.32	1.383	566	54.7	60.8	0.87	238
	RSD%	3%	3%	1%	2%	5%	3%	2%	2%	3%	6%	4%	8%	2%

U - Below detection limits

NA - Not analyzed

NC - Not certified

NS - Not spiked

- Mean of triplicated sample

NOTE All metals results are blank corrected

SAIC GLNPO (CF #361)

RETEC

REVISED
2/14/92

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	AA	MF	MF	MF	CVAA	MF	MF	MF	AA	MF
MDL		0.007	2.5	4.3	0.006	3.3	5.5	0.26	0.0003	56	7.5	6.2	0.22	7.8

361-41, Rep 1	A-TS-RE, Rep 1	0.19	15.3	789	2.74	494	47.7	3.85	0.005	520	73.3	75.2	1.60	223
361-41, Rep 2	A-TS-RE, Rep 2	0.18	16.5	811	2.67	514	44.6	3.92	0.006	533	80.7	76.8	1.49	227
361-41, Rep 3	A-TS-RE, Rep 3	0.19	17.6	775	2.67	552	52.0	3.97	0.003	538	77.7	78.9	1.49	243
Method Blank		0.020	NA	NA	0.006 U	NA	NA	NA	0.00133	NA	NA	NA	0.22 U	NA

STANDARD REFERENCE MATERIAL

1646 SRM		0.117	11.2	425	0.41	71	16.6	3.33	0.066	349	31.1	29.5	0.87	124
	certified	NC	11.6	NC	0.36	76	18	3.35	0.063	375	32	28.2	NC	138
	value	NC	11.3	NC	10.07	13	13	10.1	10.012	120	13	11.8	NC	16

MATRIX SPIKE RESULTS

Amount Spiked		2	NS	NS	2	NS	NS	NS	1.978	NS	NS	NS	2.73	NS
361-41 #		0.19	NS	NS	2.69	NS	NS	NS	0.005	NS	NS	NS	1.53	NS
361-41 + Spike		2.54	NS	NS	4.70	NS	NS	NS	1.927	NS	NS	NS	6.22	NS
Amount Recovered		2.35	NS	NS	2.01	NS	NS	NS	1.92	NS	NS	NS	4.69	NS
Percent Recovery		118%	NS	NS	100%	NS	NS	NS	97%	NS	NS	NS	172%	NS

REPLICATE ANALYSES

361-41, Rep 1	A-TS-RE, Rep 1	0.19	15.3	789	2.74	494	47.7	3.85	0.005	520	73.3	75.2	1.60	223
361-41, Rep 2	A-TS-RE, Rep 2	0.18	16.5	811	2.67	514	44.6	3.92	0.006	533	80.7	76.8	1.49	227
361-41, Rep 3	A-TS-RE, Rep 3	0.19	17.6	775	2.67	552	52.0	3.97	0.003	538	77.7	78.9	1.49	243
	RSD%	4%	1%	2%	2%	6%	8%	2%	33%	2%	5%	2%	4%	9%

U = Below detection limits

NA = Not analyzed

NC = Not certified

NS = Not spiked

= Mean of triplicated sample

NOTE All metals results are blank corrected

METALS IN WATER

(Concentrations in ug/L)

MSL Code	Sponsor ID	Ag AA	As AA	Ba ICP/MS	Cd AA	Cr AA	Cu AA	Fe AA	Hg CVAA	Mn AA	Ni AA	Pb AA	Se AA	Zn AA
MDL		0.001	0.03	0.1	0.002	0.15	0.015	0.0	0.0003	22.6	0.051	0.031	1.12	2.528
361-34, Rep 1	A-WR-RE, Rep 1	0.003	7.37	55.2	0.72	17.9	47.6	1786	35.7	481.6	151.7	43.8	1.12 U	201.7
361-34, Rep 2	A-WR-RE, Rep 2	0.001 U	7.53	56.0	0.56	17.9	45.9	1763	34.6	489.1	151.7	43.8	1.12 U	195.4
361-34, Rep 3	A-WR-RE, Rep 3	0.001 U	7.22	55.6	0.54	16.2	45.9	1857	32.3	459.0	136.5	43.8	1.12 U	208.0
Method Blank		0.003	0.03 U	NA	0.01	3.6	0.053	259	0.004	37.6	0.09	0.04	1.12 U	17.4

STANDARD REFERENCE MATERIAL

1643c		2.07	84.62	49.2	12.86	19.0	19.4	106.4	NA	37.2	51.6	36.8	1.12 U	69.0
	certified	2.21	82.1	49.6	12.20	19.0	22.3	106.9	NA	35.1	60.6	35.3	12.7	73.9
	value	±0.30	±1.2	±3.1	±1.0	±0.6	±2.8	±3.0	NA	±2.2	±7.3	±0.9	±0.7	±0.9
1641b		NA	NA	NA	NA	NA	NA	NA	1461.7	NA	NA	NA	NA	NA
	certified	NA	NA	NA	NA	NA	NA	NA	1520.0	NA	NA	NA	NA	NA
	value	NA	NA	NA	NA	NA	NA	NA	±40.0	NA	NA	NA	NA	NA

MATRIX SPIKE RESULTS

Amount Spiked		10	90	42.9	10	89.9	20	1765	48.5	97.0	20	20	42.86	888.8
361-34 #		0.001 U	7.3733	55.6	0.61	17.4	46.5	1802	34.2	476.6	146.6	43.8	1.12 U	201.7
361-34 + Spike		5.61	85.55	94.4	9.00	93.3	65.5	3385	85.7	564.4	163.8	61.3	43.90	1122.0
Amount Recovered		5.61	78.177	38.8	8.39	75.9	19.0	1582.9	51.5	87.8	17.2	17.5	43.90	920.3
Percent Recovery		56%	87%	90%	84%	84%	95%	90%	106%	91%	86%	87%	102%	104%

REPLICATE ANALYSES

361-34, Rep 1	A-WR-RE, Rep 1	0.003	7.37	55.2	0.72	17.9	47.6	1786	35.7	481.6	151.7	43.8	1.12 U	201.7
361-34, Rep 2	A-WR-RE, Rep 2	0.001 U	7.53	56.0	0.56	17.9	45.9	1763	34.6	489.1	151.7	43.8	1.12 U	195.4
361-34, Rep 3	A-WR-RE, Rep 3	0.001 U	7.37	55.6	0.54	16.2	45.9	1857	32.3	459.0	136.5	43.8	1.12 U	208.0
	RSD%	69%	1%	1%	16%	6%	2%	3%	5%	3%	6%	0%	0%	3%

U - Below detection limits

NA - Not analyzed

NC - Not certified

NS - Not spiked

- Mean of triplicated sample

NOTE All metals results are blank corrected

SAIC-GLNPO (CF #361)

RETEC

REVISED
2/14/92

PAH IN UNTREATED SEDIMENT

Low Molecular Weight PAHs (ng/g dry weight)

MSL Code	Sponsor ID	Naphthalene	Acenaphthylene	Acenaphthene	Fluorene	Phenanthrene	Anthracene
361-37, Rep 1	A-US-RE, Rep 1	211 U	254 U	389 U	334 U	1417	248 U
361-37, Rep 2	A-US-RE, Rep 2	222	224 U	344 U	295 U	1341	218 U
361-37, Rep 3	A-US-RE, Rep 3	243	134 U	206 U	247	1376	171
BLANK-8		193 U	232 U	355 U	305 U	206 U	226 U

STANDARD REFERENCE MATERIAL

SRM-NIST1941		871	190 U	291 U	250 U	521	185 U
	certified value	NC	NC	NC	NC	577	202

MATRIX SPIKE RESULTS

Amount Spiked		4673	4673	4673	4673	4673	4673
361-37 #		233	204 U	313 U	292 U	1378	212 U
361-37 + Spike		2919	3509	3548	3827	5020	4048
Amount Recovered		2919	3509	3548	3827	3642	4048
Percent Recovery		62%	75%	76%	82%	78%	87%

REPLICATE ANALYSES

361-37, Rep 1	A-US-RE, Rep 1	211 U	254 U	389 U	334 U	1417	248 U
361-37, Rep 2	A US RE, Rep 2	222	224 U	344 U	295 U	1341	218 U
361-37, Rep 3	A US RE, Rep 3	243	134 U	206 U	247	1376	171
	RSD%	7%	NA	NA	15%	3%	18%

U = Below detection limits
 NC = Not certified
 # = Mean of detected values
 NA = Not applicable

SAIC-GLNPO (CF #361)

RETEC

REVISED

2/14/92

PAH IN UNTREATED SEDIMENT

High Molecular Weight PAHs (ng/g dry weight)

MSL Code	Sponsor ID	Fluoran- thene	Pyrene	Benzo(a)- 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
361-37, Rep 1 A-US-RE, Rep 1		1124	1014	393	585	437	368	384	333	138 U	348 B
361-37, Rep 2 A-US-RE, Rep 2		971	905	324	520	385	265	300	285	122 U	283 B
361-37, Rep 3 A-US-RE, Rep 3		1015	927	356	571	522	410	326	387	88	277 B
Method Blank		167 U	175 U	167 U	166 U	127 U	117 U	145 U	121 U	126 U	118

STANDARD REFERENCE MATERIAL

SRM-NIST1941		1065	994	454	668	771	629	525	558	113	387
certified value		1220	1080	550	NC	780	444	670	569	NC	516

MATRIX SPIKE RESULTS

Amount Spiked		4673	4673	4673	4673	4673	4673	4673	4673	4673	4673
361-37#		1037	949	358	559	448	348	337	335	88	303
361-37 + Spike		5136	4992	4592	4581	4401	4265	4489	4473	4423	3145
Amount Recovered		4099	4043	4234	4022	3953	3917	4152	4138	4335	2842
Percent Recovery		88%	87%	91%	86%	85%	84%	89%	89%	93%	61%

REPLICATE ANALYSES

361-37, Rep 1 A-US-RE, Rep 1		1124	1014	393	585	437	368	384	333	138 U	348
361-37, Rep 2 A-US-RE, Rep 2		971	905	324	520	385	265	300	285	122 U	283
361-37, Rep 3 A-US-RE, Rep 3		1015	927	356	571	522	410	326	387	88	277
RSD%		8%	6%	10%	6%	15%	21%	13%	15%	22%	13%

U = Below detection limits

B = Analyte detected in blank associated with sample

NC = Not certified

= Mean of detected values

SAIC GLNPO (CF #361)

RETEC

REVISED

2/14/92

PAH IN UNTREATED SEDIMENT

MSL Code	Sponsor ID	Surrogate Recovery %		
		D8 Naphthalene	D10 Acenaphthalene	D12 Perylene
361-37, Rep 1	A-US-RE, Rep 1	58%	70%	93%
361-37, Rep 2	A-US-RE, Rep 2	72%	76%	95%
361-37, Rep 3	A-US-RE, Rep 3	74%	80%	100%
Method Blank		97%	91%	71%

STANDARD REFERENCE MATERIAL

SRM-NIST1941		77%	80%	94%
--------------	--	-----	-----	-----

certified value

MATRIX SPIKE RESULTS

Amount Spiked		NA	NA	NA
361-37 #		68%	75%	96%
361-37 + Spike		58%	68%	86%
Amount Recovered		NA	NA	NA
Percent Recovery		NA	NA	NA

REPLICATE ANALYSES

361-37, Rep 1	A-US-RE, Rep 1	58%	70%	93%
361-37, Rep 2	A-US-RE, Rep 2	72%	76%	95%
361-37, Rep 3	A-US-RE, Rep 3	74%	80%	100%
	RSD%	13%	7%	4%

= Mean of detected values.

PAH IN TREATED SEDIMENT

Low Molecular Weight PAHs (ug/kg dry wt)

MSL Code	Sponsor ID	Naphthalene	Acenaphthylene	Acenaphthene	Fluorene	Phenanthrene	Anthracene
361-41	A-TS-RE	485	280 U	429 U	368 U	249 U	273 U
Method Blank		193 U	232 U	355 U	305 U	206 U	226 U

STANDARD REFERENCE MATERIAL

SRM-NIST1941		871	190 U	291 U	250 U	521	185 U
	certified value	NC	NC	NC	NC	577	202

MATRIX SPIKE RESULTS

Amount Spiked	4167	4167	4167	4167	4167	4167
361-41	485	280 U	429 U	368 U	249 U	273 U
361-41 + Spike	2651	2514	3056	3050	3286	3121
Amount Recovered	2166	2514	3056	3050	3286	3121
Percent Recovery	52%	60%	73%	73%	79%	75%
Amount Spiked	3676	3676	3676	3676	3676	3676
361-41	485	280 U	429 U	368 U	249 U	273 U
361-41 + Spike DUP	2158	1930	2488	2533	2808	2624
Amount Recovered	1673	1930	2488	2533	2808	2624
Percent Recovery	46%	53%	68%	69%	76%	71%

U = Below detection limits

NC = Not certified

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

SAIC-GLNPO (CF #361)

RETEC

REVISED
3/6/92

PAH IN TREATED SEDIMENT

High Molecular Weight PAHs (ng/g dry weight)

MSL Code	Sponsor ID	Fluoran- thene	Pyrene	Benzo(a)- 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
361-41	A-TS-RE	202 U	211 U	201 U	201 U	153 U	142 U	175 U	146 U	152 U	97 B
Method Blank		167 U	175 U	167 U	166 U	127 U	117 U	145 U	121 U	126 U	118

STANDARD REFERENCE MATERIAL

SRM-NIST1941		1065	994	454	668	771	629	525	558	113	387
certified value		1220	1080	550	NC	780	444	670	569	NC	516

MATRIX SPIKE RESULTS

Amount Spiked		4167	4167	4167	4167	4167	4167	4167	4167	4167	4167
361-41		202 U	211 U	201 U	201 U	153 U	142 U	175 U	146 U	152 U	97 B
361-41 + Spike		3418	3413	3270	3228	3085	3076	2819	2370	2761	1683
Amount Recovered		3418	3413	3270	3228	3085	3076	2819	2370	2761	1586
Percent Recovery		82%	82%	78%	77%	74%	74%	68%	57%	66%	38% *
Amount Spiked		3676	3676	3676	3676	3676	3676	3676	3676	3676	3676
361-41		202 U	211 U	201 U	201 U	153 U	142 U	175 U	146 U	152 U	97 B
361-41 + Spike DUP		2905	2875	2693	2740	2564	2568	2219	1715	2061	1278
Amount Recovered		2905	2875	2693	2740	2564	1568	2219	1715	2061	1278
Percent Recovery		79%	78%	73%	75%	70%	43%	60%	47%	56%	35% *

B - Analyte detected in blank associated with sample

U - Below detection limits

NC - Not certified

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

PAH IN TREATED SEDIMENT

MSL Code	Sponsor ID	Surrogate Recovery %		
		D8 Naphthalene	D10 Acenaphthalene	D12 Perylene
361-41	A-TS-RE	74%	78%	61%
Method Blank		97%	91%	71%

STANDARD REFERENCE MATERIAL

SRM-NIST1941	certified value	77%	80%	94%
--------------	-----------------	-----	-----	-----

MATRIX SPIKE RESULTS

Amount Spiked	NA	NA	NA
361-41	74%	78%	61%
361-41 + Spike	61%	69%	63%
Amount Recovered	NA	NA	NA
Percent Recovery	NA	NA	NA
Amount Spiked	NA	NA	NA
361-41	74%	78%	61%
361-41 + Spike DUP	57%	65%	58%
Amount Recovered	NA	NA	NA
Percent Recovery	NA	NA	NA

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

NA - Not applicable

SAIC GLNPO (CF #361)

RETEC

REVISED
2/14/92

PAH IN WATER

Low Molecular Weight PAHs (ng/L)

MSL Code	Sponsor ID	Naphthalene	Acenaphthylene	Acenaphthene	Fluorene	Phenanthrene	Anthracene
361-40	A-WR-RE	608612	4156	52962	81969	199962	26748
Method Blank		266 U	320 U	491 U	421 U	285 U	312 U

MATRIX SPIKE RESULTS

Amount Spiked	4673	4673	4673	4673	4673	4673
361-40	608612	4156	52962	81969	199962	26748
361-40 + Spike	720383	7029	56095	84485	199620	30162
Amount Recovered	111771	2873	3133	2516	-342	3414
Percent Recovery	2392% *	61%	67%	54%	-7% *	73%
Amount Spiked	4808	4808	4808	4808	4808	4808
361-40	608612	4156	52962	81969	199962	26748
361-40 + Spike DUP	556144	6478	46387	70724	166182	26103
Amount Recovered	-52468	2322	-6575	-11245	-33780	-645
Percent Recovery	-1091% *	48%	-137% *	-234% *	-703% *	-13% *

U = Below detection limits.

NC = Not certified

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

SAIC-GLNPO (CF #361)

RETEC

REVISED

2/14/02

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(a)-pyrene	Indeno (1,2,3,c,d)-pyrene	Dibenzo(a,h)-anthracene	Benzo(g,h,i)-perylene
361-40	A-WR-RE	12973	72041	18027	43493	7909	907 U	10220	1220	1849	5509 B
Method Blank		231 U	242 U	230 U	230 U	175 U	162 U	200 U	167 U	174 U	131

MATRIX SPIKE RESULTS

Amount Spiked	4673	4673	4673	4673	4673	4673	4673	4673	4673	4673	4673
361-40	12973	72041	18027	43493	7909	907 U	10220	1220	1849	5509	5509
361-40 + Spike	15517	72862	20566	44535	8383	4315	13057	3691	4863	7269	7269
Amount Recovered	2544	821	2539	1042	474	3408	2837	2471	3014	1760	1760
Percent Recovery	54%	18%	54%	22% *	10% *	73%	61%	53%	64%	38% *	38% *
Amount Spiked	4808	4808	4808	4808	4808	4808	4808	4808	4808	4808	4808
361-40	12973	72041	18027	43493	7909	907 U	10220	1220	1849	5509	5509
361-40 + Spike DUP	16432	61907	17819	38457	7542	4136	11561	3721	4820	6706	6706
Amount Recovered	3459	-10134	-208	-5036	-367	3229	1341	2501	2971	1197	1197
Percent Recovery	72%	-211% *	-4% *	-105% *	-8% *	67%	28% *	52%	62%	25% *	25% *

U = Below detection limits

B = Analyte detected in blank associated with sample.

NC = Not certified

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

PAH IN WATER

MSL Code	Sponsor ID	Surrogate Recovery %		
		D8 Naphthalene	D10 Acenaphthalene	D12 Perylene
361-40	A-WR-RE	29% *	61%	51%
Method Blank		79%	77%	66%

MATRIX SPIKE RESULTS

Amount Spiked	NA	NA	NA
361-40	29% *	61%	51%
361-40 + Spike	57%	63%	53%
Amount Recovered	NA	NA	NA
Percent Recovery	NA	NA	NA
Amount Spiked	NA	NA	NA
361-40	29% *	61%	51%
361-40 + Spike DUP	57%	64%	56%
Amount Recovered	NA	NA	NA
Percent Recovery	NA	NA	NA

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

NA = Not applicable

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

Concentrations in ug/kg dry weight

RETEC

SAIC-GLNPO (CF #361)

2/14/92

% Surrogate
Recovery

MSL Code	Sponsor ID	Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260	Tetrachloro- m-Xylene	OTHER (1)
361-37, Rep 1	A-US-RE, Rep 1	200 U	14400	100 U	100 U	100.7%	25000
361-37, Rep 2	A-US-RE, Rep 2	200 U	13900	100 U	100 U	108.8%	24000
361-37, Rep 3	A-US-RE, Rep 3	200 U	15600	100 U	100 U	113.1%	20000
Blank-8		200 U	200 U	100 U	100 U	102.1%	NA

STANDARD REFERENCE MATERIAL

SRM-7 (HS-2)		200 U	200 U	221	100 U	84.7%	NA
	certified value	NC	NC	111	NC	NC	NA

MATRIX SPIKE RESULTS

Amount Spiked	NS	NS	4673	NS	NA	NA
361-37 #	NS	NS	100 U	NS	107.5%	NA
361-37 + Spike	NS	NS	NA(2)	NS	102.6%	NA
Amount Recovered	NS	NS	NA(2)	NS	NA	NA
Percent Recovery	NS	NS	NA(2)	NS	NA	NA

REPLICATE ANALYSES

361-37, Rep 1	A-US-RE, Rep 1	200 U	200 U	14400	100 U	100.7%	NA
361-37, Rep 2	A-US-RE, Rep 2	200 U	200 U	13900	100 U	108.8%	NA
361-37, Rep 3	A-US-RE, Rep 3	200 U	200 U	15600	100 U	113.1%	NA
	RSD%	0%	0%	6%	0%	6%	NA

(1) Numerous early eluting large peaks not corresponding to Aroclor pattern;
quantities estimated based on average Aroclor response factor.

(2) Residual peaks from presence of Aroclor 1248 masked 1254 spike.

U = Below detection limits

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

NC = Not certified.

= Mean of replicated sample.

NS = Not spiked NA = Not applicable.

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

Concentrations in ug/kg dry weight

RETEC

SAIC-GLNPO (CF #361)

3/5/92

% Surrogate
Recovery

MSL Code	Sponsor ID	Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260	Tetrachloro- m-Xylene	OTHER (1)
361-41	A-TS-RE	200 U	200 U	100 U	100 U	91.4%	NA
Blank-8		200 U	200 U	100 U	100 U	102.1%	NA
STANDARD REFERENCE MATERIAL							
SRM-7 (HS-2)		200 U	200 U	221	100 U	84.7%	NA
	certified value	NC	NC	111	NC	NC	NA
MATRIX SPIKE RESULTS							
Amount Spiked		NS	NS	4167	NS	NA	NA
361-41		NS	NS	100 U	NS	91.4%	NA
361-41+ Spike		NS	NS	3232	NS	81.3%	NA
Amount Recovered		NS	NS	3232	NS	NA	NA
Percent Recovery		NS	NS	78%	NS	NA	NA
Amount Spiked		NS	NS	3676	NS	NA	NA
361-41 DUP		NS	NS	100 U	NS	91.4%	NA
361-41 + Spike DUP		NS	NS	2789	NS	81.8%	NA
Amount Recovered		NS	NS	2789	NS	NA	NA
Percent Recovery		NS	NS	76%	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)**PCBs IN WATER SAMPLES**

Concentrations in ug/L

RETEC
SAIC-GLNPO (CF #361)

3/5/92

% Surrogate
Recovery

MSL Code	Sponsor ID	Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260	Tetrachloro- m-Xylene	OTHER (1)
361-40	A-WR-RE	5 U	5 U	5 U	5 U	NA(2)	10 to 20
Blank-9		0.2 U	0.2 U	0.1 U	0.1 U	20.2%	NA

MATRIX SPIKE RESULTS

Amount Spiked	NS	NS	5	NS	NA	NA
361-40	NS	NS	5 U	NS	NA	NA
361-40 + Spike	NS	NS	NA(3)	NS	NA	NA
Amount Recovered	NS	NS	NA(3)	NS	NA	NA
Percent Recovery	NS	NS	NA(3)	NS	NA	NA
Amount Spiked	NS	NS	5	NS	NA	NA
361-40 DUP	NS	NS	5 U	NS	NA	NA
361-40 + Spike	NS	NS	NA(3)	NS	NA	NA
Amount Recovered	NS	NS	NA(3)	NS	NA	NA
Percent Recovery	NS	NS	NA(3)	NS	NA	NA

(1) Numerous early eluting large peaks not corresponding to Aroclor pattern;
quantities estimated based on average Aroclor response factor.

(2) Not available; peaks were not quantified due to coeluting unidentified peaks.

(3) Spikes were not recovered due to high background interference.

U = Below detection limits.

NS = Not spiked. NA = Not applicable.

RE-PROCESSED RESULTS (1/92)

RETEC

3/5/92

PCBs IN OIL SAMPLES

SAIC-GLNPO (CF #361)

% Surrogate

Concentrations in ug/L

Recovery

MSL Code	Sponsor ID	Sample Density (g/ml)	Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260	Tetrachloro-m-Xylene	OTHER (1)
361-38, Rep 1	A-OR-RE, Rep 1	0.9762	2000 U	2000 U	1000 U	1000 U	107.5%	~150000
361-38, Rep 2	A-OR-RE, Rep 2	0.9762	2000 U	2000 U	1000 U	1000 U	93.0%	NA
361-38, Rep 3	A-OR-RE, Rep 3	0.9762	2000 U	2000 U	1000 U	1000 U	99.5%	NA
361-39	A-OR-RE3	0.8985	2000 U	2000 U	1000 U	1000 U	96.3%	~100000
Blank-10			2000 U	2000 U	1000 U	1000 U	34.8%	NA

OIL CONCENTRATIONS ON % OIL BASIS% Surrogate
Recovery

Concentrations in ug/kg oil

MSL Code	Sponsor ID	% Oil (%)	Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260	Tetrachloro-m-Xylene	OTHER (1)
361-38, Rep 1	A-OR-RE, Rep 1	33.97	6030 U	6030 U	3015 U	3015 U	107.5%	~150000
361-38, Rep 2	A-OR-RE, Rep 2	33.97	6030 U	6030 U	3015 U	3015 U	93.0%	NA
361-38, Rep 3	A-OR-RE, Rep 3	33.97	6030 U	6030 U	3015 U	3015 U	99.5%	NA
361-39	A-OR-RE3	48.61	4579 U	4579 U	2289 U	2289 U	96.3%	~100000

MATRIX SPIKE RESULTS

Amount Spiked	NS	NS	50000	NS	NA	NA
361-38 #	NS	NS	1000 U	NS	100.0%	NA
361-38 + Spike	NS	NS	36700	NS	107.5%	NA
Amount Recovered	NS	NS	36700	NS	NA	NA
Percent Recovery	NS	NS	73%	NS	NA	NA

REPLICATE ANALYSES

361-38, Rep 1	A-OR-RE, Rep 1	2000 U	2000 U	1000 U	1000 U	107.5%	NA
361-38, Rep 2	A-OR-RE, Rep 2	2000 U	2000 U	1000 U	1000 U	93.0%	NA
361-38, Rep 3	A-OR-RE, Rep 3	2000 U	2000 U	1000 U	1000 U	99.5%	NA
		0%	0%	0%	0%	7%	NA

(1) Numerous early eluting large peaks not corresponding to Aroclor pattern;
quantities estimated based on average Aroclor response factor.

U = Below detection limits.

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

NA = Not applicable. NS = Not spiked.

PAH IN OIL

Low Molecular Weight PAHs (ng/ml)

MSL Code	Sponsor ID	Sample Density (g/ml)	Naphthalene	Acenaphthylene	Acenaphthene	Fluorene	Phenanthrene	Anthracene
361-38, Rep 1	A-OR-RE, Rep 1	0.9762	963952	6302	73327	112104	241457	35429
361-38, Rep 2	A-OR-RE, Rep 2	0.9762	1009533	6673	75953	110830	253511	37459
361-38, Rep 3	A-OR-RE, Rep 3	0.9762	1131802	7136	83482	122398	277739	40202
361-39	A-OR-RE3	0.8985	2396562	10398	141580	195290	386564	54906
Method Blank			3121 U	3753 U	5755 U	4940 U	3339 U	3659 U

OIL CONCENTRATIONS ON % OIL BASIS

Low Molecular Weight PAHs (ug/kg oil)

MSL Code	Sponsor ID	% Oil (%)	Naphthalene	Acenaphthylene	Acenaphthene	Fluorene	Phenanthrene	Anthracene
361-38, Rep 1	A-OR-RE, Rep 1	33.97	2921067	19097	222203	339709	731688	107361
361-38, Rep 2	A-OR-RE, Rep 2	33.97	3059191	20221	230161	335848	768215	113512
361-38, Rep 3	A-OR-RE, Rep 3	33.97	3429703	21624	252976	370903	841633	121824
361-39	A-OR-RE3	48.61	5446732	23632	321773	443841	878555	124786

MATRIX SPIKE RESULTS

Amount Spiked	50000	50000	50000	50000	50000	50000
361-38 #	1035096	6704	77587	115111	257569	37697
361-38 + Spike	1136012	48182	125749	167187	329560	87079
Amount Recovered	100916	41478	48162	52076	71991	49382
Percent Recovery	202% *	83%	96%	104%	144% *	99%

REPLICATE ANALYSES

361-38, Rep 1	A-OR-RE, Rep 1	963952	6302	73327	112104	241457	35429
361-38, Rep 2	A-OR-RE, Rep 2	1009533	6673	75953	110830	253511	37459
361-38, Rep 3	A-OR-RE, Rep 3	1131802	7136	83482	122398	277739	40202
	RSD%	8%	6%	7%	6%	7%	6%

U = Below detection limits

= Mean of replicated sample

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

PAH IN OIL

High Molecular Weight PAHs (ng/ml)

MSL Code	Sponsor ID	Sample Density (g/ml)	Fluoranthene	Pyrene	Benzo(a)-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
361-38, Rep 1	A-OR-RE, Rep 1	0.9762	13676	79347	20217	46395	8827	934 U	12047	1344	1937	6292
361-38, Rep 2	A-OR-RE, Rep 2	0.9762	14358	84016	21377	48052	8658	1005 U	11877	1501	2060	6855
361-38, Rep 3	A-OR-RE, Rep 3	0.9762	15563	90336	23051	51956	9431	1621 U	13334	1672 U	2511	7574
361-39	A-OR-RE3	0.8985	16406	102881	23944	50444	7090	2280	13137	1798 U	2000	7685
Method Blank			2710 U	2833 U	2702 U	2696 U	2054 U	1900 U	2349 U	1960 U	2039 U	1254 U

High Molecular Weight PAHs (ug/kg oil)

MSL Code	Sponsor ID	% Oil (%)	Fluoranthene	Pyrene	Benzo(a)-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
361-38, Rep 1	A-OR-RE, Rep 1	33.97	41442	240445	61264	140591	26748	2830	36508	4073	5870	19067
361-38, Rep 2	A-OR-RE, Rep 2	33.97	43509	254594	64779	145612	26238	3045	35991	4548	6242	20773
361-38, Rep 3	A-OR-RE, Rep 3	33.97	47161	273745	69852	157442	28579	4912	40406	5067	7609	22952
361-39	A-OR-RE3	48.61	37286	233820	54418	114645	16114	5182	29857	4086	4545	17466

MATRIX SPIKE RESULTS

Amount Spiked	50000	50000	50000	50000	50000	50000	50000	50000	50000	50000	50000	50000
361-38 #	14532	84568	21548	48801	8972	1187 U	12419	1423	2169	6907		
361-38 + Spike	67624	139367	71401	99789	50897	45777	58889	47987	51116	40012		
Amount Recovered	53092	54801	49853	50968	41925	45777	46470	46564	48947	33105		
Percent Recovery	106%	110%	100%	102%	84%	92%	93%	93%	98%	66%		

REPLICATE ANALYSES

361-38, Rep 1	A-OR-RE, Rep 1	13676	79347	20217	46395	8827	934 U	12047	1344	1937	6292
361-38, Rep 2	A-OR-RE, Rep 2	14358	84016	21377	48052	8658	1005	11877	1501	2060	6855
361-38, Rep 3	A-OR-RE, Rep 3	15563	90336	23051	51956	9431	1621 U	13334	1672 U	2511	7574
	RSD%	7%	7%	7%	8%	5%	32%	6%	11%	14%	9%

U = Below detection limits

= Mean of replicated sample.

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

PAH IN OIL

MSL Code	Sponsor ID	Surrogate Recovery %		
		D8 Naphthalene	D10 Acenaphthalene	D12 Perylene
361-38, Rep 1	A-OR-RE, Rep 1	72%	81%	94%
361-38, Rep 2	A-OR-RE, Rep 2	61%	68%	80%
361-38, Rep 3	A-OR-RE, Rep 3	72%	80%	89%
361-39	A-OR-RE3	71%	109%	90%
Method Blank		23% *	26% *	72%

OIL CONCENTRATIONS ON % OIL BASIS

MSL Code	Sponsor ID	Surrogate Recovery %		
		D8 Naphthalene	D10 Acenaphthalene	D12 Perylene
361-38, Rep 1	A-OR-RE, Rep 1	72%	81%	94%
361-38, Rep 2	A-OR-RE, Rep 2	61%	68%	80%
361-38, Rep 3	A-OR-RE, Rep 3	72%	80%	89%
361-39	A-OR-RE3	71%	109%	90%

MATRIX SPIKE RESULTS

Amount Spiked	NA	NA	NA
361-38 #	69%	85%	88%
361-38 + Spike	75%	86%	92%
Amount Recovered	NA	NA	NA
Percent Recovery	NA	NA	NA

REPLICATE ANALYSES

361-38, Rep 1	A-OR-RE, Rep 1	72%	81%	94%
361-38, Rep 2	A-OR-RE, Rep 2	61%	68%	80%
361-38, Rep 3	A-OR-RE, Rep 3	72%	80%	89%
	RSD%	9%	9%	8%

= Mean of replicated sample

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

NA = Not applicable

SAIC-GLNPO (CF #361)

RETEC

REVISED
3/6/92

PAH IN TREATED SEDIMENT

High Molecular Weight PAHs (ng/g dry weight)

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	Dibenzo(a,h)-anthracene	Benzo(g,h,i)-perylene
361-41	A-TS-RE	202 U	211 U	201 U	201 U	153 U	142 U	175 U	146 U	152 U	97 B
Method Blank		167 U	175 U	167 U	166 U	127 U	117 U	145 U	121 U	126 U	118

STANDARD REFERENCE MATERIAL

SRM-NIST1941		1065	994	454	668	771	629	525	558	113	387
certified value		1220	1080	550	NC	780	444	670	569	NC	516

MATRIX SPIKE RESULTS

Amount Spiked	4167	4167	4167	4167	4167	4167	4167	4167	4167	4167	4167
361-41	202 U	211 U	201 U	201 U	153 U	142 U	175 U	146 U	152 U	97 B	
361-41 + Spike	3418	3413	3270	3228	3085	3076	2819	2370	2761	1683	
Amount Recovered	3418	3413	3270	3228	3085	3076	2819	2370	2761	1586	
Percent Recovery	82%	82%	78%	77%	74%	74%	68%	57%	66%	38% *	
Amount Spiked	3676	3676	3676	3676	3676	3676	3676	3676	3676	3676	3676
361-41	202 U	211 U	201 U	201 U	153 U	142 U	175 U	146 U	152 U	97 B	
361-41 + Spike DUP	2905	2875	2693	2740	2564	2568	2219	1715	2061	1278	
Amount Recovered	2905	2875	2693	2740	2564	1568	2219	1715	2061	1278	
Percent Recovery	79%	78%	73%	75%	70%	43%	60%	47%	56%	35% *	

B - Analyte detected in blank associated with sample.

U - Below detection limits

NC - Not certified

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