Ponce Harbor Section 103 Sediment Characterization

Prepared for

Infrastructure Financing Authority
235 Arterial Hostos Avenue, Suite 1601
San Juan, Puerto Rico 00918-1454

Prepared by

PPB Environmental Laboratories, Inc.
6821 S.W. Archer Road
Gainesville, Florida 32608

September 2003
02-7333-01

Purchase Order No. C-151

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Executive Summary

A dredging project has been proposed that would deepen and widen the entrance channel and turning basin for Ponce Harbor, Puerto Rico. Before ocean disposal of dredged sediments could occur, harbor sediments needed to be tested and compared to reference sediments to determine if they contained elevated concentrations of potentially toxic chemicals and/or displayed unacceptable levels of toxicity as measured by bioassays and bioaccumulation of chemicals.

This report presents field data and test results from sediment collection activities that occurred in March 2003. The purpose of this sampling effort was to provide information required by decision makers for the evaluation of sediment disposal options, including placement in the Ponce Ocean Dredged Material Disposal Site (ODMDS).

Prior to initiating field work, a Work and Quality Assurance Plan (Work/QA Plan) was developed and approved by the US Army Corps of Engineers, Jacksonville District, and the US Environmental Protection Agency, Region II. The sampling and laboratory test procedures used followed those specified in the Work/QA Plan and while some problems were encountered, all were successfully resolved. Two problems were notable: 1) One of the two reference sediment locations yielded no sediment and all reference sediment was collected from one location. 2) Elevated levels of copper (mean concentrations of 55 [harbor] and 67 µg/g [reference], wet weight basis) were found to bioaccumulate in the sand worm, *Nereis*, and these levels were attributed to copper tubing present in the newly purchased test equipment.

Although some problems were encountered, all data were judged acceptable for project needs.

Sediment Chemistry Comparison

The reference sediment was found to contain a finer material than the harbor sediment with approximately 20 percent less silt and approximately 30 percent more clay.

For all metals, the concentrations found in the harbor composite were within a factor of two of the reference composite values. Lead, mercury, and selenium concentrations were greater in reference sediment that in the harbor. Silver yielded the only undetectable concentration (i.e., for the reference composite) and varied greatly in the triplicate harbor sediment ranging between 0.0704 and 0.740 µg/g, dry weight basis.

Only three DDT-related compounds and one PCB congener were detected in sediment from either the reference station or the harbor. Reference station sediment contained the only quantifiable concentrations of these compounds. PCB-153 was detected in harbor sediment only and at levels below the method detection limit.

Unlike pesticides and PCB congeners, every PAH analyte was measured or detected in every sediment composite and replicate sample tested with values ranging from 0.12 to 9.22 ng/g, dry weight basis. Sediment from the reference station contained a higher concentration of each PAH compound than found in the harbor sediment; typically two to five times higher.

Elutriation Chemistry

Elutriation of harbor sediment resulted in chemical concentrations increasing or decreasing by a factor of three or less for all metals except arsenic which increased approximately 10 fold

relative to the site (harbor) water. No pesticides or PCB congeners were detected in any water or elutriate sample.

Elutriate Bioassay Survivorship

Mysidopsis bahia survivorship was 93 percent in the laboratory control seawater and 97 percent in the 100 percent laboratory control sediment elutriate concentration. Survivorship of M. bahia in the harbor sediment sample was 97 percent, 95 percent, and 87 percent in the 10 percent, 50 percent, and 100 percent concentrations, respectively.

Menidia beryllina survivorship was 98 percent in the laboratory control seawater and 95 percent in the 100 percent laboratory control sediment elutriate concentration. Survivorship of *M. beryllina* in the harbor sediment sample elutriate was 94 percent, 85 percent, and 72 percent in the 10 percent, 50 percent, and 100 percent concentrations, respectively.

LC50s for both *M. bahia* and *M. beryllina* were greater than 100 percent.

Mytilus edulis development (expressed as percentage of embryos resulting in live larvae with complete shell development) was 75 percent in the laboratory control seawater, with acceptable survivorship. Development in the laboratory control sediment elutriate was 72 percent, 71 percent, and 69 percent in the 10 percent, 50 percent, and 100 percent concentrations, respectively. Development of *M. edulis* in the harbor sediment elutriate was 72 percent, 70 percent, and 60 percent in the 10 percent, 50 percent, and 100 percent concentrations, respectively. EC50s were estimated as >100 percent.

Sediment Bioassay Survivorship

Mysidopsis bahia survivorship was 96 percent in the laboratory control sediment and 92 percent in the reference sediment, RS-PH03. Survivorship of *M. bahia* in the harbor composite sediment sample was 96 percent. Survival of *M. bahia* in the reference sediment was not significantly different (p=0.05) from survival in the harbor sediment sample.

Leptocheirus plumulosus survivorship was 95 percent in the laboratory control sediment and 95 percent in the reference sediment. Survivorship of *L. plumulosus* in the harbor composite sediment sample was 84 percent. Survival of *L. plumulosus* in the reference sediment was not statistically different from the harbor sediment sample.

Bioaccumulation Survivorship

Macoma nasuta survivorship in the laboratory control sediment, reference sediment, and harbor composite sediment sample was 92 percent, 94 percent, and 87 percent, respectively.

For *Nereis virens*, survivorship in the control, reference, and harbor sediments was 95 percent, 95 percent, and 88 percent, respectively.

Bioaccumulated Chemicals

Because the exposed organisms were not tested for pesticides or PCB congeners, the only applicable Food and Drug Administration action level for food was methyl mercury. The action level for methyl mercury is one part per million (ppm), wet weight basis, which is approximately 20 to 150 times higher than total mercury concentrations found in test organisms exposed to harbor and reference sediments.

Tissues were analyzed for metals and PAHs. Using one-sided t-test with a significance level of 0.5, statistical comparisons were made of chemical accumulation in organisms exposed to harbor sediment relative to those exposed to reference station sediment. The harbor sediment exposed organisms were found not to have significantly elevated concentrations of any chemical relative to the reference sediment exposed organisms.

1 Introduction

This report presents information regarding sediment proposed for dredging from Ponce Harbor under the oversight of the Puerto Rico Infrastructure Financing Authority (AFI). The area was evaluated for 103 certification in accordance with the 1991 "Green Book" ("Evaluation of Dredged Material Proposed for Ocean Disposal - Testing Manual", i.e., "The Green Book") with guidance from the US Environmental Protection Agency (USEPA) Region II draft document "Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal", dated 18 December 1992 with undated updates attached (Region II RTM). This evaluation included chemical analysis of sediments, waters, and elutriates; water column and sediment bioassays; and bioaccumulation studies.

The current ocean disposal evaluation of Ponce Harbor considered potential dredge material (DM) from widening the channel and turning basin and deepening them to a depth of 53 feet MLW plus 2 feet allowable overdraft. The planned side-slopes for the project are angled at approximately 15 $^{\circ}$ or at a rise-to-run of one to 3.8. Approximately 1.4 million cubic yards of material is proposed for dredging from the harbor.

The Ponce Ocean Dredged Material Disposal Site (ODMDS) is an area of approximately one square nautical mile that is located approximately six nautical miles offshore from the island of Puerto Rico near Ponce Harbor. It presently is undergoing a characterization process from which the US Army Corps of Engineers and the US Environmental Protection Agency will develop a Site Management and Monitoring Plan which is required before disposal activities can begin.

Figure 1 shows the existing channel and the footprint for the proposed dredging. A line on the figure approximates the limit of dredging where the existing depth equals 55 feet MLW; consequently, dredging will not be needed south of the line.

2 Materials and Methods

Before sampling procedures could be developed, a list of chemical constituents and physical parameters of interest was developed. A Tier I investigation was performed to identify site specific chemicals of interest and these were merged with the list of chemical analytes and physical parameters suggested in the Regional Implementation Manual (USACE and USEPA 1992), with some modification based on guidance by EPA Region II and the Jacksonville District Corps. The final list includes regulated as well as other common, unregulated contaminants. Correspondence pertinent to the development of the chemicals of interest and the Tier I Report appear in Appendix A.

After identifying the chemicals and physical parameters of interest, a Work and Quality Assurance Plan was developed by PPB and approved by EPA Region II. This document, "Ponce ODMDS Section 103 Sediment Evaluation Work and Quality Assurance Plan" (PPB 2002) appears as Appendix B.

2.1 Physical Parameters and Chemicals of Interest and Toxicity Testing

The following physical parameters, chemical constituents, and analytical groups were identified as important for this project. Individual organic compounds are listed in Section 2.3 along with the analytical methods used, target and achieved detection limits.

PHYSICAL PARAMETERS

Grain Size Distribution
Percent Solids

CHEMICAL CONSITUENTS

Metals

- Aluminum (sediment only)
- Arsenic
- Cadmium
- Chromium
- Copper
- Iron (sediment only)
- Lead
- Mercury
- Nickel
- Silver
- Selenium
- Zinc

Total Organic Carbon (TOC)

Chlorinated Hydrocarbon Pesticides

Polychlorinated Biphenyls (PCBs) Congeners

Polynuclear Aromatic Hydrocarbons (PAHs)

In addition to the chemical and physical testing, toxicity testing also was proposed as described in the "Green Book" and "Region II RTM". A complete description of the bioassay and bioaccumulation procedures, associated quality assurance and control information, and statistical methods is provided in Section 4 of Appendix B. Because of the unavailability of *Ampelisca abdita*, *Leptocheirus plumulosu* was used in the benthic bioassays. Brief toxicity testing summaries follow:

TOXICITY TESTING

Organism	Test
Water Column/Elutriate Bioassays	
Mysidopsis bahia	96 hours, static, non-renewal
Mytilus edulis	48 hours, static, non-renewal
Menidia beryllina	96 hours, static, non-renewal
Benthic Bioassays	
Mysidopsis bahia	10-day, static renewal (twice/day)
Leptocheirus plumulosus	10-day, static renewal (twice/day)
Bioaccumulation	
Nereis virens	28 days, flow through - 6 volume changes/day
Macoma nasuta	28 days, flow through - 6 volume changes/day

After water and sediment test results had been reported, it was determined that metals and PAHs were the chemicals of interest in tissues.

2.2 Field Methods and Procedures

Field and laboratory procedures for sample and data collection and subsequent physical and chemical analyses are described in detail in Sections 2 and 3 of Appendix B. The following text summarizes the methods and procedures used.

2.2.1 Field Equipment

Equipment shipped to the Ponce Harbor area in preparation for the field work was placed in containers that protected the items from damage due to rough handling and wet weather conditions. To the extent practical, critical items were duplicated so that failure of any one item would not stop the field work. Similarly, spare parts and replacement items were included as appropriate to prevent avoidable down time. When practical, sampling equipment was pre-cleaned before shipment to the field if it was ensured that integrity of such equipment would not be compromised during shipment and storage. All sample containers were supplied by the laboratory and the integrity of all containers was maintained during shipment to and from the Ponce area and while in storage awaiting use.

2.2.2 Decontamination and Clean Area Procedures

All equipment surfaces with the potential to contact sample material were decontaminated, e.g., stainless steel spoons and bowls. Such equipment was cleaned using a water/Liqui-nox solution, followed by a tap water rinse, deionized water rinse, isopropanol rinse, and air dried. If not immediately used after cleaning, decontaminated equipment was wrapped in food-grade aluminum foil with the shiny side facing/contacting the equipment. Field personnel wore Nitrile gloves that were non-powdered, talc- and phthalate-free, and were changed between sampling stations, when cleaning equipment, and when they became "dirty" or were ripped or torn.

After welding equipment and safety railings to the deck, the deck area where sampling was to occur was scrubbed with Liqui-nox solution or other appropriate non-contaminating cleaner to remove petroleum residues, loose debris, and any other materials that might contaminate collected sediment. It was the opinion of the field team leader that no additional protective measures were needed, e.g., placing polyethylene or polypropylene sheeting on the deck. A piece of cleaned wood was used to protect the vibracore nose-cone, i.e., the cutting edge, from scraping paint from the deck during retrieval.

Care was taken to prevent wash water that potentially contained oil or petroleum compounds from entering the surface water. Sediment debris was removed from the sample collection area before departing each station.

Hydraulic and petroleum-fueled equipment were maintained in an area away from the vibracoring area and any drips or leaks of hydraulic fluids, fuels, or lubricants were cleaned up immediately. Gasoline- and diesel-fueled equipment was positioned downwind to minimize the potential for sample contamination.

2.2.3 Sediment Sample Collection and Handling

All samples were collected using appropriately cleaned, non-contaminating equipment (e.g., constructed of stainless steel or coated with Teflon®) as required for the analyses of interest, with the exception of vibracore tubes that were constructed of ridged non-stainless steel tubes with Layflat food-grade polyethylene sleeves and stainless-steel cutting nose-cones.

Harbor sediments were collected to approximately the projected dredging depth, i.e., project depth (53 feet MWL) plus 2 feet for overdredging. A VT6 Vibracoring Head, weighing approximately 500 lbs, was used with 23-foot long coring tubes to collect sediment from the sediment surface to dredging depth or refusal. At one station, water depth was approximately 10 feet shallower than expected, consequently the vibracore was unable to sample to dredging depth. The EPA representative was consulted for guidance to determine the best course of action, i.e., accepting the 18-foot core sample as collected.

After collecting sediment in the vibracore tube, the sampler was withdrawn from the sediment and placed on deck where the inner sleeve was removed. After visually inspecting and describing the sediment, the sleeve was opened and the sediment was placed in sample containers using stainless steel spoons and bowls only. The volume of sediment was recorded to ensure that there would be sufficient volume for generating the composited sample.

Reference samples were collected by grab sampler, i.e., a box corer. Collected sediments were placed in containers made of stainless steel or other material with a protective coating of Teflon®

or similar material. Excess water was decanted before placing the sample in the laboratory-supplied one-gallon containers.

Samples were shipped overnight delivery to PPB in ice chests with wet ice. Custody seals were secured to each ice chest in a manner to disclose if the ice chest had been opened before delivery to the laboratory.

The original sampling plan had anticipated that a fine sand layer would be found to overlay a silty clay layer in the harbor and that these two layers would be sampled and composited separately. Vibracoring harbor sediments yielded no fine sand layer at any station. Consequently, after transport to the laboratory, the silty clay sediment from each station was homogenized separately. After one liter was removed for TOC, grain size, and percent moisture testing and another liter was archived, the remaining homogenized sediments were stored until all harbor sediments had been collected and similarly processed.

After all harbor stations had been sampled, information was available to calculate the proportions needed to form a composite sample for the silty clay sediment. The amount of sediment used to form the composite sample was in proportion to the thickness of the sediment, i.e., silty clay layer, encountered at each station.

Predetermining the positions for the reference stations resulted in selecting one location where there was insufficient sediment for sample collection. After consulting with the EPA Project Manager, it was decided that the sampling team would return to the first reference location and collect the remaining 25 gallons of needed reference sediment there. Consequently, all 50 gallons of reference sediment was a homogenized sample of sediment collected from one location. Reference station locations are provided in Section 3.

Control sediment was provided by HESE (MACTEC) in sufficient quantity for all specified physical, chemical, and biological tests. Information regarding the location from which this sediment was collected can be found in Section 3.

Table 1 is included to show the numbers and types of samples that actually were collected in the field as well as the numbers and types that were planned. Other field- and laboratory-generated quality control samples that were used are described in the Chemical and Physical Evaluation QAP section.

Table 2 was reproduced from Section 3 of this Work/QA Plan and shows the types of sample containers, sample preservation, and holding times for the water and sediment collected in the field. Also included on this table is information for the elutriate and tissue samples that were generated in the laboratory. The holding times for sediment and water analysis and elutriate preparation began when the water and sediment were collected and brought on board the sampling vessel.

2.2.4 Site Water Collection and Handling

Site water for organic analytes was collected directly into pre-cleaned, 1-gallon glass containers with Teflon® lid-liners. Site water for metals was collected in a similar manner using Teflon® containers. Sample containers were inverted and submerged at least 0.5 feet below the water surface before they were turned upright and allowed to fill. Water sample containers were

placed in ice chests with wet ice and transported to PPB in a manner similar to the sediment samples. The location from which the site water was collected is provided in Section 3.

2.2.5 Hydrographic Measurements

Collection of sediment and water from Ponce Harbor and the reference stations included the following general ancillary observations and hydrographic measurements made on water from mid-depth, or, for the reference stations, near the water surface:

Sea State	Water Temperature	Dissolved Oxygen
Tidal Stage	Water Depth	Turbidity
Direction of Tidal Flow	pH	Conductivity
	Weather Conditions	Salinity

2.3 Physical and Chemical Laboratory Methods and Procedures

Analytical procedures followed those prescribed in the guidance documents previously referenced and target detection limits were defined to meet EPA Region II guidance detection limits. Table 3 lists the analytical methods used for the physical and chemical testing and the target detection limit for each parameter or analyte.

2.4 Biological Testing

Biological testing included elutriate bioassays with *Mysidopsis bahia*, *Mytilus edulis*, and *Menidia beryllina*; benthic bioassays using *Mysidopsis bahia* and *Leptocheirus plumulosus*; and bioaccumulation by exposing *Nereis virens* and *Macoma nasuta*. *Leptocheirus plumulosus* was substituted for *Ampelisca abdita* because the latter was not unavailable when needed. Complete descriptions of the biological testing procedures appear in Appendix B, Section 4.

The supplier of these test organisms was:

Aquatic Research Organisms
P.O. Box 1271
One Lafayette Road
Hampton, NH 03842
Phone (603) 926-1650

Water used during toxicity testing was provided by HESE (MACTEC) and comes from their routine source, i.e., the University of Florida's Whitney Center saltwater storage tank. The Whitney Center is located along US A1A and is north of and adjacent to Marineland, Florida. Water from this source has proven to be reliably free of contaminants with regard to success of past control groups for test organisms. A report documenting the results of chemical analyses on the waters used during toxicity testing, i.e., bioassays and bioaccumulation, appears in Appendix C.

Control sediment was collected from the Matanzas River approximately 100 meters north of the Whitney Center and west of US A1A. This location is approximately at N 29.6719° and W -081.2148° (NAD 83).

3 Locations of Sampling Areas

The proposed disposal site, the Ponce ODMDS, is located approximately 3.5 nautical miles (NM) offshore from the island of Puerto Rico near Ponce Harbor and is designated for the disposal of dredged material. The site is one nautical mile square with center coordinates of +17.88976° N, -066.61969° W and is in water that averages 1,400 feet (425 meters) deep. Using the NAD 83 datum, the corners of the ODMDS are located at:

+17.97139° N, -066.61611° W
+17.97250° N, -066.61833° W
+17.88134° N, -066.62822° W
+17.88138° N, -066.61100° W

Nine harbor sampling locations were selected that included existing channel areas as well as areas where dredging would occur for the first time, i.e., new work areas. Two reference stations were selected from water 100 to 200 feet deep from areas with relatively flat topographies between the harbor and the ODMDS. The actual locations sampled appear on Table 4 which also shows the distance between each target and actual sampling location.

The sampling locations within the harbor area are shown in Figure 2.

Figure 3 shows the sampling locations for the harbor and reference sampling stations. This figure also shows sampling locations in the ODMDS that were part of a different but concurrent sampling effort to characterize sediments before disposal activities could begin.

Positioning of the sampling equipment at the predetermined sampling stations was accomplished using a Magnavox Model 412 DGPS receiver coupled with a Starlink Model MRB-2A beacon receiver. Differential corrections were acquired using the Coast Guard beacon from Isabela, Puerto Rico from which real-time GPS differential corrections are broadcast at 295 KHz. This system has a proven accuracy of ±5 m (1 m to 3 m is typical) because of the high quality of the GPS and beacon receivers and the use of an "H-field" antenna.

Copies of completed field sheets developed for this sampling effort appear in Appendix D.

In addition to location information, other sampling measurement information was collected at each station. Table 5 presents information regarding anticipated water depth, observed water depth, vibracore penetration, core length recovered, and core length needed to reach project plus overdredging depth.

4 Description of Testing Approach

As reported in Section 2, a Tier I investigation was performed and from it and agency guidance, a list of chemicals and physical parameters of interest was developed.

Limited testing of the harbor sediment had previously been performed and some information was available from sediment borings. These borings indicated that a layer of silty clay sediment was present throughout the harbor and, in a portion of the harbor, a fine sand layer was found overlying the silty clay. Based on this sediment layering information, a sampling plan was designed to sample each layer separately and to generate a composite sample for each layer for testing. Sediment for each composite sample was to be proportioned based on the thickness of the layer at each station relative to the sum of the thicknesses for that layer at all stations.

Selecting nine sampling stations throughout the harbor was believed adequate to provide a representative sample of each layer. Stations were distributed within the existing channel and turning basin and also in areas proposed for first time dredging.

Two reference station locations were selected for generating a composite sample by combining equal proportions from each station. If the fine sand layer was found to be less than two feet in thickness, it would be included as part of the silty clay sample.

The two composited harbor samples were to be tested along with the reference station composite sample for metals, pesticides, PCB congeners, PAHs, specific gravity, bulk density, and Atterberg limits. These three composites and a control sediment were to be tested for TOC, grain size, percent moisture, elutriate and sediment bioassays, and bioaccumulation.

Elutriate samples were generated from one harbor composite sample. This elutriate was to be tested for metals, pesticides, and PCB congeners and the results used in the STFATE model. The modeling results previously have been reported (PPB 2003).

A list of chemicals of interest was developed for the bioaccumulation chemicals in tissues. This list was to be reevaluated after sediment and water test data became available. Ultimately, it was decided that tissues would be tested for metals and PAHs.

5 Final Results

Prior to the initiation of field work, preprinted field sheets were designed specifically for this project. Completed field documentation appears in Appendix D. All laboratory reports of analyses for Ponce Harbor and reference station sediments, water and elutriates, and tissues appear in Appendix E.

5.1 Field Measurement Data and Sediment Descriptions

The weather during the field effort was generally sunny with 5- to 10-knot winds in the mornings which rose to greater than 15 knots later in the day. Tidal height ranged from 0.21 to 0.46 feet above mean low water during the sampling period. Table 6 presents the water quality data measured at mid-depth for each harbor station and near the surface for the reference stations.

Sediment in the harbor is a consistent dark gray, silty clay. A trace amount of fine sand with or without a small amount of shell fragments was found in the upper one to two feet of some cores.

Reference station sediments were described as "soft mud" indicating that they were composed of very fine particulates. It should be noted that repeated attempts to collect sediment at the predetermined location for Reference Station RS-PH03-2 failed to produce sufficient sediment for analysis. After discussion with the EPA project manager, it was decided that additional sediment would be collected at the first reference station and that these replicates would be labeled with the predetermined second reference station identifier.

Sediment descriptions have been consolidated and are presented in Table 7.

5.2 Sediment Physical and Organic Carbon Data

Sediment samples from the individual harbor stations and composited samples for the harbor and reference stations were sieved to determined grain size distribution and tested to determine the content of organic carbon and moisture. The composited samples also were tested for specific gravity, density, and Atterberg limits.

For all harbor samples including the composite, gravel was found to be 0.7 percent or less. Sand ranged from 4.7 and 13.4 percent while silt ranged from 57.3 and 65.5 percent and the clay values were from 22.4 to 34.2 percent. Moisture content ranged from 30 to 38 percent.

The reference station composited sediment contained a finer material than the harbor sediment with approximately 20 percent less silt and approximately 30 percent more clay. Gravel and sand at the reference station fell into the low end of the ranges found in the harbor. Moisture was 54 percent of the sample weight.

Control sediment used for toxicity testing was coarser than harbor and reference sediments with sand comprising 73.6 percent and gravel 15.5 percent. Moisture content in the control sediment at 30 percent was similar that of the harbor sediment.

Total organic carbon ranged from 0.63 to 0.81 percent in the harbor and control sediments. The reference sediment contained 0.91 percent TOC.

5.3 Sediment Chemical Data

Harbor and reference composite sediment samples were analyzed for metals, pesticides, PCB congeners, and PAHs. The harbor composite sample was tested in triplicate to provide precision information using the actual sample matrix. All chemical laboratory reports, complete with chain of custody forms and laboratory quality assurance narratives, appear in Appendix E.

5.3.1 Sediment Metals

For all metals, the concentrations found in the harbor composite were within a factor of two of the reference composite values. Lead, mercury, and selenium concentrations were greater in reference sediment that in the harbor. Silver yielded the only undetectable concentration (i.e., for the reference composite) and varied greatly in the triplicate harbor sediment ranging between 0.0704 and 0.740 µg/g, dry weight basis. Table 9 presents the sediment metals and percent moisture data including triplicate analysis information and the detection limits achieved by the laboratory.

5.3.2 Sediment Pesticides and PCB Congeners

Only three DDT-related compounds and one PCB congener were detected in sediment from either the reference station or the harbor. Reference station sediment contained the only quantifiable concentrations of these compounds with 4,4-DDT at 63 ng/g, 4,4-DDD at 0.070 ng/g, and 4,4-DDE at 320 ng/g, all on a dry weight basis. Two of these, 4,4-DDD and 4,4-DDE also were detected in harbor sediment at levels below the sample-specific method detection limits.

The only PCB congener detected was PCB-153. It was found in the replicate harbor samples at levels estimated to be approximately 0.019 ng/g or approximately one-fourth the sample-specific method detection limit.

Table 10 presents the pesticide and PCB congener data for the reference and harbor sediments. Because most of these compounds were not found in these sediments, quantifiable values have been bolded on the table to facilitate locating them.

5.3.3 Sediment PAHs

Unlike pesticides and PCB congeners, every PAH analyte was measured or detected in every sediment composite and replicate sample tested with values ranging from 0.12 to 9.22 ng/g, dry weight basis. Sediment from the reference station contained a higher concentration of each PAH compound than found in the harbor sediment; typically two to five times higher. PAH data are shown on Table 11.

5.4 Water and Elutriate Chemical Data

Site water and elutriates were tested for metals and pesticides and PCB congeners. All chemical laboratory reports, complete with chain of custody forms and laboratory quality assurance narratives, appear in Appendix E.

5.4.1 Aqueous Metals

Subjecting harbor sediment to the elutriation procedure increased concentrations of arsenic (approximately 10 times higher), chromium (two times), nickel (three times), and selenium (at

least doubled). Less than two-fold increases were seen for cadmium, lead, and mercury. Silver showed little change and copper and zinc concentrations were lower by a factor of approximately two to three. Table 12 shows averaged data for the harbor site water and the elutriates.

5.4.2 Aqueous Pesticides and PCB Congeners

No pesticides or PCB congeners were detected in any water or elutriate sample. Table 13 provides information on the method detection limits achieved for these samples. Because no detectable values were found, the precision statistic, percent relative standard deviation could not be calculated.

5.5 Bioassay Results

Static, non-renewal elutriate bioassays were performed using *Mysidopsis bahia* and *Menidia beryllina* in 96-hour tests and *Mytilus edulis* for 48-hour tests. *Mysidopsis bahia* and *Leptocheirus plumulosus* were used in 10-day, static renewal tests. *L. plumulosus* was a substitution for *Ampelisca sp.* which was unavailable for use. The complete toxicity testing report appears in Appendix F.

5.5.1 Elutriate Bioassays

Elutriate bioassay test conditions, including temperature, DO, pH, and salinity were maintained at acceptable levels throughout the testing period. The relevant laboratory raw data pertaining to the elutriate tests are provided in Appendix F for *M. bahia, M. beryllina*, and *M. edulis*.

Elutriate Bioassays with *Mysidopsis bahia*

M. bahia survivorship was 93 percent in the laboratory control seawater. Survivorship in the laboratory control sediment (Table 14) was 96 percent, 95 percent, and 97 percent in the 10 percent, 50 percent, and 100 percent concentrations, respectively. Survivorship of *M. bahia* in the one harbor composite sediment sample (E-PH03-SC) was 97 percent, 95 percent, and 87 percent in the 10 percent, 50 percent, and 100 percent concentrations, respectively. Surviving *M. bahia* appeared healthy at test termination. *M. bahia* survivorship among replicates was relatively uniform (Appendix F).

The 100 percent elutriate survival dataset was found to be both normal and with homogeneity of variance. Analysis of variance followed by Dunnett's comparison indicated that the survival of *M. bahia* in the laboratory control seawater was not significantly different from either the 100 percent control sediment elutriate exposure or the site sample (Table 15). Results for the 2 sample t-Test between the 100 percent elutriate exposures for the control sediment and the site sample showed a statistically significant difference between these two samples (Table 15). LC_{50}s were >100 percent (Table 16).

Elutriate Bioassays with *Menidia beryllina*

M. beryllina survivorship was 98 percent in the laboratory control seawater. Survivorship in the laboratory control sediment (Table 17) was 97 percent, 96 percent, and 95 percent in the 10 percent, 50 percent, and 100 percent concentrations, respectively. Survivorship of *M. beryllina* in the harbor composite sediment sample (E-PH03-SC) was 94 percent, 85 percent, and 72 percent in the 10 percent, 50 percent, and 100 percent concentrations, respectively. Surviving *M. beryllina* appeared healthy at test termination. *M. beryllina* survivorship among replicates

was relatively uniform, except in sample E-PH03-SC which exhibited some variance timing replicates (Appendix F).

This 100 percent elutriate survival dataset did not meet the assumptions for a parametric test, and a suitable transformation was not found. The non parametric Kruskal-Wallis comparison showed a statistically significant difference between the survival in the control seawater and the 100 percent elutriate of E-PH03-SC (Table 18). With the removal of the control seawater data, the dataset met the assumptions for a parametic comparison and a t-Test was conducted. Survival in sample E-PH03-SC was found to be statistically different from the 100 percent control sediment elutriate survival in this comparison (Table 18). LC_{50}s were >100 percent (Table 16).

Elutriate Bioassays with *Mytilus edulis*

M. edulis development (expressed as percentage of embryos resulting in live larvae with complete shell development) was 75 percent in the laboratory control seawater, with acceptable survivorship (Table 19, Appendix F). Development in the laboratory control sediment (Table 19) was 72 percent, 71 percent, and 69 percent in the 10 percent, 50 percent, and 100 percent concentrations, respectively. Development of *M. edulis* in the one site sediment (E-PH03-SC) was 72 percent, 70 percent, and 60 percent in the 10 percent, 50 percent, and 100 percent concentrations, respectively. *M. edulis* development among replicates was relatively uniform (Appendix F).

The 100 percent elutriate development dataset was found to be both normal and with homogeneity of variance. Analysis of variance followed by Dunnett's comparison indicated that the development of *M. edulis* in the laboratory control seawater was significantly different from both the 100 percent control sediment elutriate exposure or the site sample (Table 20). Results for the 2 sample t-Test between the 100 percent elutriate exposures for the control sediment and the site sample showed a statistically significant difference between these two samples (Table 20). EC_{50}s were estimated as >100 percent (Table 16).

Elutriate Reference Toxicant Test

Concurrent reference toxicant tests were conducted to determine the general health of each test species. The reference toxicant for the *M. bahia* and *M. beryllina* tests was sodium dodecyl sulfate (SDS) with a test duration of 48 hours. The reference toxicant for the *M. edulis* test was copper sulfate (CuSO4) with a duration of 48 hours and was performed concurrently with the fertilization tests. The 48-hour LC50 result for *M. bahia* was 8.91 mg SDS/L (95 percent confidence limits of 6.88 to 11.53 mg SDS/L) and for *M. beryllina* was 2.18 mg SDS/L (95 percent confidence limits of 1.78 and 2.66 mg SDS/L). The current LC50 values were within the upper and lower control limits (±2S) of the Harding ESE control charts running average LC50, and indicated that the test organisms were within their normal sensitivity ranges (USEPA, 1994). The reference toxicant EC_{50} for the mussel was also consistent with others performed by the laboratory. The reference toxicant data sheets and the LC50 and EC_{50} calculations for the elutriate tests are presented in Appendix F.

5.5.2 Sediment Bioassays

Sediment bioassay test conditions, including temperature, DO, pH, and salinity were maintained at acceptable levels throughout the testing period. Ammonia results are discussed below. The relevant laboratory raw data pertaining to the sediment tests are provided in Appendix F for *M. bahia* and *L. plumulosus*.

Sediment Bioassays with Mysidopsis bahia

Pore water samples were collected on Day 0 and total ammonia (NH_3-N) values were 10.8 ppm (Control PH03), 28.8 ppm (E-PH03-SC), and 7.2 ppm (RS-PH03). As per the guidance, samples were aerated and overlying water samples were collected on Day 0 through 10 for sample E-PH03-SC, and total ammonia ranged from 7.3 ppm (Day 0) to 0.408 ppm (Day 10). Ammonia for other samples remained low throughout the test duration (Appendix F).

M. bahia survivorship was 96 percent in the laboratory control sediment and 92 percent in the reference sediment, RS-PH03 (Table 21). Survivorship of *M. bahia* in the harbor composite sediment sample was 96 percent (Station E-PH03-SC). Surviving *M. bahia* appeared healthy at test termination. *M. bahia* survivorship among replicates was relatively uniform (Appendix F).

Statistical analysis indicated that the survival of *M. bahia* in the laboratory control sediment was not significantly different (p=0.05) from survival in the Ponce Harbor sample or reference sample (Table 22). Survival of *M. bahia* in the reference sediment was not significantly different (p=0.05) from survival in the harbor sediment sample (Table 22).

Sediment Bioassays with *Leptocheirus plumubosus*

Pore water samples were collected on Day 0 and total ammonia (NH_3-N) values were 7.3 ppm (Control PH03), 38.0 ppm (E-PH03-SC), and 7.5 ppm (RS-PH03). Overlying waters were vigorously aerated to reduce the ammonia to less that 20 ppm prior to initiation of testing. Additional replicates ("dummy" replicates, without organisms) of the harbor sediment were assembled for pore water monitoring throughout the test duration. Total ammonia in pore water samples ranged from 23.2 ppm (Day 0) to 2.92 ppm (Day 10).

L. plumulosus survivorship was 95 percent in the laboratory control sediment and 95 percent in the reference sediment (Table 23). Survivorship of *L. plumulosus* in the harbor composite sediment sample was 84 percent (sample E-PH03-SC). It should be noted, at test termination and prior to counting of organisms, one replicate of the laboratory control was accidentally broken, and is not included in the survivorship. Surviving *L. plumulosus* appeared healthy at the termination of the tests.

Statistical analysis using Dunnett's T-test indicated that the survival of *L. plumulosus* in the laboratory control sediment was not significantly different (p=0.05) from survival in the reference sediment, but was significantly different from the Ponce Harbor survival (Table 24). However, survival of *L. plumulosus* in the reference sediment was not statistically different from the harbor sediment sample (Table 24).

Sediment Reference Toxicant Test

Reference toxicant tests were conducted on each species tested in the sediment tests. The reference toxicant used for *M. bahia* was SDS and the duration of the test was 96 hours. The reference toxicant used for *L. plumulosus* was cadmium chloride (CdCl2), measured as Cd, for a duration of 96 hours. The 96-hour LC50 result for *M. bahia* was 4.39 mg SDS/L (95 percent confidence limits of 1.69 to 11.39mg SDS/L) and the 96-hour LC50 result for *L. plumulosus* was 1.93 mg Cd/L (95 percent confidence limits of 1.56 to 2.37 mg Cd/L). The current LC50 values were within the upper and lower control limits (±2S) of the Harding ESE control charts running average LC50, and indicated that the test organisms were within their normal sensitivity ranges (USEPA, 1994). The reference toxicant data sheets and LC50 calculations for the sediment tests are presented in Appendix F.

5.6 Bioaccumulation Results

The bioaccumulation test conditions, including temperature, DO, pH, and salinity were maintained at acceptable levels throughout the 28-day testing period. Flow-through conditions were maintained with minimal interruptions to adjust flows, and on two occasions to replace parts. The toxicity testing laboratory raw data and reference toxicant data are provided in Appendix F. Results of the reference toxicant testing confirm that the organisms were in good health at test initiation and are consistent with results of similar testing by the laboratory (Appendix F).

5.6.1 Organism Survival Rates

An adequate number of *Macoma nasuta* and *Nereis virens* were exposed to control, reference, and harbor sediment to provide sufficient quantities of tissue for subsequent chemical testing.

Bioaccumulation using *Macoma nasuta*

Data for the survival of *M. nasuta* in the bioaccumulation tests are presented in Table 25. *M. nasuta* survivorship in the laboratory control sediment was 92. Survival of *M. nasuta* in the harbor composite sediment sample (Sample E-PH03-SC) was 87 percent (Table 25). Survival of *M. nasuta* in the reference sediment (Sample RS-PH03) was 94 percent (Table 25). Adequate mass of *M. nasuta* tissue was available for chemical analyses.

Bioaccumulation using *Nereis virens*

Data for the survival of *N. virens* in the bioaccumulation tests are also presented in Table 25. *N. virens* survivorship in the laboratory control sediment was 95 percent. Survival of *N. virens* in the site sediment was 88 percent (sample station E-PH03-SC) (Table 25). Survival of *N. virens* in the reference sediment was 95 percent. Adequate mass of *N. virens* tissue was available for chemical analyses in the remaining samples.

5.6.2 Bioaccumulation Chemical Test Results

After review of the chemical testing results for sediment samples, it was decided that tissue samples of organisms exposed to either harbor sediment or reference sediment would be analyzed for metals and PAHs. Laboratory reports for tissue analyses appear in their entirety in Appendix E.

Five groups of test the *Macoma nasuta* and *Neries virens* were exposed to separate subsamples of either harbor or reference sediment. From the resulting analytical data, mean chemical concentrations were calculated for each species exposed to sediment. Statistical comparisons were made using the one-sided t-test to determine if there were a difference at a significance level of 0.05. Summary data for *Macoma* are presented on Table 26 showing the concentration means, relative lower or upper confidence limit values, and the t-test finding for significance. Only five chemicals were found to have higher average concentrations in harbor sediment exposed organisms than those exposed to reference sediment. Four of these five chemicals averaged less than concentrations found in pretest organisms. No chemical was significantly higher in the harbor organisms than in the reference organisms.

As with *Macoma*, *Nereis* tissue samples had only five chemicals that averaged higher in organisms exposed to harbor sediment than those exposed to reference sediment and two of these

averaged less than the concentrations of the pretest organisms. Summary results for *Nereis* appear on Table 27.

6 Discussion and Analysis Of Data

6.1 Historical Data

The Tier I investigation noted that a Final Environmental Impact Statement (EIS) was prepared in 1974. This EIS included "receiving water" test results for some metals without providing a location for the sample or units. No other chemical data were presented.

6.2 Comparison of Harbor and Reference Sediments

Sediments were collected and composited to generate samples representative of the harbor and the reference locations. Initial plans called for creation of two harbor composite samples, i.e., one for a fine sand layer and one for a silty clay layer. Because only silty clay was encountered, a single harbor sample was composited and tested. This sample was analyzed in triplicate to provide an indication of the precision obtained.

In order to provide a statistical comparison, ranges for 90 percent confidence limits were calculated around mean values for analytes detected in the harbor composite sample. Each confidence interval was calculated by taking a mean harbor value and adding to and subtracting from it the product of the standard deviation and a risk and probability level factor (i.e., 2.920) divided by the square root of 3 (i.e., 1.732).

These ranges are presented on Table 28 along with detectable analyte values for the reference station sample. Only a single result from the reference sediment was obtained for each analyte.

When comparing the mean harbor values to the reference station values, seven of the 12 metals tested were found to have higher levels in harbor sediment than in reference sediment while three were higher at the reference station. With the exception of silver, all sediment metal values found in the harbor sediment fell in a range of 60 to 167 percent of the values obtained for the reference station. Replicate silver analysis of the harbor sediment produced disparate results of 0.0704, 0.111, and 0.740 ng/g, dry weight basis, while reference sediment yielded a value of <0.03 ng/g, dry weight basis.

Three DDT-related compounds and all 17 of the PAHs for which testing was performed were found in measurable quantities in the reference sediment. The DDT-related compounds were undetected or below the method detection limits in the harbor sediment and 16 PAHs were at measurable levels in harbor sediment. In all cases, the reference sediment contained levels of the DDT-related compounds and the PAHs that were greater than the 90 percent confidence limits calculated from the harbor sediment analyses.

No PCB congeners were found in the reference sediments and only one, PCB 153, was found in the harbor composite sample. The concentration of this one detected congener was estimated to be approximately one third the method detection limit, i.e., 0.019 ng/g, dry weight basis, compared to less than 0.062 ng/g, dry weight basis, for the reference sediment.

6.3 Bioaccumulation of Copper

In general, the chemical concentrations observed in the tissue of test organisms are within the bounds normally observed at similar sites. Only copper concentrations in *Nereis virens* appear to be elevated above what would be expected based upon the levels of copper found in the

sediments, the fate and transport properties of copper, and the results for *Macoma nasuta.* The results are inconsistent with the observed tissue results for *Macoma nasuta* because bivalve mussels are well documented to have higher bioaccumulation potential for copper (USEPA 1984; Eisler 1981), and yet the values measured for *M. nasuta* in this study are more than an order of maginitude lower than the corresponding values for *N. virens.* In addition, the results for *N. virens* are higher than would be predicted based upon standard modeling assumptions, and available site data (TrophicTrace 2003). Therefore, it appears highly likely that the observed copper concentrations in *N. virens* are not the result of simple accumulation from the harbor or reference sediments.

The original 28-day bioaccumulation test failed in the laboratory. It then was discovered that the new chillers, installed before these tests, had exposed copper piping inconsistent with the specifications for the equipment. These were replaced and the system cleaned. Although the test was reinstated and successfully completed, there may have been another fugitive source of copper related to the original discovery. There is a more likely explanation for the unusual copper body burdens in *N. virens*.

7 Quality Assurance and Control

Chemical data quality was monitored and assured within the Battelle laboratory and with a field quality assurance audit that is summarized in this section. No fine sand layer was found in the harbor, consequently up to nine discrete station samples and one harbor composite sample were not obtained for analysis. However, all other field and laboratory data, that were planned to be collected, were collected and are reported in this document.

Review of the field data and sample collection process and laboratory data and quality assurance reports show that all data are usable for project purposes of this Section 103 predredging sediment evaluation.

7.1 Field Quality Assurance Audit Summary

A field quality assurance audit was performed during which all field activities were observed. This field audit report appears in its entirety in Appendix G.

Before field activities began, the project goals and approach for field activities were described in a USEPA Region II and US Army Corps of Engineers approved Work/Quality Assurance Plan (Appendix B). These were provided to field team members prior to traveling to Puerto Rico. The field team was staffed with personnel with appropriate training and experience for the tasks they were assigned. These individuals followed the procedures and methods described in the W/QAP for sample collection, preservation, storage, and transport. A problem experienced with the second reference station was resolved with adequate communication among field team members and the project manager for EPA Region II. Stations were located using differentially-corrected global positioning system receivers. Information was properly documented on field sheets, sample labels, and chain of custody forms. All problems encountered were successfully resolved with the corrective actions taken. Except as noted previously, all samples that were planned for collection were collected. One glass water container was broken during shipping but sufficient sample had been collected, avoiding a problem of insufficient sample volume. All other sample containers were received in good condition by laboratory personnel.

All samples and collected field data were judged acceptable for project needs.

7.2 Chemical Laboratory Quality Assurance and Control

Battelle quality assurance narrative reports appear in Appendix E with the laboratory data reports that they describe. All routine problems encountered during analyses were successfully addressed.

7.2.1 Sediment Results

For sediment results, there is an apparent inconsistency between the detection limits reported with the data and the target detection limits as shown in the Work/QAP for some analytes. The target method detection limits (MDLs) presented in the Work/QAP were the current 2002 annual laboratory-achieved MDLs for Battelle. The Ponce samples were collected and analyzed in 2003, consequently the data were reported using the current and appropriate 2003 laboratory-generated MDLs.

The target detection limits for metals were achieved; however, for the reason explained previously, testing for many of the pesticide and congeneres did not meet the MDLs listed in the Work/QAP. The target MDLs for most of the PAH compounds were achieved or bettered. Table 29 shows each sediment analyte with Work/QAP target detection limit, the USEPA Region II guidance detection limit, and an approximation of the achieved detection limit. Detection limits are calculated on a dry weight basis which yields some variability among samples in a single analytical batch because of differing moisture content and the weight of sample extracted. Consequently approximate detection limits are presented on the table. The detection limits achieved with procedural blank samples, where available, are reported on Table 29 as the achieved detection limits.

Generally the achieved detection limits for pesticides and PCB congeners were 1.5 to eight times higher than the targets. Unlike the PAHs, few pesticide and PCB congeners were detected in the sediment samples.

As seen on Table 29 all detection limits achieved during the analysis of the ODMDS and reference sediment samples were equal to or lower than those specified by EPA Region II guidance or the approved Work/QAP target MDLs. The MDLs achieved for pesticides and PCB congeners in Ponce sediments were typically 10 percent of the guidance limits and for PAHs, typically less than one percent.

Because all of the regional guidance detection limits were met using the 2003-generated MDLs, any inconsistency with the detection limits listed in the Work/QAP is not judged to be an issue of concern.

7.2.2 Water and Elutriate Results

As with the sediment results, there is an apparent inconsistency between the detection limits reported with the data and the target detection limits as shown in the Work/QAP for some analytes. The data were reported with the appropriate 2003 laboratory-generated MDLs instead of the MDLs for the previous year.

The detection limits achieved for metals in a water matrix were equal to or lower than the target MDLs of the approved Work/QAP.

Pesticide detection limits typically were one-half to two times the target MDLs. These achieved MDLs were lower than the EPA Region II guidance concentrations for each pesticide.

For the PCB congeners, the detection limits achieved typically were two to three times higher than the EPA Region II guidance concentrations. The PCB 8 congener was a notable exception with an achieved detection limit of 32 ng/L compared to a guidance level of 0.5 ng/L.

Only one PCB congener, PCB-153, was detected in harbor sediment. This congener was estimated to be 0.019 ng/g, or approximately one-third the target MDL. With a dry bulk density of 1.21 g/cm^3, an elutriate containing as much as 4.6 ng/L could have been generated in the unlikely event that all of the PCB-153 congener were to dissolve during the elutriation procedure. This calculated maximum concentration for PCB-153 in a harbor elutriate would exceed the guidance concentration and would be greater than 4 times the achieved detection limit for PCB-153. Consequently, there exists a possibly that PCB-153 was present between the

guidance concentration (0.5 ng/L) and the MDL (1.06 ng/L) and that it was not detected at levels great enough to result in an estimated value qualified with a "J" data flag.

7.2.3 Tissue Results

The detection limits achieved for PAHs and metals in tissue samples were equal to or lower than the target MDLs of the approved Work/QAP. Table 31 shows the target MDLs for tissues, the guidance concentrations, and the MDLs that were achieved.

8 Conclusions

Sediment sampling in Ponce Harbor in March 2003 found only a silty clay layer between the water-sediment interface and the proposed project depth of 53 feet plus 2 feet for overdredging. An expected fine sand sediment layer was not found. With the exception of information for the fine sand layer, all data were collected as planned for the chemical, physical, and toxicological characterization of sediment from Ponce Harbor along with similar data from a reference site for comparison. Appropriate methods and procedures were employed to collect and deliver sediment samples to the analytical laboratory in a condition that was representative of the site at the time of collection. While attempting to sample a second reference station, a problem was encountered that was successfully resolved with input from the USEPA project manager. Some target detection limits were not attainable for a variety of reasons; however, all of the achieved detection limits met or were lower than those specified by EPA Region II guidance documentation for sediment and tissue data and all water matrix guidance concentrations except for PAHs. All data are judged to be of acceptable quality for project needs.

9 References

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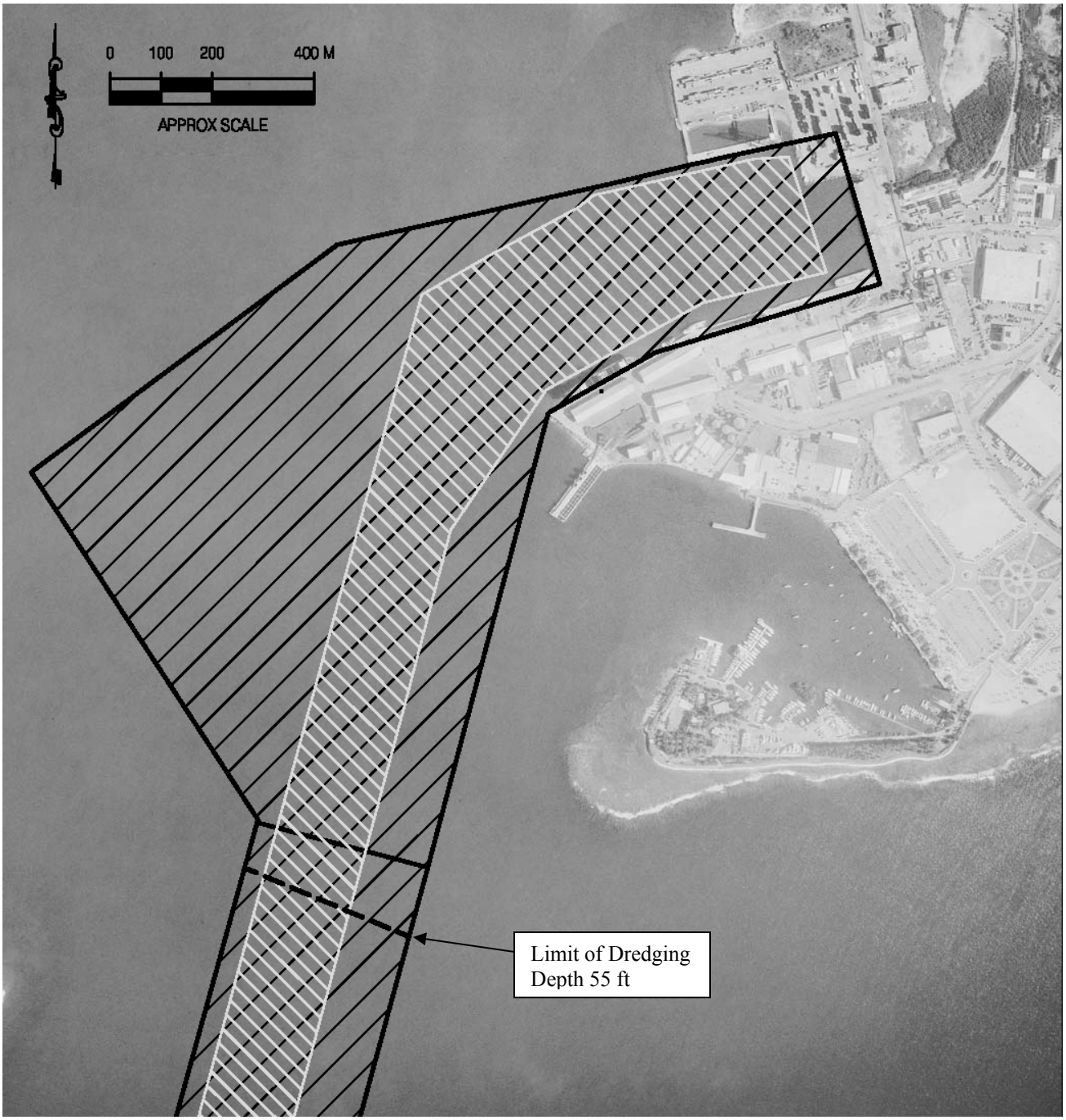
Figures



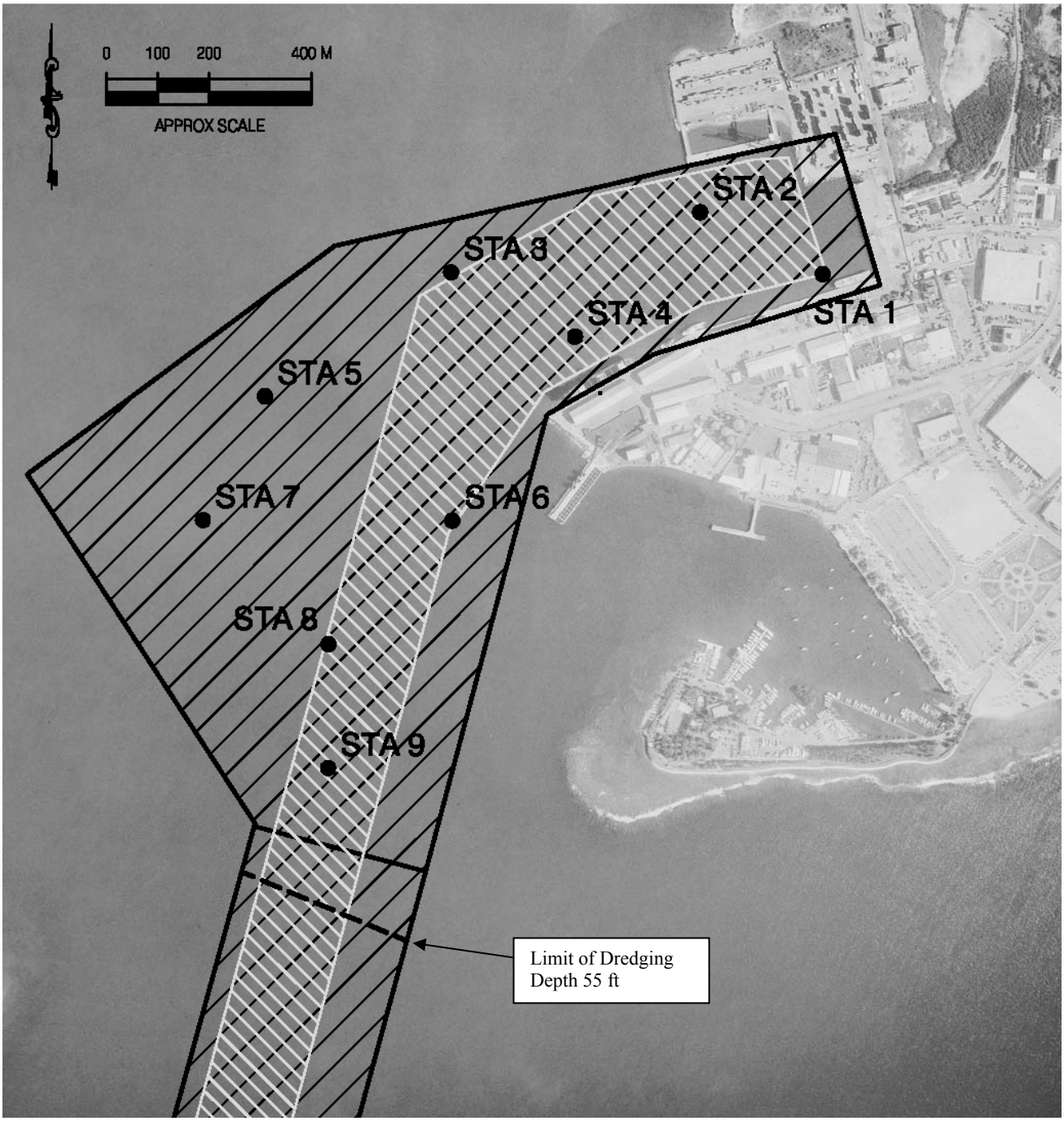
LEGEND

Area Proposed for Deepening

Existing Channel and Turning Basin

Figure 1.
Existing Channel and Turning Basin and Area of Proposed Deepening
Ponce Harbor, Puerto Rico

Source: CSA Group, 2002; Water & Air Research, Inc., 2003.



LEGEND

Area Proposed for Deepening

Existing Channel and Turning Basin

Figure 2.
Sediment Sampling Locations
Ponce Harbor, Puerto Rico

Source: CSA Group, 2002; Water & Air Research, Inc., 2003.

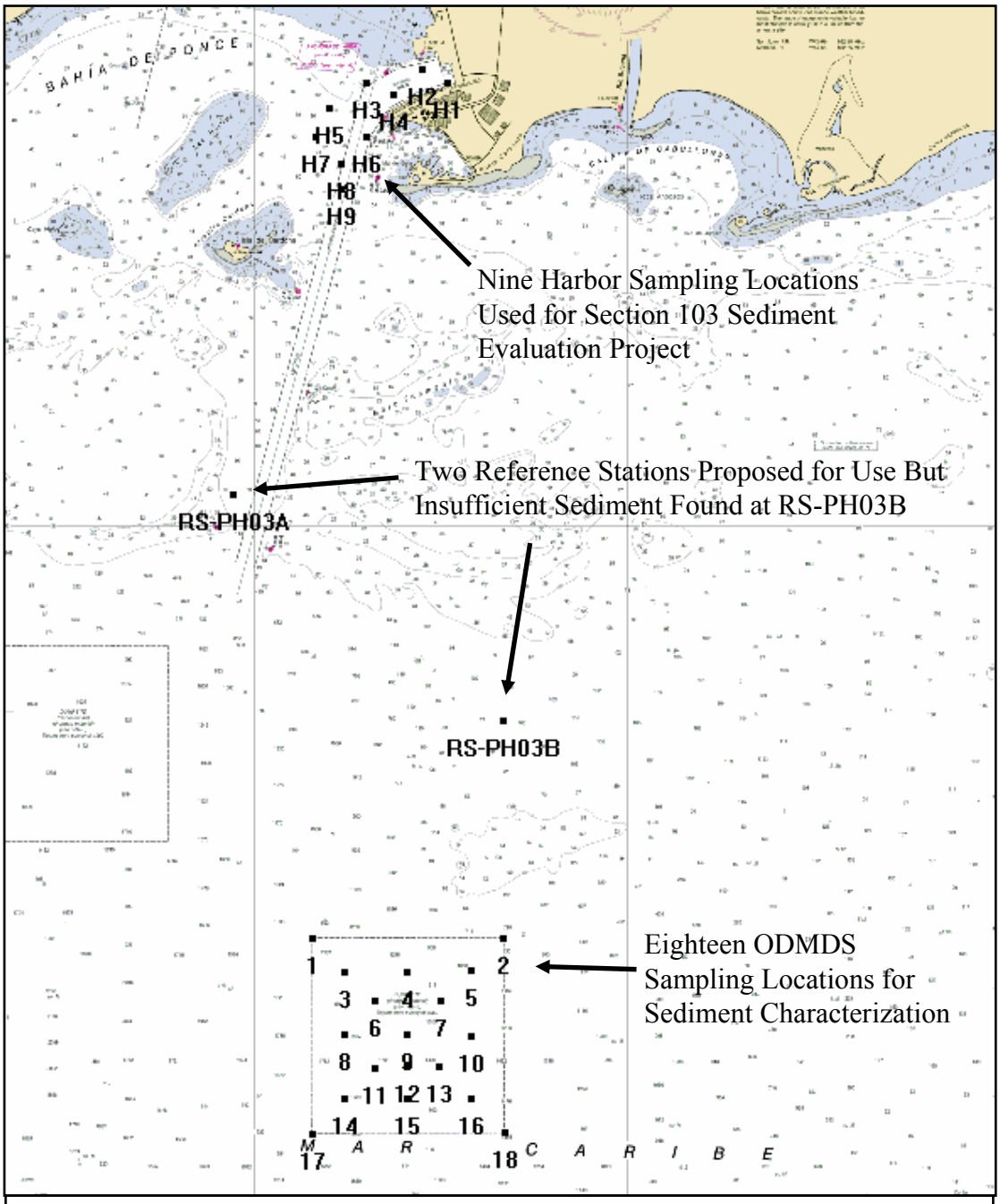


Figure 3. Sampling Locations for Ponce ODMDS and Reference Sediment Characterization Associated with Ponce Harbor, Puerto Rico, March 2003. True north is to the top of the figure. Each Ocean Dredged Material Disposal Site (ODMDS) is approximately one nautical mile square. Map project is mercator and the datum is NAD 83.

Tables

TABLE 1
Numbers and Types of Samples Collected for Ponce Harbor Evaluation

	Metals	Pesticides	PCB Congeners	PAHs	TOC	Grain Size + Moisture
Sediment						
Upper Layer Core	--	--	--	--	9 (9)	9 (9)
Deeper Layer Core	--	--	--	--	0 (9)	0 (9)
Harbor Composite	1 (2)	1 (2)	1 (2)	1 (2)	1 (2)	1 (2)
Reference Composite	1	1	1	1	1	1
Control	--	--	--	--	1	1
Total Number of Samples	2 (3)	2 (3)	2 (3)	2 (3)	12 (22)	12 (22)
Water/Elutriate						
Site Water	1	1	1	--	--	--
Upper Layer Core	--	--	--	--	--	--
Deeper Layer Core	--	--	--	--	--	--
Harbor Composite	1 (2)	1 (2)	1 (2)	--	--	--
Reference Composite	--	--	--	--	--	--
Control	--	--	--	--	--	--
Total Number of Samples	2 (3)	2 (3)	2 (3)	--	--	--
Tissue						
Harbor Composite	1 (2)	1 (2)	1 (2)	1 (2)	--	--
Reference Composite	1	1	1	1	--	--
Control	--	--	--	--	--	--
Pretest Organisms	1	1	1	1	--	--
Number of Species	2	2	2	2	--	--
Total Number of Samples	6 (8)	6 (8)	6 (8)	6 (8)	--	--

Numbers shown in parentheses indicate number of samples planned when deviation from plans occurred.

	Physical Tests	Percent Moisture + Lipids	Water Bioassays	Benthic Bioassays	Bioaccumulation
Sediment					
Upper Layer Core	--	--	--	--	--
Deeper Layer Core	--	--	--	--	--
Harbor Composite	1 (2)	--	--	--	--
Reference Composite	--	--	--	--	--
Control	--	--	--	--	--
Total Samples/Reps for Testing	1 (2)	--	--	--	--
Water/Elutriate					
Site Water	--	--	--	--	--
Upper Layer Core	--	--	--	--	--
Deeper Layer Core	--	--	--	--	--
Harbor Composite	--	--	--	--	--
Reference Composite	--	--	--	--	--
Control	--	--	--	--	--
Total Samples/Reps for Testing	--	--	--	--	--

TABLE 1
Numbers and Types of Samples Collected for Ponce Harbor Evaluation

Tissue	Physical Tests	Percent Moisture + Lipids	Water Bioassays	Benthic Bioassays	Bioaccumulation
			M. bahia Mytilus edulis *Me. beryllina*	M. bahia Ampelisca abdita	Nereis virens Macoma nasuta
Harbor Composite	--	--	1 (2)	1 (2)	1 (2)
Reference Composite	--	--	--	1	1
Control	--	--	1	1	1
Pretest Organisms	--	1	--	--	--
Number of Species	--	2	3	2	2
Total Samples/Reps for Testing	--	2	6 (9)	6 (8)	6 (8)

Physical Tests = specific gravity, bulk density, Atterberg Limits
Numbers shown in parentheses indicate number of samples planned, when deviation from plans occurred

TABLE 2
Sample Holding Times, Container Types, and Preservation

Matrix	Preservation	Container Type	Holding Time (from Green Book and RTM)
Water	Organics: 4°C Metals: acidify in field with HNO3 within 48 h of collection	Organics: Amber glass	7 days from collection; 40 days after extraction
		Metals: Teflon, acid-washed	Hg 28 days from collection; 6 months from collection for all other metals
Elutriate	Organics: 4°C Metals: acidify with HNO3 within 48 h of preparation		Elutriate preparation within 14 days of sediment collection and 7 days of water collection
		Organics: Amber glass	7 days from elutriate preparation; 40 days after extraction
		Metals: Teflon, acid-washed	Hg 28 days from elutriate preparation; 6 months from preparation for all other metals
Sediment	4°C	Organics: Glass with Teflon-lined lid	14 days from collection at 4°C; 1 year frozen; 40 days after extraction
		Metals: Pre-tared, acid-washed, plastic Spex jar	Hg 28 days at 4°C; other metals 6 months at 4°C; all metals 1 year frozen
		Physical Parameters/TOC: Glass with Teflon-lined lid	21 days from collection at 4°C
Tissue	-20°C	Organics: Glass with Teflon-lined lid	Initiate within 14 days of completion of bioaccumulation study at 4°C; 1 year frozen; 40 days after extraction
		Metals: Pre-tared, acid-washed plastic Spex jar	Hg 28 days at 4°C; other metals 6 months at 4°C; all metals 1 year frozen

TABLE 3
Methods and Target Detection Limits for Ponce Harbor and Reference Sediment

Test Parameter	Methods Used		Target Detection Limits		
	Sediment and Tissues	Water and Elutriates	Sediment (dry wgt)	Tissue (wet wgt)	Water and Elutriates
1. Metals(units)			µg/g	µg/g	µg/L
a. Aluminum (sediment only)	1638/1640	---	0.5	---	---
b. Arsenic	1638/1640	200.8	0.05	0.03	0.05
c. Cadmium	1638/1640	200.8	0.01	0.003	0.01
d. Chromium	1638/1640	200.8	0.04	0.03	0.04
e. Copper	1638/1640	200.8	0.02	0.01	0.02
f. Iron (sediment only)	1638/1640	---	5	---	---
g. Lead	1638/1640	200.8	0.01	0.004	0.01
h. Mercury	245.5	1631	0.002	0.0003	0.002
i. Nickel	1638/1640	200.8	0.03	0.007	0.04
j. Silver	1638/1640	200.8	0.03	0.006	0.03
k. Selenium	1638/1640	200.8	0.08	0.02	0.5
l. Zinc	1638/1640	200.8	0.1	0.14	0.1
2. Organic Compounds					
a.Pesticides(units)			ng/g	ng/g	ng/L
1) Aldrin	8081	8081	0.01	0.03	0.4
2) Chlordane	8081	8081	0.01	0.05	0.5
3) 2, 4' -DDT	8081	8081	0.08	0.05	3
4) 2, 4' -DDD	8081	8081	0.01	0.04	1
5) 2, 4' -DDE	8081	8081	0.07	0.07	1
6) 4, 4' -DDT	8081	8081	0.02	0.06	2.5
7) 4, 4' -DDD	8081	8081	0.01	0.05	1
8) 4, 4' -DDE	8081	8081	0.02	0.04	0.5
9) Dieldrin	8081	8081	0.03	0.04	0.5
10) Endosulfan I	8081	8081	0.03	0.05	0.5
11) Endosulfan II	8081	8081	0.01	0.04	1

TABLE 3
Methods and Target Detection Limits for Ponce Harbor and Reference Sediment

Test Parameter	Methods Used		Target Detection Limits		
	Sediment and Tissues	Water and Elutriates	Sediment (dry wgt)	Tissue (wet wgt)	Water and Elutriates
12) Endosulfan Sulfate	8081	8081	0.01	0.08	1
13) Heptachlor	8081	8081	0.02	0.08	1.5
14) Heptachlor Epoxide	8081	8081	0.02	0.03	0.5
15) Trans nonachlor	8081	8081	0.01	0.05	0.5
b.PCB Congeners(units)			ng/g	ng/g	ng/L
1) PCB 8	8082	8082	0.1	0.4	0.6
2) PCB 18	8082	8082	0.01	0.2	0.42
3) PCB 28	8082	8082	0.01	0.2	0.5
4) PCB 44	8082	8082	0.02	0.2	0.44
5) PCB 49	8082	8082	0.01	0.2	0.45
6) PCB 52	8082	8082	0.01	0.2	0.43
7) PCB 66	8082	8082	0.02	0.4	0.45
8) PCB 87	8082	8082	0.01	0.2	0.34
9) PCB 101	8082	8082	0.01	0.8	0.35
10) PCB 105	8082	8082	0.02	0.2	0.18
11) PCB 118	8082	8082	0.02	0.4	0.26
12) PCB 128	8082	8082	0.02	0.4	0.42
13) PCB 138	8082	8082	0.02	0.4	0.4
14) PCB 153	8082	8082	0.02	0.6	0.32
15) PCB 170	8082	8082	0.02	0.2	0.3
16) PCB 180	8082	8082	0.02	0.4	0.29
17) PCB 183	8082	8082	0.02	0.2	0.28
18) PCB 184	8082	8082	0.02	0.2	0.28
19) PCB 187	8082	8082	0.02	0.2	0.26
20) PCB 195	8082	8082	0.02	0.2	0.33
21) PCB 206	8082	8082	0.02	0.2	0.37

TABLE 3
Methods and Target Detection Limits for Ponce Harbor and Reference Sediment

Test Parameter	Methods Used		Target Detection Limits		
	Sediment and Tissues	Water and Elutriates	Sediment (dry wgt)	Tissue (wet wgt)	Water and Elutriates
22) PCB 209	8082	8082	0.02	0.1	0.38
c. PAHs(units)			ng/g	ng/g	---
1) Acenaphthene	8270	Not Tested	0.04	0.02	---
2) Acenaphthylene	8270	Not Tested	0.04	0.04	---
3) Anthracene	8270	Not Tested	0.03	0.04	---
4) Benzo (a) Anthracene	8270	Not Tested	0.05	0.03	---
5) Benzo (a) Pyrene	8270	Not Tested	0.1	0.05	---
6) Benzo (b) Fluoranthene	8270	Not Tested	0.04	0.04	---
c. PAHs (continued) (units)			ng/g	ng/g	---
7) Benzo (k) Fluoranthene	8270	Not Tested	0.03	0.03	---
8) Benzo (g,h,i) perylene	8270	Not Tested	0.05	0.2	---
9) Biphenyl	8270	Not Tested	0.05	0.03	---
10) Chrysene	8270	Not Tested	0.04	0.04	---
11) Dibenzo (a,h) Anthracene	8270	Not Tested	0.04	0.08	---
12) Fluoranthene	8270	Not Tested	0.04	0.05	---
13) Fluorene	8270	Not Tested	0.04	0.05	---
14) Indeno(1,2,3-c,d) Pyrene	8270	Not Tested	0.08	0.1	---
15) Naphthalene	8270	Not Tested	0.1	0.1	---
16) Phenanthrene	8270	Not Tested	0.05	0.06	---
17) Pyrene	8270	Not Tested	0.04	0.2	---
d.Total Organic Carbon (SEDIMENT ONLY)	Lloyd Kahn	Not Tested	0.01%	---	---
e.Lipids (tissue only)	Bligh and Dyer	Not Tested	---	0.10%	---
3.Physical Parameters					
a. Grain Size (sediment only)	Plumb 1981	Not Tested	1.0%	---	---

TABLE 3
Methods and Target Detection Limits for Ponce Harbor and Reference Sediment

Test Parameter	Methods Used		Target Detection Limits		
	Sediment and Tissues	Water and Elutriates	Sediment (dry wgt)	Tissue (wet wgt)	Water and Elutriates
b. Specific Gravity (sed only)	ASTM D854	Not Tested	0.01 g	---	---
c. Percent Solids	SM 2540G	Not Tested	1.0%	0.10%	---
d. Atterburg limits (sed only)	ASTM D4318	Not Tested			

Note: Many of these methods are modified by Battelle to meet Data Quality objectives.
Refer to Section 3 of the "Ponce Harbor Section 103 Sediment Evaluation Work and Quality Assurance Plan", PPB, 2002

TABLE 4
Sampling Locations for Ponce Harbor, Reference Stations, and Control Sediment*

Area	Station ID	Actual Sampling Location		Distance Between Actual and Target Locations (m)
		Latitude	Longitude	
Harbor Stations	E-PH03-1	+17.97135	-066.61611	4.2
	E-PH03-2	+17.97244	-066.61851	19.6
	E-PH03-3	+17.97134	-066.62320	14.4
	E-PH03-4	+17.97023	-066.62083	5.3
	E-PH03-5	+17.96916	-066.62673	6.3
	E-PH03-6	+17.96680	-066.62318	20.6
	E-PH03-7	+17.96684	-066.62784	18.9
	E-PH03-8	+17.96458	-066.62553	14.0
	E-PH03-9	+17.96231	-066.62562	11.3
Reference Stations	RS-PH03A	+17.93622	-066.63529	11.6
	RS-PH03B	+17.93619	-066.63529	8.5
Control Sediment	E-PH03-Control	+29.6719	-081.2148	----

Note: RS-PHO3B moved to RS-PH03A due to lack of available sediment
* Geographical coordinates shown in decimal degrees NAD 83

TABLE 5
Station Specific Information for Ponce Harbor Sampling Locations

Station ID	Water Depths (ft)		Vibracore Lengths (ft)		
	Anticipated	Observed	Penetration	Recovered	Needed
E-PHO3-1	35.4	37.6	17.5	17.5	17.4
E-PHO3-2	39.9	42.0	13.5	12.0	13.0
E-PHO3-3	40.4	30.3	20.0	18.0	24.7
E-PHO3-4	39.2	40.0	12.0	12.0	15.0
E-PHO3-5	40.5	34.0	19.0	19.0	21.0
E-PHO3-6	39.0	39.1	17.0	14.0	15.9
E-PHO3-7	39.3	39.0	17.0	15.0	16.0
E-PHO3-8	40.7	40.0	16.0	13.5	15.0
E-PHO3-9	47.7	48.1	18.0	11.5*	6.9

Notes: Observed depth adjusted for mean low water
* indicates 7.0 feet were retained

TABLE 6
Water Quality Data from Ponce Harbor and Reference Stations, March 2003

Station ID	Date and Time (AST)	Water Depth (ft)	Temp (C°)	pH (units)	DO (mg/L)	Turbidity (NTU)	Salinity (ppt)
E-PH03-1	3/5/03-1130	38.1	23.0	7.84	7.31	7.1	35.4
E-PH03-2	3/06/03-1200	42.5	26.8	8.02	6.59	8.9	36.4
E-PH03-3	3/04/03-1425	30.5	27.1	7.98	7.56	2.5	35.5
E-PH03-4	3/04/03-1215	40.3	26.9	8.62	7.03	2.4	35.3
E-PH03-5	3/06/03-1042	34.4	26.9	8.08	6.40	6.1	36.0
E-PH03-6	3/06/03-0815	39.5	22.4	8.25	7.33	7.7	40.1
E-PH03-7	3/06/03-0942	39.1	26.9	8.13	6.52	6.0	36.1
E-PH03-8	3/05/03-1001	40.5	26.8	7.96	6.44	1.8	36.2
E-PH03-9	3/05/03-0830	48.5	26.5	8.06	6.18	2.0	36.6
RS-PH03A	3/04/03-1246	121	26.8	7.57	4.53	<1.0	36.1
RS-PH03B*	3/04/03-1552	107	26.8	7.71	5.50	<1.0	36.2

* = Unlike the sediment, field data were collected at the predetermined station location

TABLE 7
General Description of Sediments by Station from Ponce Harbor and Reference Locations, March 2003

Station ID	General Description of Sediment
Harbor Stations	
E-PH03-1	trace fine sand 0-1'; dark gray silty clay with occasional shell fragments
E-PH03-2	0-1½' trace fine sand; dark gray silty clay
E-PH03-3	dark gray silty clay
E-PH03-4	dark gray silty clay
E-PH03-5	0-2' fine sand and shell particles present, dark gray silty clay
E-PH03-6	0-1' trace fine sand and shell fragments, dark gray silty clay
E-PH03-7	0-2' fine sand and shell particles present, dark gray silty clay
E-PH03-8	0-1½' trace fine sand; dark gray silty clay
E-PH03-9	dark gray silty clay
Reference Stations	
RS-PH03A	soft fine silty mud
RS-PH03B*	very soft mud

Notes: Both referenced sediments were collected in the vicinity of RS-PH03A
"General descriptions" may combine and distill observations from one or more cores

TABLE 8
Physical Analysis and Total Organic Carbon Data for Ponce Harbor and Reference Sediments Collected March 2003

Sample ID	Grain Size Distribution (percent)				TOC (dry weight) percent	Percent Moisture
	Gravel >2.00 mm	Sand 2.00 - 0.0625 mm	Silt 0.0625 - 0.0039 mm	Clay <0.0039 mm		
Harbor Sediment						
E-PH03-1SC	0.7	7.5	57.7	34.1	0.78	34
E-PH03-2SC	<0.1	8.4	57.3	34.2	0.73	32
E-PH03-3SC	0.1	13.4	63.4	23.1	0.66	31
E-PH03-4SC	<0.1	4.7	64.1	31.2	0.69	32
E-PH03-5SC	0.6	11.3	64.8	23.4	0.69	30
E-PH03-6SC	0.1	5.9	65.1	28.9	0.81	34
E-PH03-7SC	<0.1	12.0	64.3	23.6	0.64	33
E-PH03-8SC	<0.1	12.1	65.5	22.4	0.63	31
E-PH03-9SC	0.3	11.1	59.0	29.6	0.63	38
E-PH03-SC*	0.3	9.3	62.2	28.1	0.69	34
Reference Sediment						
RS-PH03*	0.1	4.9	39.7	55.4	0.91	54
Control Sediment						
E-PH03-Control	15.5	73.6	3.4	7.5	0.64	30

Sample ID	Wet Bulk Density (g/cm³)	Dry Bulk Density (g/cm³)	Specific Gravity	Plastic Limit	Liquid Limit	Plasticity Index
E-PH03-SC*	1.82	1.21	2.75	46	27	19
RS-PH03*	1.42	0.65	2.74	77	30	47

Asterisk (*) indicates a composited sample.

TABLE 9
Sediment Metals Concentrations for Ponce Harbor and Reference Station for Composited Samples Collected March 2003

Sample ID	% Moisture	Al	Ag	As	Cd	Cr	Cu
RS-PH03	54.7	59900 B	0.03 U	7.65 B	0.0369	39.7 B	42.5
E-PH03-SC Replicate 1	32.7	70000 B	0.740	7.70 B	0.0620	65.0 B	47.0
E-PH03-SC Replicate 2	---	71700 B	0.0704	7.90 B	0.0588	65.7 B	48.8
E-PH03-SC Replicate 3	---	71000 B	0.111	7.95 B	0.0652	63.9 B	48.9
Mean	---	70900 B	0.307	7.85 B	0.0620	64.9 B	48.2
RSD	---	1%	NC	2%	5%	1%	2%
Laboratory Achieved Detection Limits	---	0.5	0.03	0.05	0.01	0.04	0.02
B flag criteria (3 * MDL)	---	1.5	0.09	0.15	0.03	0.12	0.06

Sample ID	Fe	Hg	Ni	Pb	Se	Zn
RS-PH03	44200	0.0447	20.1	9.33	0.273	64.9 B
E-PH03-SC Replicate 1	53000	0.0266	23.8	5.05	0.224	71.6 B
E-PH03-SC Replicate 2	54600	0.0272	24.6	5.11	0.247	73.4 B
E-PH03-SC Replicate 3	54100	0.0271	24.6	5.07	0.214	73.0 B
Mean	53900	0.0270	24.3	5.08	0.228	72.7 B
RSD	2%	1%	2%	1%	7%	1%
Laboratory Achieved Detection Limits	5	0.002	0.03	0.01	0.08	0.1
B flag criteria (3 * MDL)	15	0.006	0.09	0.03	0.24	0.3

Notes NC = Not calculated. QC sample not appropriate for evaluation of DQOs.
U = Undetected at or above MDL; MDL reported. B = Analyte detected in blank is greater than 3x MDL
* Metals concentrations are in µg/g units, dry weight basis

TABLE 10
Pesticide and PCB Congener Contained in Ponce Harbor and Reference Station Sediments Collected in March 2003

Pesticide	Reference Composite	Harbor Composite				
		Rep 1	Rep 2	Rep 3	Mean	%RSD
Aldrin	0.027 U	0.034 U	0.034 U	0.035 U	0.034 U	NA
a Chlordane	0.031 U	0.039 U	0.039 U	0.040 U	0.040 U	NA
trans Nonachlor	0.035 U	0.044 U	0.044 U	0.045 U	0.045 U	NA
Dieldrin	0.041 U	0.052 U	0.052 U	0.053 U	0.052 U	NA
4,4 DDT	0.063	0.050 U	0.050 U	0.051 U	0.050 U	NA
2,4 DDT	0.053 U	0.068 U	0.068 U	0.069 U	0.068 U	NA
4,4 DDD	0.070	0.044 J	0.045 J	0.046 J	0.045 J	2
2,4 DDD	0.045 U	0.058 U	0.058 U	0.059 U	0.058 U	NA
4,4 DDE	0.320	0.022 J	0.029 J	0.029 J	0.027 J	15
2,4 DDE	0.057 U	0.072 U	0.072 U	0.073 U	0.073 U	NA
Endosulfan I	0.043 U	0.055 U	0.055 U	0.056 U	0.055 U	NA
Endosulfan II	0.042 U	0.054 U	0.054 U	0.054 U	0.054 U	NA
Endosulfan sulfate	0.044 U	0.056 U	0.056 U	0.057 U	0.056 U	NA
Heptachlor	0.035 U	0.044 U	0.044 U	0.045 U	0.045 U	NA
Heptachlor epoxide	0.032 U	0.041 U	0.041 U	0.042 U	0.041 U	NA
PCB Congener						
PCB 08	0.070 U	0.089 U	0.089 U	0.090 U	0.089 U	NA
PCB 18	0.033 U	0.042 U	0.042 U	0.043 U	0.042 U	NA
PCB 28	0.048 U	0.061 U	0.061 U	0.062 U	0.062 U	NA
PCB 44	0.043 U	0.055 U	0.055 U	0.056 U	0.055 U	NA
PCB 49	0.050 U	0.064 U	0.064 U	0.065 U	0.064 U	NA
PCB 52	0.045 U	0.058 U	0.058 U	0.059 U	0.058 U	NA
PCB 66	0.051 U	0.064 U	0.064 U	0.065 U	0.065 U	NA
PCB 87	0.037 U	0.048 U	0.048 U	0.048 U	0.048 U	NA
PCB 101	0.044 U	0.056 U	0.056 U	0.056 U	0.056 U	NA
PCB 105	0.038 U	0.049 U	0.049 U	0.049 U	0.049 U	NA
PCB 118	0.050 U	0.063 U	0.063 U	0.064 U	0.064 U	NA

TABLE 10
Pesticide and PCB Congener Contained in Ponce Harbor and Reference Station Sediments Collected in March 2003

Pesticide	Reference Composite	Harbor Composite				%RSD
		Rep 1	Rep 2	Rep 3	Mean	
PCB 128	0.081 U	0.103 U	0.103 U	0.105 U	0.103 U	NA
PCB 138	0.041 U	0.053 U	0.053 U	0.054 U	0.053 U	NA
PCB 153	0.062 U	0.018 J	0.018 J	0.022 J	0.019 J	12
PCB 170	0.041 U	0.052 U	0.052 U	0.053 U	0.053 U	NA
PCB 180	0.044 U	0.055 U	0.055 U	0.056 U	0.056 U	NA
PCB 183	0.039 U	0.049 U	0.049 U	0.050 U	0.049 U	NA
PCB 184	0.031 U	0.039 U	0.039 U	0.039 U	0.039 U	NA
PCB 187	0.040 U	0.051 U	0.051 U	0.052U	0.051U	NA
PCB 195	0.038 U	0.048 U	0.048 U	0.049 U	0.048 U	NA
PCB 206	0.037 U	0.047 U	0.047 U	0.048 U	0.047 U	NA
PCB 209	0.038 U	0.051 U	0.051 U	0.052 U	0.051 U	NA

Notes: All values are reported in units of ng/g, dry weight basis
U = analyte not detected, sample specific method detection limit (ssMDL) inserted
J = analyte detected below the ssMDL
"Rep" indicates replicate sample
NA = not applicable
%RSD = present relative standard deviation

TABLE 11
PAH Compounds Contained in Ponce Harbor and Reference Station Sediments Collected in March 2003

PAH Compound	Reference Composite	Harbor Composite				Percent RSD
		Rep 1	Rep 2	Rep 3	Mean	
Naphthalene	5.63	1.60 J	1.38 J	2.14 J	1.71 J	22.7
Biphenyl	0.72	0.36	0.34	0.46	0.39	16.6
Acenaphthylene	0.86	0.22	0.19	0.54	0.32	61.6 &
Acenaphthene	0.38	0.17	0.15	0.20	0.17	14.6
Fluorene	0.38	0.24	0.22	0.27	0.24	10.8
Phenanthrene	4.81	1.25	1.14	1.40	1.26	10.2
Anthracene	0.87	0.29	0.28	0.40	0.32	19.4
Fluoranthene	8.35	1.57	1.48	1.89	1.65	13.0
Pyrene	9.22	1.78	1.66	2.85	2.09	31.4 &
Benzo(a)anthracene	4.84	0.78	0.62	1.39	0.93	44.0 &
Chrysene	5.44	1.08	0.90	2.29	1.42	53.2 &
Benzo(b)fluoranthene	5.86	1.31	1.03	2.24	1.53	41.8 &
Benzo(k)fluoranthene	5.08	0.99	0.80	2.37	1.39	61.9 &
Benzo(a)pyrene	5.75	1.05	0.77	2.72	1.51	69.7 &
Indeno(1,2,3-c,d)pyrene	4.12	0.64	0.47	1.37	0.83	57.6 &
Dibenz(a,h)anthracene	0.79	0.15	0.12	0.27	0.18	44.2 &
Benzo(g,h,i)perylene	4.29	0.83	0.71	1.57	1.04	44.6 &

Notes: All values are reported in units of ng/g, dry weight basis
& = QC value outside the accuracy or precision range
J = analyte detected below the ssMDL
"Rep" indicates replicate sample
NA = not applicable
%RSD = present relative standard deviation

TABLE 12
Mean Metals Concentrations of Triplicate Analyses of Ponce Harbor Site Water and Elutriate Samples Generated from the March 2003 Collection Effort*

Sample ID	As	Cd	Cr	Cu	Pb
Harbor Site Water (mean)	0.908	0.01 U	0.300	2.70	0.0511
Elutriate Sample (mean)	10.2	0.0114	0.573	1.27	0.0853
% RSD Harbor	3	NA	3	1	2
% RSD Elutriate	4	2	2	2	7
Laboratory Achieved Detection Limits	0.05	0.01	0.04	0.02	0.01
Sample ID	**Hg**	**Ni**	**Se**	**Ag**	**Zn**
Harbor Site Water (mean)	0.000565	0.357	0.5 U	0.0289	4.30
Elutriate Sample (mean)	0.000846	0.998	1.14	0.0295	1.63
% RSD Harbor	2	0	NA	4	4
% RSD Elutriate	14	3	14	7	12
Laboratory Achieved Detection Limits	0.0002	0.04	0.5	0.0128	0.1

Notes: NA = Not Applicable. U = Undetected at or above MDL; MDL reported
%RSD = percent relative standard deviation
* Metals concentrations are in µg/L

TABLE 13
Mean Pesticide and PCB Congener Concentrations in Water and Elutriate Samples Generated from the Ponce Harbor Sampling in March 2003

Pesticide	Harbor Water	Elutriate Samples	Harbor Percent RSD	Elutriate Percent RSD
Aldrin	2.83 U	2.83 U	NA	NA
a Chlordane	1.08 U	1.08 U	NA	NA
trans Nonachlor	1.01 U	1.01 U	NA	NA
Dieldrin	0.978 U	0.978 U	NA	NA
4,4 DDT	0.557 U	0.557 U	NA	NA
2,4 DDT	1.98 U	1.98 U	NA	NA
4,4 DDD	0.595 U	0.595 U	NA	NA
2,4 DDD	0.747 U	0.747 U	NA	NA
4,4 DDE	0.838 U	0.838 U	NA	NA
2,4 DDE	1.71 U	1.71 U	NA	NA
Endosulfan I	1.11 U	1.11 U	NA	NA
Endosulfan II	0.510 U	0.510 U	NA	NA
Endosulfan sulfate	0.565 U	0.565 U	NA	NA
Heptachlor	1.17 U	1.17 U	NA	NA
Heptachlor epoxide	0.948 U	0.948 U	NA	NA
PCB Congener				
PCB 08	16.0 U	16.0 U	NA	NA
PCB 18	1.38 U	1.38 U	NA	NA
PCB 28	1.73 U	1.73 U	NA	NA
PCB 44	1.45 U	1.45 U	NA	NA
PCB 49	1.49 U	1.49 U	NA	NA
PCB 52	1.44 U	1.44 U	NA	NA
PCB 66	1.49 U	1.49 U	NA	NA
PCB 87	1.13 U	1.13 U	NA	NA
PCB 101	1.14 U	1.14 U	NA	NA
PCB 105	0.575 U	0.575 U	NA	NA
PCB 118	0.868 U	0.868 U	NA	NA

TABLE 13
Mean Pesticide and PCB Congener Concentrations in Water and Elutriate Samples Generated from the Ponce Harbor Sampling in March 2003

Pesticide	Harbor Water	Elutriate Samples	Harbor Percent RSD	Elutriate Percent RSD
PCB 128	1.40 U	1.40 U	NA	NA
PCB 138	1.32 U	1.32 U	NA	NA
PCB 153	1.06 U	1.06 U	NA	NA
PCB 170	1.02 U	1.02 U	NA	NA
PCB 180	0.960 U	0.960 U	NA	NA
PCB 183	0.930 U	0.930 U	NA	NA
PCB 184	0.918 U	0.918 U	NA	NA
PCB 187	0.862 U	0.862 U	NA	NA
PCB 195	1.08 U	1.08 U	NA	NA
PCB 206	1.22 U	1.22 U	NA	NA
PCB 209	1.26 U	1.26 U	NA	NA

Notes: All values are reported in units of ng/L
U = analyte not detected, sample-specific method detection limit (ssMDL) inserted
NA = not applicable
Percent RSD = present relative standard deviation

TABLE 14
96-Hour *Mysidopsis bahia* Survival in Three Elutriate Concentrations Prepared from Sediments Collected from Ponce Harbor, March 2003

Sample	Replicate	Number of Survivors[a]		Percent (?)
Control	A	19		
Seawater	B	18		
	C	18		
	D	19		
	E	19		
Total		93		
Percent		93		
Sample	**Replicate**		**Elutriate Concentration**	
		10	50	100
Control Sediment	A	19	17	19
	B	18	19	20
	C	20	20	20
	D	19	19	19
	E	20	20	19
Total		96	95	97
Percent		96	95	97
E-PH03-SC	A	19	19	19
	B	19	20	16
	C	20	19	19
	D	20	18	17
	E	19	19	16
Total		97	95	87
Percent		97	95	87

[a] - Based upon 100 organisms exposed

Source: Harding ESE, now known as MACTEC

TABLE 15
Summary of ANOVA and Dunnett's Tests of Control Seawater and ANOVA and Two Sample t-Test for Control Sediment and Test Sediment Survival for Ponce Harbor Elutriate of *Mysidopsis bahia*, April 2003

***Mysidopsis bahia* 100 percent Elutriates vs. Control Seawater**

ANOVA for Differences Between Means

Source of Variation	df	Sum of Squares	Mean Square	F
Between Means	2	10.13	5.07	5.24
Within Means	12	11.60	0.97	
Total	14	21.73		

Critical F = 3.89 (a = 0.05, df = 2,12)
Since F is > Critical F reject H_o: all groups equal; with a = 0.05, and 2,12 df

Dunnett's Test
Critical value = 2.11

Sample ID	*a* = 0.05	Difference between means	T Stat
Control Seawater			
E-PH03-Control	=	-0.80	-1.29
E-PH03-SC	=	1.20	1.93

= Indicates no significant difference between the sample station and the control seawater
* Indicates a significant difference exists between the sample station and the control seawater

Summary of ANOVA and Dunnett's Tests of Control Seawater and ANOVA and Two Sample t-Test for Control Sediment and Test Sediment Survival for Ponce Harbor Elutriate of *Mysidopsis bahia*, April 2003

***Mysidopsis bahia* 100 Percent Elutriates vs. Control Sediment**

ANOVA for Differences Between Means

Source of Variation	df	Sum of Squares	Mean Square	F
Between Means	1	10.00	10.00	7.69
Within Means	8	10.40	1.30	
Total	9	20.40		

Critical F = 5.32 (a = 0.05, df = 1,8)
Since F is > Critical F reject H_o: all groups equal; with a = 0.05, and 1,8 df

2 Sample t-Test

Critical T = 1.86

Sample ID	*a* = 0.05	Difference Between Means	T Stat
E-PH03-CONTROL		-	-
E-PH03-SC	*	2.00	2.77

= Indicates no significant difference between the sample station and the reference sediment
* Indicates a significant difference exists between the sample station and the reference sediment

TABLE 16
LC_{50} or EC_{50} (*Mysidopsis bahia*, *Menidia beryllina* and *Mytilus edulis*) Values[a] for Elutriate Bioassays Conducted for Ponce Harbor with Sediment Collected in March 2003

Sample ID	*Mysidopsis bahia*	*Menidia beryllina*	*Mytilus edulis*
E-PH03-CONTROL	>100	>100	>100
E-PH03-SC	>100	>100	>100
RS-PH03	>100	>100	>100

[a] LC_{50} values recorded as greater than 100% had greater than 50% survivorship. In the case of *Mytilus edulis*, an EC_{50} value greater than 100% indicated development greater than 50%.

TABLE 17

96-Hour *Menidia beryllina* Survival in Three Elutriate Concentrations Prepared from Sediments Collected from Ponce Harbor, Puerto Rico, in March 2003

Sample	Replicate	Number of Survivors[a]		Percent
Control	A	20		
Seawater	B	20		
	C	20		
	D	20		
	E	19		
Total		99		
Percent		99%		
Sample	**Replicate**		**Elutriate Concentration**	
		10%	50%	100%
E-PH03-Control	A	19	20	19
Sediment	B	20	20	20
	C	19	20	18
	D	20	19	19
	E	19	17	19
Total		97	96	95
Percent		97%	96%	95%
E-PH03-SC	A	19	12	14
	B	19	19	16
	C	18	16	18
	D	19	18	14
	E	19	20	10
Total		94	85	72
Percent		94%	85%	72%

a - Based upon 100 organisms exposed.

Source: Harding ESE, 2003, now known as MACTEC

TABLE 18
Summary of Kruskal – Wallis ANOVA by Ranks of Control Seawater and ANOVA with Two Sample t-Test for Control Sediment and Test Sediment Survival for Ponce Harbor Elutriate of *Menidia beryllina*, for Sediments Collected in March 2003

***Menidia beryllina* 100% Elutriate vs. Control Seawater**

Kruskal – Wallis ANOVA by Ranks

Sample	Transformed Mean	Rank Sum
Control Seawater	0.0505	19.50
E-PH03-SC	0.0722	64.50
E-PH03-Control	0.0527	36.00

Critical H = 5.78 (a = 0.05, df = 2,)
Since Calc H is > Critical H reject H_o: all groups equal; with a = 0.05, and 2 df

Dunn's Multiple Comparison

Table q value = 2.3940

Sample ID	a = 0.05
Control Seawater	=
E-PH03-SC	*
E-PH03-CONTROL	=

= Indicates no significant difference between the sample station and the control seawater
* Indicates a significant difference exists between the sample station and the control seawater.

***Menidia beryllina* 100% Elutriate vs. Control Sediment**

ANOVA for Differences Between Means

Source of Variation	df	Sum of Squares	Mean Squares	F
Between Means	1	52.90	52.90	11.38
Within Means	8	37.20	4.65	
Total	9	90.10		

Critical F = 5.32 (a = 0.05, df = 1,8)
Since F is > Critical F reject H_o: all groups equal; with a = 0.05, and 1,8 df.

2 Sample t-Test

Critical T = 1.86

Sample ID	a = 0.05	Difference between Means	T Stat
E-PH03-CONTROL		-	-
E-PH03-SC	*	4.60	3.37

= Indicates no significant difference between the sample station and the reference sediment
* Indicates a significant difference exists between the sample station and the reference sediment

TABLE 19
48-Hour *Mytilus edilus* Development in Three Elutriate Concentrations Prepared from Sediments Collected in and near Ponce Harbor, Puerto Rico, March 2003

Sample	Replicate	Percentage of Full Development[a]		Percent
Control	A	72		
Seawater	B	80		
	C	79		
	D	71		
	E	75		
Average Percentage		75%		
Sample	**Replicate**		**Elutriate Concentration**	
		10%	50%	100%
E-PH03-CONTROL	A	70	75	68
	B	66	81	62
	C	80	62	77
	D	72	70	67
	E	71	69	70
	Average Percentage	72%	71%	69%
E-PH03-SC	A	76	73	60
	B	62	78	62
	C	75	63	58
	D	68	68	67
	E	80	68	54
Average Percentage		72%	70%	60%

[a] – Expressed as percentage of mussel embryos resulting in live larvae with complete shell development.

Source: Harding ESE, now known as MACTEC

TABLE 20
Summary of ANOVA and Dunnett's Tests of Control Seawater and ANOVA and Two Sample t-Test for Control Sediment and Test Sediment Larval Development for Ponce Harbor Elutriate of *Mytilus edulis* for Sediments Collected in March 2003

***M. edulis* 100 percent Elutriates vs. Control Seawater**

ANOVA for Differences Between Means

Source of Variation	df	Sum of Squares	Mean Square	F
Between Means	2	580.93	290.47	12.59
Within Means	12	276.80	23.07	
Total	14	857.73		

Critical F = 3.89 (a = 0.05, df = 2,12)
Since F is > Critical F reject H_o: all groups equal; with a = 0.05, and 2,12 df.

Dunnett's Test

Critical value = 2.11

Sample ID	a = 0.05	Difference Between Means	T Stat
Control Seawater	–	–	
E-PH03-Control	*	6.6	2.17
E-PH03-SC	*	15.2	5.00

= Indicates no significant difference between the sample station and the control seawater
* Indicates a significant difference exists between the sample station and the control seawater

***Mytilus edulis* 100 percent Elutriates vs. Control Sediment**

ANOVA for Differences Between Means

Source of Variation	df	Sum of Squares	Mean Square	F
Between Means	1	184.90	184.90	6.99
Within Means	8	211.60	26.45	
Total	9	396.50		

Critical F = 5.32 (a = 0.05, df = 1,8)
Since F is > Critical F reject H_o: all groups equal; with a = 0.05, and 1,8 df..

2 Sample t-Test

Critical T = 1.86

Sample ID	a = 0.05	Difference Between Means	T Stat
E-PH03-CONTROL	–	–	-
E-PH03-SC	*	8.60	2.64

= Indicates no significant difference between the sample station and the reference sediment
* Indicates a significant difference exists between the sample station and the reference sediment

TABLE 21
10-Day Sediment *Mysidopsis bahia* Survival for Ponce Harbor Sediments Collected in March 2003

Sample	Replicate	Number of Survivors
E-PH03 – Control	A	19
	B	18
	C	20
	D	20
	E	19
Total		96
Percent		96
RS-PH03	A	17
	B	19
	C	19
	D	17
	E	20
Total		92
Percent		92
E-PH03-SC	A	20
	B	20
	C	20
	D	17
	E	19
Total		96
Percent		96

[a] - Based upon 100 organisms exposed.

Source: Harding ESE, now known as MACTEC

TABLE 22
Summary of ANOVA, Dunnett's, and Two Sample t-Tests of Control Sediment or Reference Sediment and Test Sediment Survival for Ponce Harbor Whole Sediment of *Mysidopsis bahia*, for Sediments Collected in March 2003

***Mysidopsis bahia* Whole Sediment vs. Control**

ANOVA for Differences Between Means

Source of Variation	df	Sum of Squares	Mean Square	F
Between Means	2	2.13	1.07	0.76
Within Means	12	16.80	1.40	
Total	14	18.93		

Critical F = 3.89 (a = 0.05, df = 2,12)
Since F is < Critical F fail to reject H_o: all groups equal; with a = 0.05, and 2,12 df.

Dunnett's Test

Critical T = 2.11

Sample ID	a = 0.05	Difference Between Means	T Stat
E-PH03-CONTROL		–	–
E-PH03-SC	=	0.00	0.00
RS-PH03	=	0.80	1.07

= Indicates no significant difference between the sample station and the control sediment
* Indicates a significant difference exists between the sample station and the control sediment.

***Mysidopsis bahia* Reference Sediment vs. Control**

ANOVA for Differences Between Means

Source of Variation	df	Sum of Squares	Mean Square	F
Between Means	1	1.60	1.60	0.91
Within Means	8	14.00	1.75	
Total	9	15.60		

Critical F = 5.32 (a = 0.05, df = 1,8)
Since F is < Critical F fail to reject H_o: all groups equal; with a = 0.05, and 1,8 df

2 Sample t-Test

Critical T = 1.86

Sample ID	a = 0.05	Difference Between Means	T Stat
RS-PH03		–	–
E-PH03-SC	=	-0.80	-0.96

= Indicates no significant difference between the sample station and the reference sediment
* Indicates a significant difference exists between the sample station and the reference sediment

TABLE 23
10-Day Sediment *Leptocheirus plumulosus* Survival for Ponce Harbor Sediments Collected in March 2003

Sample	Replicate	Number of Survivors[a]
PH03 - Control	A	19
	B	17
	C	-
	D	20
	E	20
Total		76
Percent		95
RS-PH03	A	18
	B	19
	C	20
	D	19
	E	19
Total		95
Percent		95
E-PH03- SC	A	16
	B	16
	C	17
	D	17
	E	18
Total		84
Percent		84

[a] - Based upon 100 organisms exposed, except sample PH03-Control, based on 80 organisms exposed as Replicate C test vessel was broken during testing.

SOURCE: Harding ESE, 2003, now known as MACTEC

TABLE 24
Summary of ANOVA, Bonferroni, and 2 Sample t-Tests of Control Sediment or Reference Sediment and Test Sediment Survival for Ponce Harbor Whole Sediment of *Leptocherirus plumulosus*, for Sediments Collected in March 2003

***L. plumulosus* Whole Sediment vs. Control**

ANOVA for Differences Between Means

Source of Variation	df	Sum of Squares	Mean Square	F
Between Means	2	15.56	7.78	7.92
Within Means	11	10.80	0.98	
Total	13	26.36		

Critical F = 3.98 (a = 0.05, df = 2,11)
Since F is > Critical F reject H_o: all groups equal; with a = 0.05, and 2,11 df.

Bonferroni Test

Critical T = 2.20

Sample ID	a = 0.05	Difference Between Means	T Stat
E-PH03-CONTROL		–	–
E-PH03-SC	*	2.20	3.31
RS-PH03	=	–	–

= Indicates no significant difference between the sample station and the control sediment
* Indicates a significant difference exists between the sample station and the control sediment.

Source of Variation	df	Sum of Squares	Mean Square	F
Between Means	1	12.10	12.10	20.17
Within Means	8	4.80	0.60	
Total	9	16.90		

Critical F = 5.32 (a = 0.01, df = 1,8)
Since F is > Critical F reject H_o: all groups equal; with a = 0.05, and 1,8 df.

2 Sample t-Test

Critical T = 2.11

Sample ID	a = 0.05	Difference Between Means	T Stat
RS-PH03	=	–	–
E-PH03-SC	*	2.20	4.49

= Indicates no significant difference between the sample station and the reference sediment
* Indicates a significant difference exists between the sample station and the reference sediment

TABLE 25
Survivorship of *Macoma nasuta* and *Nereis virens* During 28-Day Bioaccumulation Bioassays with Sediments from Ponce Harbor Collected in March 2003

Sample ID	Replicate	*Macoma nasuta*[a]	*Nereis virens*[a]
E-PH03 -Control Sediment	A	18	20
	B	18	19
	C	19	18
	D	19	18
	E	18	20
	Total	92	95
	Percent	92	95
RS-PH03	A	18	20
	B	18	19
	C	20	20
	D	19	17
	E	19	19
	Total	94	95
	Percent	94	95
E-PH03-SC	A	16	16
	B	18	20
	C	18	16
	D	19	18
	E	18	17
	F	16	20
	G	17	17
	H	18	16
	I	16	18
	Total	156	158
	Percent	87	88

[a] – based upon 100 organisms, except for sample E-PH03-SC for which extra replicates were exposed to ensure sufficient tissue for triplicates, spikes, etc.

TABLE 26
Comparison of Tissue Chemical Concentrations of *Macoma nasuta* for Organisms Exposed to Ponce Harbor and Reference Station Sediments that were Collected March 2003

Chemical Constituent	Mean Concentration in Tissue (ng/g, wet weight basis)			Harbor Lower Confidence Limit	Reference Upper Confidence Limit	t-test Harbor Exceeds Reference
	Harbor	Reference	Pretest			
PAHs						
1,4-Dichlorobenzene	0.0680	0.0820	0.470	0.0580	0.0920	NSE
Naphthalene	0.300	0.350	0.880	0.250	0.461	NSE
Biphenyl	0.0780	0.0820	0.110	0.0600	0.116	NSE
Acenaphthylene	0.0150	0.0150	0.0150	0.0150	0.0150	NSE
Acenaphthene	0.0250	0.0280	0.0600	0.0250	0.0360	NSE
Fluorene	0.0660	0.0840	0.110	0.0550	0.118	NSE
Phenanthrene	0.372	0.502	0.660	0.333	0.663	NSE
Anthracene	0.0330	0.0330	0.0600	0.0240	0.0430	NSE
Fluoranthene	1.07	1.45	3.24	0.849	2.03	NSE
Pyrene	0.732	0.902	1.84	0.665	1.57	NSE
Benzo(a)anthracene	0.0980	0.138	0.200	0.0660	0.194	NSE
Chrysene	0.520	0.624	0.850	0.428	0.800	NSE
Benzo(b)fluoranthene	0.140	0.194	0.230	0.117	0.238	NSE
Benzo(k)fluoranthene	0.113	0.222	0.180	0.113	0.222	NSE
Benzo(a)pyrene	0.104	0.106	0.120	0.0870	0.138	NSE
Indeno(1,2,3cd)pyrene	0.0290	0.0270	0.0350	0.0260	0.0300	NSE
Dibenz(a,h)anthracene	0.0250	0.0250	0.0250	0.0250	0.0250	NSE
Benzo(g,h,i)perylene	0.0330	0.0380	0.0400	0.0180	0.0480	NSE
Metals						
Ag	0.0120	0.0140	0.0125	0.0100	0.0180	NSE
As	1.81	1.91	1.80	1.59	2.47	NSE
Cd	0.0190	0.0190	0.0286	0.0150	0.0270	NSE
Cr	0.211	0.155	0.142	0.0110	0.248	NSE
Cu	1.15	1.11	7.50	0.650	1.55	NSE
Hg	0.00700	0.00700	0.00859	0.00600	0.0120	NSE
Ni	0.287	0.256	0.486	0.177	0.336	NSE
Pb	0.0820	0.0890	0.0930	0.0510	0.128	NSE
Se	0.285	0.281	0.300	0.268	0.367	NSE
Zn	6.25	7.13	9.80	5.51	9.46	NSE

TABLE 27
Comparison of Tissue Chemical Concentrations of *Nereis virens* for Organisms Exposed to Ponce Harbor and Reference Station Sediments that were Collected March 2003

Chemical Constituent	Mean Concentration in Tissue (ng/g, wet weight basis)			Harbor Lower Confidence Limit	Reference Upper Confidence Limit	t-test Harbor Exceeds Reference
	Harbor	Reference	Pretest			
PAHs						
1,4-Dichlorobenzene	0.354	2.03	1.72	0.271	2.68	NSE
Naphthalene	1.62	2.59	2.47	0.935	3.59	NSE
Biphenyl	0.222	0.262	0.293	0.144	0.338	NSE
Acenaphthylene	0.0940	0.140	0.123	0.0460	0.210	NSE
Acenaphthene	0.236	0.218	0.250	0.0680	0.309	NSE
Fluorene	0.648	0.232	0.287	-0.653	0.295	NSE
Phenanthrene	2.42	1.43	1.91	-1.78	1.93	NSE
Anthracene	1.57	0.194	0.247	-2.45	0.321	NSE
Fluoranthene	2.81	3.78	4.34	1.61	5.00	NSE
Pyrene	2.47	4.05	4.81	1.73	5.11	NSE
Benzo(a)anthracene	0.680	0.956	1.37	0.163	1.66	NSE
Chrysene	2.65	2.29	2.71	-0.381	3.05	NSE
Benzo(b)fluoranthene	0.656	1.05	1.19	0.568	1.52	NSE
Benzo(k)fluoranthene	0.902	1.19	1.70	0.712	1.60	NSE
Benzo(a)pyrene	0.626	1.19	1.44	0.547	1.77	NSE
Indeno(1,2,3cd)pyrene	0.432	0.730	0.973	0.391	1.09	NSE
Dibenz(a,h)anthracene	0.0580	0.084	0.113	0.0440	0.138	NSE
Benzo(g,h,i)perylene	0.596	0.874	1.18	0.511	1.20	NSE
Metals						
Ag	0.0230	0.0350	0.0218	0.0190	0.0420	NSE
As	0.921	1.75	1.29	0.759	2.15	NSE
Cd	0.0260	0.0360	0.0231	0.0210	0.0440	NSE
Cr	0.622	1.13	0.920	0.307	1.49	NSE
Cu	55.5	66.6	1.67	33.2	98.2	NSE
Hg	0.0220	0.0370	0.0349	0.0180	0.0430	NSE
Ni	0.470	0.783	0.426	0.342	1.05	NSE
Pb	0.283	0.852	2.77	0.218	1.59	NSE
Se	0.367	0.524	0.407	0.313	0.664	NSE
Zn	13.0	22.3	10.1	6.72	35.1	NSE

TABLE 28
Detectable Chemical Concentrations from the Ponce Reference Station Compared to the 90 percent Confidence Limits for Chemical Levels Found in Ponce Harbor from Sediment Samples Collected in March 2003*

Analyte	Single Value for Reference Station	Mean Value for Harbor Triplicates	90 percent Confidence Limit for Harbor Replicates	Reference Sediment Value Compared to Harbor 90 percent Confidence Limit		
				Less Than	Within Limits	Greater Than
Metals						
Aluminum	59,900 B	70,900 B	69,500 B-72,300 B			XXX
Arsenic	7.65 B	7.85 B	7.63 B - 8.07 B		XXX	
Cadmium	0.0369	0.0620	0.0566 - 0.0674			XXX
Chromium	39.7 B	64.9 B	63.3 B - 66.4 B			XXX
Copper	42.5	48.2	46.4 - 50.0			XXX
Iron	44,200	53,900	52,500 - 55,300			XXX
Lead	9.33	5.08	5.03 - 5.13	XXX		
Mercury	0.0447	0.0270	0.0264 - 0.0275	XXX		
Nickel	20.1	24.3	23.6 - 25.1			XXX
Selenium	0.273	0.228	0.200 - 0.257	XXX		
Silver	0.03 U	0.307	Not Calculated &	---	---	---
Zinc	64.9 B	72.7 B	71.1 B - 74.3 B			XXX
Pesticides						
4,4'-DDT	0.063	0.050 U	0.050 U	---	---	---
4,4'-DDD	0.070	0.045 J	0.043 J - 0.047 J	XXX		
4,4'-DDE	0.320	0.027 J	0.020 J - 0.034 J	XXX		
PCB Congener						
PCB 153	0.062 U	0.019 J	0.015 J - 0.023 J	---	---	---
PAHs						
Naphthalene	5.63	1.71 J	1.05 J - 2.36 J	XXX		
Biphenyl	0.72	0.39	0.28 - 0.50	XXX		
Acenaphthylene	0.86	0.32	-0.01 - 0.65 &	XXX		
Acenaphthene	0.38	0.17	0.13 - 0.22	XXX		
Fluorene	0.38	0.24	0.20 - 0.28	XXX		
Phenanthrene	4.81	1.26	1.05 - 1.48	XXX		
Anthracene	0.87	0.32	0.22 - 0.43	XXX		
Fluoranthene	8.35	1.65	1.28 - 2.01	XXX		
Pyrene	9.22	2.09	0.98 - 3.20 &	XXX		
Benzo(a)anthracene	4.84	0.93	0.24 - 1.62 &	XXX		
Chrysene	5.44	1.42	0.15 - 2.70 &	XXX		
Benzo(b)fluoranthene	5.86	1.53	0.45 - 2.60 &	XXX		
Benzo(k)fluoranthene	5.08	1.39	-0.06 - 2.83 &	XXX		
Benzo(a)pyrene	5.75	1.51	-0.27 - 3.29 &	XXX		

TABLE 28
Detectable Chemical Concentrations from the Ponce Reference Station Compared to the 90 percent Confidence Limits for Chemical Levels Found in Ponce Harbor from Sediment Samples Collected in March 2003*

Analyte	Single Value for Reference Station	Mean Value for Harbor Triplicates	90 percent Confidence Limit for Harbor Replicates	Reference Sediment Value Compared to Harbor 90 percent Confidence Limit		
				Less Than	Within Limits	Greater Than
Indeno(1,2,3-c,d)pyrene	4.12	0.83	0.02 - 1.63 &	XXX		
Dibenz(a,h)anthracene	0.79	0.18	0.05 - 0.32 &	XXX		
Benzo(g,h,i)perylene	4.29	1.04	0.26 - 1.82 &	XXX		

Notes:

1 - B indicates analyte found in the samples at a level less than 3 times the method detection limit
2 - J indicates estimated value of analyte detected below the method detection limit
3 - U indicates analyte not detected and the method detection limit has been reported
4 - & indicates the Quality Control limits for precision were exceeded for these replicates

* = All values expressed as ng/g, dry weight basis

TABLE 29
Target and Achieved Detection Limits for Ponce Harbor and Reference Sediments*

Analyte	Detection Limit			
	Work/QAP Target	USEPA Guidance	Achieved Approx.	Units
1. Metals				
a. Aluminum	0.5		0.5	µg/g
b. Arsenic	0.05	0.1	0.05	µg/g
c. Cadmium	0.01	0.01	0.01	µg/g
d. Chromium	0.04	0.02	0.04	µg/g
e. Copper	0.02	0.1	0.02	µg/g
f. Iron	5	0.1	5	µg/g
g. Lead	0.01	0.1	0.01	µg/g
h. Mercury	0.002	0.02 (wet weight)	0.002	µg/g
i. Nickel	0.03	0.1	0.03	µg/g
j. Selenium	0.08	---	0.08	µg/g
k. Silver	0.03	0.1	0.03	µg/g
l. Zinc	0.1	0.1	0.1	µg/g
2. Organic Compounds				
a. Pesticides				
1) Aldrin	0.01	1	0.04	ng/g
2) Chlordane	0.01	1	0.05	ng/g
3) 2, 4' –DDT	0.08	1	0.08	ng/g
4) 2, 4' –DDD	0.01	1	0.07	ng/g
5) 2, 4' -DDE	0.07	1	0.09	ng/g
6) 4, 4' –DDT	0.02	1	0.06	ng/g
7) 4, 4' –DDD	0.01	1	0.06	ng/g
8) 4, 4' –DDE	0.02	1	0.06	ng/g
9) Dieldrin	0.03	1	0.06	ng/g
10) Endosulfan I	0.03	1	0.07	ng/g
11) Endosulfan II	0.01	1	0.06	ng/g
12) Endosulfan Sulfate	0.01	1	0.07	ng/g
13) Heptachlor	0.02	1	0.05	ng/g
14) Heptachlor Epoxide	0.02	1	0.05	ng/g
15) Trans nonachlor	0.01	1	0.05	ng/g
b. Polychlorinated Biphenyls Congeners				
1) PCB 8	0.1	1	0.11	ng/g
2) PCB 18	0.01	1	0.05	ng/g
3) PCB 28	0.01	1	0.07	ng/g
4) PCB 44	0.02	1	0.06	ng/g
5) PCB 49	0.01	1	0.08	ng/g
6) PCB 52	0.01	1	0.07	ng/g

TABLE 29
Target and Achieved Detection Limits for Ponce Harbor and Reference Sediments*

Analyte	Detection Limit			
	Work/QAP Target	USEPA Guidance	Achieved Approx.	Units
7) PCB 66	0.02	1	0.08	ng/g
8) PCB 87	0.01	1	0.06	ng/g
9) PCB 101	0.01	1	0.07	ng/g
10) PCB 105	0.02	1	0.06	ng/g
11) PCB 118	0.02	1	0.08	ng/g
12) PCB 128	0.02	1	0.12	ng/g
13) PCB 138	0.02	1	0.06	ng/g
14) PCB 153	0.02	1	0.09	ng/g
15) PCB 170	0.02	1	0.06	ng/g
16) PCB 180	0.02	1	0.07	ng/g
17) PCB 183	0.02	1	0.06	ng/g
18) PCB 184	0.02	1	0.05	ng/g
19) PCB 187	0.02	1	0.06	ng/g
20) PCB 195	0.02	1	0.06	ng/g
21) PCB 206	0.02	1	0.06	ng/g
22) PCB 209	0.02	1	0.06	ng/g
c. Total Organic Carbon	0.01	0.01	All Detected %	
d. PAHs				
1) Acenaphthene	0.04	10	0.04	ng/g
2) Acenaphthylene	0.04	10	0.07	ng/g
3) Anthracene	0.03	10	0.06	ng/g
4) Benzo (a) anthracene	0.05	10	0.04	ng/g
5) Benzo (a) pyrene	0.1	10	0.06	ng/g
6) Benzo (b) fluoranthene	0.04	10	0.05	ng/g
7) Benzo (k) fluoranthene	0.03	10	0.03	ng/g
8) Benzo (g,h,i) perylene	0.05	10	0.03	ng/g
9) Biphenyl	0.05	---	0.04	ng/g
10) Chrysene	0.04	10	0.03	ng/g
11) Dibenzo (a,h) anthracene	0.04	10	0.05	ng/g
12) Fluoranthene	0.04	10	>0.04	ng/g
13) Fluorene	0.04	10	0.06	ng/g
14) Indeno(1,2,3-c,d) pyrene	0.08	10	0.03	ng/g

TABLE 29
Target and Achieved Detection Limits for Ponce Harbor and Reference Sediments*

Analyte	Detection Limit			
	Work/QAP Target	USEPA Guidance	Achieved Approx.	Units
15) Naphthalene	0.1	10	>0.31	ng/g
16) Phenanthrene	0.05	10	>0.06	ng/g
17) Pyrene	0.04	10	>0.02	ng/g

Notes: USEPA Guidance is from "Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal". US Environmental Protection Agency, Region II. Draft. December 18, 1992 with undated attachment "Updates to the 1992 Testing Manual". Dashes (---) indicate no guidance concentration is provided.

* = All results are on a dry weight basis.

TABLE 30

Target, Guidance, and Achieved Detection Limits for Ponce Harbor Water and Elutriate Samples

Analyte	Detection Limit			
	Work/QAP Target	USEPA Guidance	Achieved (Approx.)	Units
1. Metals				
a. Arsenic	0.05	---	0.05	µg/L
b. Cadmium	0.01	0.025	0.01	µg/L
c. Chromium	0.04	1.0	0.04	µg/L
d. Copper	0.02	0.3	0.02	µg/L
e. Lead	0.01	0.3	0.01	µg/L
f. Mercury	0.002	0.002	0.0002	µg/L
g. Nickel	0.04	0.3	0.04	µg/L
h. Selenium	0.5	---	0.5	µg/L
i. Silver	0.03	0.1	0.0128	µg/L
j. Zinc	0.1	0.15	0.1	µg/L
2. Organic Compounds				
a. Pesticides				
1) Aldrin	0.4	4	2.83	ng/L
2) Chlordane	0.5	14	1.08	ng/L
3) 2, 4' –DDT	3	20	1.98	ng/L
4) 2, 4' –DDD	1	20	0.75	ng/L
5) 2, 4' –DDE	1	20	1.71	ng/L
6) 4, 4' –DDT	2.5	12	0.56	ng/L
7) 4, 4' –DDD	1	11	0.60	ng/L
8) 4, 4' –DDE	0.5	4	0.84	ng/L
9) Dieldrin	0.5	2	0.98	ng/L
10) Endosulfan I	0.5	14	1.11	ng/L
11) Endosulfan II	1	4	0.51	ng/L
12) Endosulfan Sulfate	1	10	0.56	ng/L
13) Heptachlor	1.5	3	1.17	ng/L
14) Heptachlor Epoxide	0.5	100	0.95	ng/L
15) Trans-nonachlor	0.5	14	1.01	ng/L
b. Polychlorinated Biphenyls Congeners				
1) PCB 8	0.6	0.5	16.0	ng/L
2) PCB 18	0.42	0.5	1.38	ng/L
3) PCB 28	0.50	0.5	1.73	ng/L
4) PCB 44	0.44	0.5	1.45	ng/L

TABLE 30

Target, Guidance, and Achieved Detection Limits for Ponce Harbor Water and Elutriate Samples

	Detection Limit			
Analyte	**Work/QAP Target**	**USEPA Guidance**	**Achieved (Approx.)**	**Units**
5) PCB 49	0.45	0.5	1.49	ng/L
6) PCB 52	0.43	0.5	1.44	ng/L
7) PCB 66	0.45	0.5	1.49	ng/L
8) PCB 87	0.34	0.5	1.13	ng/L
9) PCB 101	0.35	0.5	1.14	ng/L
10) PCB 105	0.18	0.5	0.58	ng/L
11) PCB 118	0.26	0.5	0.87	ng/L
12) PCB 128	0.42	0.5	1.40	ng/L
13) PCB 138	0.40	0.5	1.32	ng/L
14) PCB 153	0.32	0.5	1.06	ng/L
15) PCB 170	0.3	0.5	1.02	ng/L
16) PCB 180	0.29	0.5	0.96	ng/L
17) PCB 183	0.28	0.5	0.93	ng/L
18) PCB 184	0.28	0.5	0.92	ng/L
19) PCB 187	0.26	0.5	0.86	ng/L
20) PCB 195	0.33	0.5	1.08	ng/L
21) PCB 206	0.37	0.5	1.22	ng/L
22) PCB 209	0.38	0.5	1.26	ng/L

Notes: USEPA Guidance is from "Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal". US Environmental Protection Agency, Region II. Draft. December 18, 1992 with undated attachment "Updates to the 1992 Testing Manual". Dashes (---) indicate no guidance concentration is provided.

TABLE 31
Target and Achieved Detection Limits for Tissue Samples Associated with Ponce Harbor and Reference Station, March 2003

Analyte	Wet Weight Detection Limit			
	Work/QAP Target	USEPA Region II Guidance	Macoma Achieved (Approx.)	Nereis Achieved (Approx.)
1. Metals (units)	µg/g	µg/g	µg/g	µg/g
a). Arsenic	0.03	1.0	0.03	0.03
b) Cadmium	0.003	0.1	0.003	0.003
c) Chromium	0.03	0.2	0.03	0.03
d) Copper	0.01	1.0	0.01	0.01
e) Lead	0.004	0.1	0.004	0.004
f) Mercury	0.0003	0.02	0.0003	0.0003
g) Nickel	0.007	0.1	0.007	0.007
h) Silver	0.006	0.05	0.006	0.006
i) Selenium	0.02	---	0.02	0.02
j) Zinc	0.14	1.0	0.14	0.14
2. PAHs (units)	ng/g	ng/g	ng/g	ng/g
a) Acenaphthene	0.02	4	0.04	0.05
b) Acenaphthylene	0.04	4	0.02	0.03
c) Anthracene	0.04	4	0.07	0.08
d) Benzo (a) Anthracene	0.03	4	0.07	0.08
e) Benzo (a) Pyrene	0.05	4	0.07	0.09
f) Benzo (b) Fluoranthene	0.04	4	0.08	0.1
g) Benzo (k) Fluoranthene	0.03	4	0.05	0.06
h) Benzo (g,h,i) perylene	0.2	4	0.04	0.05
i) Biphenyl	0.03	---	0.03	0.03
j) Chrysene	0.04	4	0.07	0.08
k) Dibenzo (a,h) Anthracene	0.08	4	0.05	0.05
l) Fluoranthene	0.05	4	0.04	0.05
m) Fluorene	0.05	4	0.03	0.04
o) Indeno(1,2,3-c,d) Pyrene	0.1	4	0.05	0.06
p) Naphthalene	0.1	4	0.07 J	0.11 J
q) Phenanthrene	0.06	4	0.03	0.02 J
r) Pyrene	0.2	4	0.03	0.04
s) 1,4-Dichlorobenzene	---	0.4	0.03 J	0.07 J
3. Lipids	0.10 %	0.1 %	---	---

Notes: USEPA Guidance is from "Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal". US Environmental Protection Agency, Region II. Draft. December 18, 1992 with undated attachment "Updates to the 1992 Testing Manual". Dashes (---) indicate no concentration was provided. "J" indicates detected below the MDL.

Appendix A

Appendix A-1

Correspondence

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 2
290 BROADWAY
NEW YORK, NY 10007-1866

Edwin Muñiz
Chief, Antilles Regulatory Section
U.S. Army Corps of Engineers, Jacksonville District
400 Fernandez Juncos Avenue
San Juan, Puerto Rico 00901-3299

MAR 6 2002

Dear Mr. Muñiz,

This letter responds to your February 26, 2002 request for guidance regarding sampling and testing requirements for concurrently obtaining a baseline environmental assessment of the Ponce Harbor, Puerto Rico Ocean Dredged Material Disposal Site and a 103 evaluation of dredged materials proposed for dredging and ocean disposal as part of improvements to Ponce Harbor in preparation for the Las Americas Transshipment Port Complex.

Combining the two efforts to the maximum extent practicable would seem to make sense. However, it is important to stress that the two study areas represent extremely different oceanographic regimes and sampling would require significantly different types of equipment. The sampling and testing that will be required for the two efforts needs to be coordinated with the U.S. Army Corps of Engineers (USACE) and the Environmental Protection Agency (EPA). It is imperative that a separate quality assurance/work plan covering each effort be submitted to the Agencies for review and concurrence prior to commencement of any work. The quality assurance/work plans must cover all aspects of the field and laboratory procedures and must reflect the sampling and testing requirements of the two Agencies for the two efforts.

In order to initiate the process for the Ponce Harbor portion of the work, the project proponent should begin the formal Department of the Army permit process. EPA will coordinate with USACE on the sampling and testing requirements following receipt of a request for such requirements from your office. This request should be accompanied by a clear description of the proposed work, a recent (no older than one year) bathymetric survey (engineering scale, with areas to be dredged clearly delineated), 8.5" x 11" site maps showing locations of outfalls, creeks or other pertinent features, and as much background information regarding the site as possible. Types of relevant background information are listed in Section 4.1 of the joint USACE/EPA ocean and inland dredged material testing guidance manuals.

We look forward to further coordinating with your office on this important project. If you have any questions please do not hesitate to contact Mark Reiss at 212-637-3799.

Sincerely,

Douglas Pabst, Team Leader
Dredged Material Management Team
Division of Environmental Planning and Protection

Internet Address (URL) • http://www.epa.gov
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TOTAL P.02

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DEPARTMENT OF THE ARMY
JACKSONVILLE DISTRICT CORPS OF ENGINEERS, ANTILLES OFFICE
400 FERNANDEZ JUNCOS AVENUE
SAN JUAN, PUERTO RICO 00904-3299

REPLY TO
ATTENTION OF

Antilles Regulatory Section

March 11, 2002

F-up
Antilles
3/18

RECIBIDO
AFI
02 MAR 13 PM 4:16

Mr. Ramón Amador
Executive Director
Autoridad para el Finaciamento de la
Infraestructura (AFI)
Edificio Capital Center 235
Ave. Arterial Hostos Suite 1601
San Juan, Puerto Rico 00919-1433

Dear Mr. Amador:

This is in reference to your letter dated February 22, 2002, in which AFI notified the Corps that they hired PPB Laboratories to conduct the characterization of the sediments that would be dredged and disposed at the ODMDS located south of Ponce. AFI has also indicated their desire to also conduct the sampling necessary at the disposal site to expedite the preparation of the Site Management Plan for the Ponce ODMDS.

On February 26, 2002, we communicated your position to the Environmental Protection Agency (EPA). On March 6, 2002, the EPA indicated that combining the two efforts to the maximum extent practicable would seem to make sense. However, they indicated that the two areas are different and that this represents different site conditions, sampling requirements and equipment. In their response, which is enclosed with this letter, they provided requirements that must be met. Please be advised that contrary to what the EPA letter say about the need of a permit application, at this time it is not necessary. However, shortly we will provide additional guidance on the submittal of a permit application.

As soon as you provide us with the information requested by the EPA, we will initiate the process with the EPA for both sites.

If you have any questions please feel free to contact the undersigned.

Sincerely,

Edwin E. Muñiz
Chief, Antilles Regulatory Section

Enclosure

020 313037

CSA Group

CKE 9/9/02

facsimile transmittal

FAXED

To:	Mr. Ward Dickens	Fax:	1-352-395-6639
From:	Mr. Angel García	Date:	9/9/02
Re:	Port of the Americas	Pages:	10
CC:			

☐ Urgent x For Review ☐ Please Comment ☐ Please Reply ☐ Please Recycle

Enclosed please find copy of letter dated September 6, 2002 of the US Army Corps of Engineers addressed to Mr. Ramón A. Amador.

DEPARTMENT OF THE ARMY
JACKSONVILLE DISTRICT CORPS OF ENGINEERS, ANTILLES OFFICE
400 FERNANDEZ JUNCOS AVENUE
SAN JUAN, PUERTO RICO 00901-3299

REPLY TO
ATTENTION OF

Antilles Regulatory Section

SEP 6 2002

Mr. Ramón Amador
Executive Director
Autoridad para el Finaciamento de la
Infraestructura (AFI)
Edificio Capital Center 235
Ave. Arterial Hostos Suite 1601
San Juan, Puerto Rico 00919-1433

Dear Mr. Amador:

This is in reference to the report titled "Ponce Harbor Tier I Report for Section 103 Ocean Disposal Evaluation" and the Work Plans and Quality Assurance Project Plans (QAPP) for the Ponce Harbor and the Ponce Ocean Dredged Material Disposal Site (ODMDS), prepared by the Puerto Rico Infrastructure Financing Authority (AFI, acronym in Spanish), as part of the proposed Port of the Americas Complex in Puerto Rico.

Enclosed with this letter you will find copy of the response submitted by the Environmental Protection Agency (EPA), dated August 22, 2002. The EPA is providing the sampling and testing requirements for the characterization of the sediments proposed for dredging at the Ponce site, as part of the proposed Port the Americas project. Please be advised that the EPA has made changes to that proposed in the referenced documents. All future work associated with the sampling and testing of sediments must be done to conformance with EPA's approved work plan enclosed with their letter. If you have any questions please feel free to contact this office at 787-729-6905 or directly with Mr. Mark Reiss from the EPA at 212-637-3799.

Sincerely,

Edwin E. Muñiz
Chief, Antilles Regulatory Section

Enclosures

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 2
290 BROADWAY
NEW YORK, NY 10007-1866

AUG 22 2002

Edwin Muñiz
Chief, Antilles Regulatory Section
U.S. Army Corps of Engineers, Jacksonville District
400 Fernandez Juncos Avenue
San Juan, PR 00901-3299

RE: Ocean Disposal Suitability Sampling and Testing Requirements for Sediments Proposed for Dredging from Ponce Harbor Improvements Associated with the Las Americas Trans-Shipment Port Proposal

Dear Mr. Muñiz,

Please find attached the sampling and testing requirements that EPA Region 2 will require for characterization of sediments proposed for dredging from Ponce Harbor Improvements as part of the Las Americas Trans-Shipment Port Project.

The sampling and testing outlined in the enclosed documentation departs significantly from that proposed by the applicant in their work plan. Their proposal was not directly related to the sediments actually proposed for dredging and therefore could not be used for regulatory determinations. All work associated with this sampling and testing must conform to an EPA-approved work plan.

If you (or the applicant) have any questions regarding the outlined requirements, please do not hesitate to contact Mark Reiss of my team. He can be reached at (212)-637-3799.

Sincerely,

Douglas Pabst, Team Leader
U.S. EPA Region 2, Dredged Material Management Team

REV. 4/98

SAMPLING SCHEME and LIST of REQUIRED TESTING as OUTLINED in the 1992 EPA REGION II REGIONAL IMPLEMENTATION MANUAL on Dredged Material Proposed for Ocean Disposal and the 1991 GREEN BOOK

AUG 22 2002

Applicant CITY OF PONCE – LAS AMERICAS TRANSSHIPMENT PORT

Applicant No. _____

Address Ponce, Puerto Rico

Waterway Ponce Harbor and Approach Channel

Proposed Volume: 1.4 Million cubic yards (based on 2001 bathymetric surveys)

Project Depth in feet: -55 MLW

(NOTE: The applicant is required to collect cores from each sampling location to project depth plus 2 ft)

A preapplication meeting X was held on _____ at EPA Regional Office in New York

___ was not held at the EPA Regional Office in New York.

The applicant has indicated that testing will be performed by the following laboratories:

Biological: Harding ESE Analytical: HBEL/Lew/PPB/Microbac

The sampling scheme and required testing described on the reverse side was approved for the proposed project area based upon the information contained in the attached memo.

Sample dredging sites and are indicated on attached map.

The proposed dredging area is subdivided into TWO reaches for sampling and testing purposes.

The applicant is required to collect cores from each sampling location to project depth plus 2 ft.

BROOKLYN NAVY YARD: Reach 1: ≤9 locations (Sandy material from Tops of Cores) Reach 2: ≤9 locations (Silty-clay material from core bottoms)

- The District/Region reserve the right to require additional sampling and testing at any time.

COMMENTS:

1. If there is any evidence of stratification in core samples, contact NYD prior to compositing.

2. Additional cores may have to be taken from each station in order to ensure adequate volumes of sediment to meet testing requirements. If so, the same number of cores must be taken and composited from each location.

3. Core locations may have to be moved if sampling difficulties are encountered. If so, contact Region 2 during sampling to confirm new locations.

ADDITIONAL LOCAL REQUIREMENTS:

Contact Mr. Edwin Muniz of the USACE Antilles Regulatory Section for any applicable requirements

* If you have any questions regarding sampling, test protocols, test species, qa/qc, etc. please contact the Dredged Material Management Team of the EPA Region 2 at the extension of person completing this form.

PREPARED BY: MARK REISS (MR) DATE: 22 AUGUST 2002 PHONE: 212-637-3799

SEE REVERSE SIDE FOR SAMPLING AND TESTING REQUIREMENTS

AUG 22 2002

APPLICANT: PONCE HARBOR-LAS AMERICAS TRANSSHIPMENT PORT

Any Box Checked Off Indicates an Analysis or Assay that is Required for a Given Project.
Archiving requires saving a sample for possible analysis at a later time pending further instruction.

X=Per Homogenized Sediment Core + Reference (Composited Grabs) + Control
C=Per Bioassay Sediment Composite
A=Archive
W=Site Water and Elutriate
T=Per Tissue Replicate (Ref, Test, Pre-test, A=Archive Ctl and any remaining tissue from Ref, Test, Pre-test)

1. SEDIMENT PHYSICAL ANALYSIS (If stratification is observed, each stratum within a core must be analyzed separately)
   a. X **Grain Size Analysis (% sand, % silt, & % clay)**
   b. X **% Moisture**
   c. C **Specific Gravity**
   d. C **Bulk Density**
   e. C **Plastic and Liquid limits (Atterberg limits)**

Me
~~126~~

2. SEDIMENT CHEMICAL ANALYSIS

REQUIRED

   a. X **% Total Organic Carbon**

CASE BY CASE BASIS

   a. C A **Metals (Ag, As, Cd, Cr, Cu, Hg, Ni, Pb, Zn)**
   b. C A **PAHs (LMWs: acenapthene, acenaphthylene, anthracene, fluorene, naphthalene, phenanthrene)**
   c. C A **PAHs (HMWs: benzo(a)anthracene, benzo(a)pyrene, benzo(g,h,i)perylene, benzo(b)fluoranthene, benzo(k)fluoranthene, chrysene, dibenzo(a,h)anthracene, fluoranthrene, indeno(1,2,3-c,d)pyrene, pyrene)**
   d. C A **Semi-volatiles (1,4 dichlorobenzene)**
   e. C A **Pesticides (aldrin, alpha chlordane, trans nonachlor, dieldrin, p,p' and o,p' DDT/DDD/DDE, endosulfans(I,II, and sulfate), heptaclor, heptachlor epoxide)**
   f. C A **PCBs (#8,18,28,44,49,52,66,87,101,105,118,128,138,153,170,180,183,184,187,195,206,209)**
   g. ___ **PCB coplanar (#77,126,156,169)**
   h. ___ **Dioxins/Furans (2,3,7,8 - substituted isomers, n=17)**
   i. ___ **Other:**

These
49 ~~105~~ 87
77/110 M 126 169
183
184

3. CHEMICAL ANALYSIS OF SITE WATER AND ELUTRIATE

REQUIRED

   a. W **Metals (Ag, Cd, Cr, Cu, Hg, Ni, Pb, Zn)**
   b. W **PCBs (#8,18,28,44,49,52,66,87,101,105,118,128,138,153,170,180,183,184,187,195,206,209)**
   c. W **Pesticides (aldrin, alpha chlordane, trans nonachlor, dieldrin, p,p' and o,p' DDT/DDD/DDE, endosulfans(I,II, and sulfate), heptaclor, heptachlor epoxide)**
   d. ___ **Other:**

CASE BY CASE BASIS

   a. ___ **PAHs (_ all 16, _ LMW, _ HMW, _ as specified)**
   b. ___ **2,3,7,8-TCDD**
   c. ___ **2,3,7,8-TCDF**
   d. ___ **PCB coplanar (#77,126,156,169)**
   e. ___ **Other:**

Add Lipids

4. BIOASSAYS (species listed in guidance manual)
   a. C **Water Column Acute Tox. (bivalve larvae, M. bahia, Menidia sp.)**
   b. C **10-Day Benthic Acute Tox. (A. abdita, R. abronius, E. estuarius, or L. plumulosus)**
   c. C **10-Day Benthic Acute Tox. (M. bahia)**
   d. C **28-Day Bioaccumulation (N. virens, and Macoma nasuta or M. nasuta)**

5. 28-DAY WHOLE-SEDIMENT BIOACCUMULATION TISSUE ANALYSIS

REQUIRED

   a. T A **Metals (Ag, As, Cd, Cr, Cu, Hg, Ni, Pb, Zn)**
   b. T A **Pesticides (aldrin, alpha chlordane, trans nonachlor, dieldrin, p,p' and o,p' DDT/DDD/DDE, endosulfans(I,II, and sulfate), heptachlor, heptachlor epoxide)**
   c. T A **PCBs (#8,18,28,44,49,52,66,87,101,105,118,128,138,153,170,180,183,184,187,195,206,209**
   d. T A **Semi-volatiles (1,4 dichlorobenzene)**
   e. T A **PAHs (LMWs: acenapthene, acenaphthylene, anthracene, fluorene, naphthalene, phenanthrene)**
   f. T A **PAHs (HMWs: benzo(a)anthracene, benzo(a)pyrene, benzo(g,h,i)perylene, benzo(b)fluoranthene, benzo(k)fluoranthene, chrysene, dibenzo(a,h)anthracene, fluoranthrene, indeno(1,2,3-c,d)pyrene, pyrene)**

CASE BY CASE BASIS

   a. ___ **Dioxins/Furans (2,3,7,8 - substituted isomers, n=17)**
   b. ___ **PCB coplanar (#77,126,156,169)**
   c. ___ **Other:**

REFER TO COMMENT SECTION ON REVERSE SIDE FOR ADDITIONAL INFORMATION

AUG 22 2002

Sampling and Testing Requirements for Proposed Improvement of Ponce Harbor for the Las Americas Transhipment Port Project

Sampling and Compositing

Areas of Ponce Harbor that are proposed for dredging with disposal at the Ponce Harbor Ocean Dredged Material Disposal Site have been divided into two sub-areas, or reaches. The entire project is to be sampled by vibracore at nine (9) locations. The Lambert coordinates of sample locations are reported in Attachment 1. For planning and estimating purposes, an anticipated core length is also reported for each location. This length is simply the difference between the existing depths (at the time of the latest survey) and the project depth (including two (2) feet of allowable overdepth).

If the volume of composite sample obtained by a single deployment at each location in a given reach yields insufficient sediment to conduct all sampling and testing (as described below), then multiple deployments must be taken at each location to provide the necessary volume for each reach. Care must be taken to ensure that **an equal number of cores is taken from each location contributing to a given composite.**

Reach A: Overlying Sandy Material: Composite of up to nine (9) vibracore samples. All samples must be taken to the depth at which underlying silty-clay material is encountered (approximately -40 to -45 MLW. Sampling is representative of all overlying sandy materials in the project area. It is possible that sands may not be present at Station 9, see Attachment 1.

Reach B: Underlying Silty-Clay Material: Composite of up to nine (9) vibracore samples. All samples must be taken from the interface with overlying sandy materials to -57 feet MLW (Project depth + 2 feet). Sampling is representative of all underlying silty-clay material in the project area.

It is possible that one or the other strata (particularly overlying sands) may not be represented at individual sampling locations selected for this project. Records of visible strata must be kept in a description of the core retrieved. The overall strategy is to composite sandy materials and silty-clay materials as separate units for testing.

Sediment Testing

PLEASE REFER TO REGION 2 IMPLEMENTATION MANUAL FOR SPECIFIC METHODS AND DETECTION LIMITS AND QA/TEST ACCEPTABILITY CRITERIA

A. Core Samples/Composites/Reference/Control:
The following parameters must be measured in the upper and lower portions of each core sample, in each composite sample and in the reference and control samples:

- -Grain Size (%Sand, %Silt, %Clay)
- -Percent Moisture and Moisture Content
- -Total Organic Carbon

AUG 22 2002

-Sufficient sediment to conduct bulk sediment chemical analyses should be archived for each core location).

There should therefore be up to 18 core samples (or fractions), 2 reach composites, 1 reference, and 1 (or more) control datasets for the above parameters.

B. Reach Composites/Reference Sediment:
In addition to the above parameters, the following must be reported for each reach composite (and for the reference sediment where indicated).

-Atterberg limits
-Bulk Density (wet and dry)
-Specific Gravity

-Bulk sediment chemistry (Reference sediment also) for metals, PCBs, PAHs, pesticides. In addition to standard laboratory QA (blanks, surrogates, internal standards), QA for these parameters must include (per analytical batch of one to twenty (1-20) samples):

-One SRM
-One triplicate analysis
-One set of four MDL verification samples
-One matrix spike

-Suspended particulate phase and 10-d acute solid phase toxicity tests. *Mysidopsis bahia* and *Ampelisca abdita* are the preferred organisms for use in the solid phase tests,

-28-d bioaccumulation tests. *Nereis virens* and *Macoma nasuta* are preferred organisms, if these are unavailable contact USEPA-Region 2.

C. Site-Water/Elutriate Analyses:

-Analyze for the same analyte list as sediments (except for PAHs which will not be required). Apply the same QA standards, (triplicate analysis should be performed on the elutriate, not the site-water).

D. Bioaccumulation Tissue Analyses:

-Three samples of each of the pretest organisms should be retained and analyzed in triplicate for all parameters listed for sediment. This can be performed along with the remainder of the tissue samples.

-Lipid content should be determined in triplicate for each organism using Bligh and Dyer (or comparable) method

-All compounds listed for sediment must be analyzed for in tissues. However, if the concentration of a specific contaminant is below the detection limit in the sediment contact the USEPA-Region 2 before proceeding in tissue.

* It is recommended that any extra sediment/tissue from reach composites be archived*

AUG 22 2002

ATTACHMENT 1

Sampling and Testing Reaches - Ponce Harbor 2002

Reach A: Overlying Sands, Reach B: Underlying Silty Clays

Sample	x	y	Exist. Depth	Proj. Depth (+2)	Estimated Stratif. Depth	Anticipated Core Length (A)	Anticipated Core Length (B)
1	133000	15500	35.4	57	40	4.6	17
2	132750	15625	39.9	57	40	0.1	17
3	132250	15500	40.4	57	47	6.6	10
4	132500	15250	39.2	57	40	0.8	17
5	131875	15250	40.5	57	47	6.5	10
6	132250	15000	39	57	45	6	12
7	131750	15000	39.3	57	45	5.7	12
8	132000	14750	40.7	57	45	4.3	12
9	132000	14500	47.7	57	45	0	12
		15375					
					Total (ft)	34.6	119

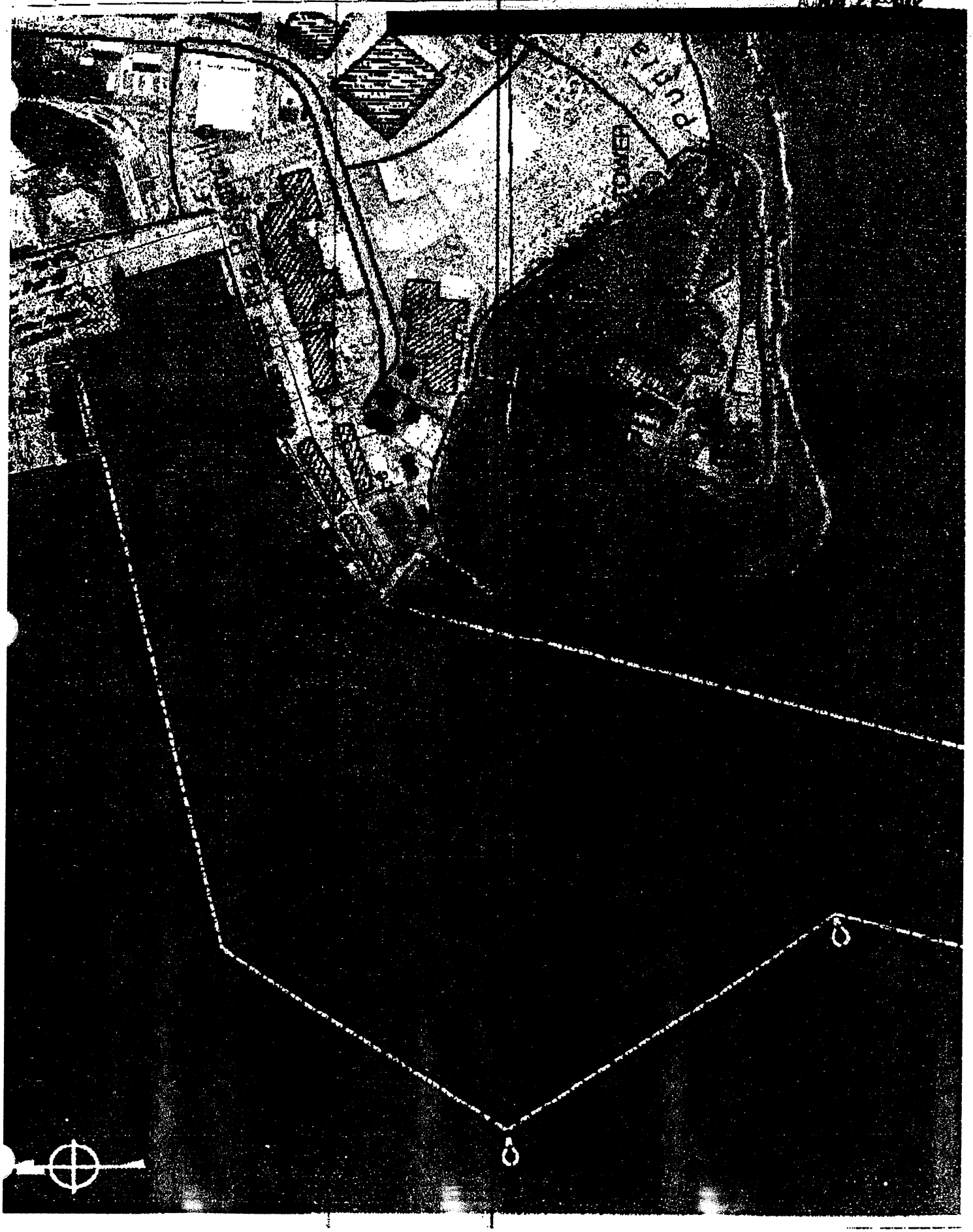
Visual descriptions of sediment strata underlying areas proposed for dredging in Ponce Harbor

Feet (MLW)	Borings 1	2	6	7
15			Fine sand	Fine sand
20			Fine sand	Fine sand
25			Fine sand	Fine sand
30			Fine sand	Fine sand
35			Fine sand	Fine sand
40	Silty clay	Fine sand	Fine sand	Fine sand
45	Silty clay	Silty clay	Silty clay	Fine sand
50	Silty clay	Silty clay	Silty clay	Fine sand
55	Silty clay	Silty clay	Silty clay	Silty clay
60	Silty clay	Silty clay	Silty clay	Silty clay

Mark

561/747-2954

Reference: ERTEC. 2001. Status Report 1 & 2 Las Americas Transshipment Port. Ponce, Puerto Rico. Prepared for Puerto Rico Infrastructure Financing Authority, Hato Rey PR. Dated April 27, 2001.


POND

*** TRANSMISSION REPORT ***

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JUN-18-2002 TUE 10:06 AM ANTILLES REGULATORY 787 729 6906 P. 01

DEPARTMENT OF THE ARMY
JACKSONVILLE DISTRICT CORPS OF ENGINEERS, ANTILLES OFFICE
400 FERNANDEZ JUNCOS AVENUE
SAN JUAN, PUERTO RICO 00901-3299

REPLY TO ATTENTION OF

Antilles Regulatory Section

June 13, 2002

Post-it® Fax Note 7671	Date June 02	# of pages ▸ 5
To Ward Dickens	From Fred Quiñones	
Co./Dept. PPB Labs	Co. CSA Group	
Phone # 352-895 0352	Phone # 787 409-2861	
Fax # 352 895-6639	Fax # 787-763-4445	

· Ward: As discussed. Please call. FQ

Mr. Ramón Amador
Executive Director
Autoridad para el Finaciamento de la Infraestructura (AFI)
Edificio Capital Center 235
Ave. Arterial Hostos Suite 1601
San Juan, Puerto Rico 00919-1433

Dear Mr. Amador:

This is in reference to the report titled "Ponce Harbor Tier I Report for Section 103 Ocean Disposal Evaluation" and the Work Plans and Quality Assurance Project Plans (QAPP) for the Ponce Harbor and the Ponce Ocean Dredged Material Disposal Site (ODMDS), prepared by the Puerto Rico Infrastructure Financing Authority (AFI, acronym in Spanish), as part of the proposed Port of the Americas Complex in Puerto Rico.

Enclosed with this letter you will find copy of the response submitted by the Environmental Protection Agency (EPA) dated Jun 5, 2002, to the above referenced documents. The EPA provided comments to the work plans. The issues raised by EPA must be satisfactorily addressed before work can begin.

As soon as you provide us with the information requested by the EPA, we will continue with the process with the EPA.

If you have any questions please feel free to contact the undersigned.

DEPARTMENT OF THE ARMY
JACKSONVILLE DISTRICT CORPS OF ENGINEERS, ANTILLES OFFICE
400 FERNANDEZ JUNCOS AVENUE
SAN JUAN, PUERTO RICO 00901-3299

REPLY TO
ATTENTION OF

Antilles Regulatory Section

June 13, 2002

Mr. Ramón Amador
Executive Director
Autoridad para el Finaciamento de la
Infraestructura (AFI)
Edificio Capital Center 235
Ave. Arterial Hostos Suite 1601
San Juan, Puerto Rico 00919-1433

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This is in reference to the report titled "Ponce Harbor Tier I Report for Section 103 Ocean Disposal Evaluation" and the Work Plans and Quality Assurance Project Plans (QAPP) for the Ponce Harbor and the Ponce Ocean Dredged Material Disposal Site (ODMDS), prepared by the Puerto Rico Infrastructure Financing Authority (AFI, acronym in Spanish), as part of the proposed Port of the Americas Complex in Puerto Rico.

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As soon as you provide us with the information requested by the EPA, we will continue with the process with the EPA.

If you have any questions please feel free to contact the undersigned.

Sincerely,

Edwin E. Muñiz
Chief, Antilles Regulatory Section

Enclosure

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 2
290 BROADWAY
NEW YORK, NY 10007-1866

Edwin Muñiz
Chief, Antilles Regulatory Section
U.S. Army Corps of Engineers, Jacksonville District
400 Fernandez Juncos Avenue
San Juan, Puerto Rico 00901-3299

JUN 5 2002

Dear Mr. Muñiz,

We have reviewed the materials forwarded by your office on May 16, 2002. These materials include: a Tier 1 evaluation report of the suitability of dredged materials that are proposed for dredging from areas of Ponce Harbor for disposal at the Ponce Harbor Ocean Dredged Material Disposal Site (Ponce ODMDS); a work and quality assurance plan for sampling and testing of Ponce Harbor dredged material, and a work and quality assurance plan for sampling and testing of surficial sediments as part of a baseline assessment of conditions at the Ponce ODMDS. This letter serves to inform you of our determinations and actions regarding these submissions.

We concur with the general conclusion of the Tier 1 evaluation report regarding the need for further testing. Specifically, we agree that sufficient information has not been presented to conclude that dredged material from Ponce Harbor meets the exclusionary criteria listed in 40 CFR 227.13(b). Therefore, testing of the material is required to establish whether or not it can be disposed of in the ocean. We however disagree with the specific conclusion that there is no basis for expecting mercury to be present (see Table 4-1 of the Inland Testing Manual). This contaminant must therefore be retained in the list of analytes for this project.

Attached are the Dredged Material Management Team's comments to the work plans. There appear to be several significant issues that need to be addressed before work can begin. A notable concern however is that the applicant has failed to provide the information that we requested in our March 6th letter. Without this information, EPA cannot prepare an appropriate sampling and testing strategy for the Ponce Harbor dredging project.

The work and quality assurance plans for the dredged material evaluation and baseline assessments have been sent to our Division of Environmental Science and Assessment for review. Please inform the project applicant (AFI) that work cannot commence until they have completed their review and all issues have been addressed to EPA's satisfaction.

If you have any questions please contact Mr. Mark Reiss at 212-637-3799.

Sincerely,

Douglas A. Pabst, Team Leader
EPA Region 2, Dredged Material Management Team

Comments on the Work Plan and Quality Assurance Project Plan for Ponce ODMDS
The quality assurance plan for the baseline assessment has been sent to our Division of Environmental Science and Assessment for review. Work cannot commence until they have completed their review and all issues have been addressed to their satisfaction.

With respect to the work and sampling plan, we have several comments. Comments are provided below and are preceded by the name of the section(s) of the plan to which they refer.

Pollution Sources/Evaluation Needs - Information provided in the section entitled "Pollution Sources" and the first two paragraphs of the section entitled "Evaluation Needs" is irrelevant. These sections are apparently included because of cut and paste errors. These paragraphs should be deleted from the final work plan.

Potential Contaminants of Concern - The list of analytes is satisfactory. However the plan indicates that PCBs will be analyzed as Aroclors, rather than individual congeners. The individual congeners analyzed by NOAA in their Status and Trends program must be analyzed.

Project Coordination and Quality Assurance - A complete review of project coordination and quality assurance will be provided by our Division of Environmental Science and Assessment. Therefore, we will offer no comments on this section, at this time.

Sample Station Selection - ODMDS Stations - Figure 1 was not included with the report. However, based on the text description and Figure 5-1 of the QAPP it is clear that the sampling design that was used to assess baseline conditions at the Yabucoa ODMDS is being contemplated for this site as well. EPA concurs with this site selection strategy. Figure 1 should be provided in the final work plan.

Sample Station Selection - Reference Stations - Triplicate samples for reference sediment should be from three separate grabs of sediment rather than triplicates from within a single box core sample. This was an oversight during the Yabucoa study.

Quality Control Samples/Sampling Methods and Procedures - Box corer sampling is not necessary, surficial grab samplers will be sufficient. A complete review of project coordination, procedures, and quality assurance will be provided by our Division of Environmental Science and Assessment. Therefore, we will offer no comments on this section, at this time.

Sediment Physical and Chemical Analyses - The list of analytes is satisfactory. However the plan indicates that PCBs will be analyzed as Aroclors, rather than individual congeners. The individual congeners analyzed by NOAA in their Status and Trends program must be analyzed. Also the specific methods proposed differ from those used in Region 2 testing programs, the Division of Environmental Science and Assessment will review the acceptability of these methods.

Region 2 will use the laboratory performance tolerances stated in the EPA-Region 2/USACE-New York District Regional Testing Manual to determine whether data are acceptable. **EPA Region 2 strongly recommends that sufficient sediment be archived to allow for re-analysis of any/all compounds.**

Comments on the Work Plan and Quality Assurance Project Plan for Proposed Dredging in Ponce Harbor

The quality assurance plan for the 103 assessment of Ponce Harbor sediments has been sent to our Division of Environmental Science and Assessment for review. Work cannot commence until they have completed their review and all issues have been addressed to their satisfaction.

With respect to the work and sampling plan, we have several comments. Comments are provided below and are preceded by the name of the section(s) of the plan to which they refer.

We concur with conclusions of the Tier 1 evaluation report. Specifically, we agree that sufficient information has not been presented to conclude that dredged material from Ponce Harbor meets the exclusionary criteria listed in 40 CFR 227.13(b). Therefore, testing of the material is required to establish whether or not it can be disposed of in the ocean. However, we disagree with the specific conclusion that there is no basis for expecting mercury to be present (see Table 4-1 of the Inland Testing Manual). Mercury must therefore be retained in the list of analytes for this project.

Introduction - To which areas of Ponce Harbor does "flood control" refer?

Tier II Evaluation - As noted above, we disagree with the omission of mercury from the analyte list. Mercury must be analyzed. In addition, the plan indicates that PCBs will be analyzed as Aroclors, rather than individual congeners. The individual congeners analyzed by NOAA in their Status and Trends program must be analyzed.

Project Coordination and Quality Assurance - A complete review of project coordination and quality assurance will be provided by our Division of Environmental Science and Assessment. Therefore, we will offer no comments on this section, at this time.

Sample Station Selection - Harbor Stations

As indicated in our March 6, 2002, EPA will determine an appropriate sampling and testing strategy for appropriately characterizing Ponce Harbor sediments based on a review of the information detailed in that letter. The requested information has not been provided and therefore EPA is not in a position to accept the level of sampling and testing proposed by the applicant, nor can EPA accept the locations and compositing strategies proposed in Figure 5-1 (Figure 1 was omitted from the Work Plan). The vague statements provided in the report regarding considering point and non-point sources of contaminants and sediment type in designing sampling strategies are not supported by any actual data or maps. The applicant should submit the information requested in our March 6, 2002 letter along with their request for a sampling and testing plan.

While detailed comments on sampling and sample handling methods will be provided by our Division of Environmental Science and Assessment, we note that 1 gallon containers will be very small for the amount of sediment that is likely to be sampled.

Quality Control Samples - A complete review of project coordination, procedures, and quality assurance will be provided by our Division of Environmental Science and Assessment. Therefore, we will offer no comments on this section, at this time.

Sampling Methods and Procedures - A complete review of project coordination, procedures, and quality assurance will be provided by our Division of Environmental Science and Assessment. Therefore, we will offer no comments on these aspects, at this time. However, there are various statements in the proposed work with which EPA takes exception. EPA disagrees with the limitation of sampling with a Vibracore to depths below 35 feet and with minimum core lengths of 4 feet. All harbor samples are to be collected *to project depth* using a Vibracore or other coring unit unless EPA concurs in writing with an alternate approach. Reference sediments can be collected with a grab sampler, such as van Veen.

Sediment Physical and Chemical Analyses - With the exception of excluding mercury from the analyte list, he list of analytes is satisfactory. The plan indicates that PCBs will be analyzed as Aroclors, rather than individual congeners. This is not acceptable to EPA. The individual congeners analyzed by NOAA in their Status and Trends program must be analyzed. Also the specific methods proposed differ from those used in Region 2 testing programs, the Division of Environmental Science and Assessment will review the acceptability of these methods.

Site Water/Elutriate Analyses - Contrary to the assertion in the report, site water from Ponce Harbor must be used to prepare the 100% elutriate, natural dilution water can be obtained from the Ponce ODMDS site or elsewhere or artificial seawater may be used.

Sediment Bioassays/Elutriate Bioassays/Bioaccumulation - Tests should not be performed with the field duplicate sample.

Chemical Analyses of Tissues - Tissues of both *Nereis* and *Macoma* must be analyzed for all contaminants detected in the sediment (including PAHs). Comments provided above regarding mercury, PCB congeners and analytical methods are applicable here as well.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 2
290 BROADWAY
NEW YORK, NY 10007-1866

MAR 31 2003

Bryan F. Cotter
PPB Environmental Laboratories, Inc.
6821 SW Archer Road
Gainesville, FL 32608

Dear Mr. Cotter,

EPA Region 2 has reviewed your submission requesting a change in the scope of work for the characterization of sediments that are proposed for dredging from areas of Ponce Harbor with disposal at the Ponce Harbor PR Ocean Dredged Material Disposal Site and approves of the proposed changes. Specifically, in light of the lack of vertical stratification in project sediments, EPA approves of testing of the entire project as a single composite sample and the collection of all reference sediment from a single location (i.e., Reference Station A).

If you have any further questions, please contact me. My telephone number is 212-637-3799 and my email address is reiss.mark@epa.gov.

Thank you,

Mark Reiss
Division of Environmental Planning and Protection

Cc: Edwin Muñiz, USACE-Antilles Regulatory Section

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 2
290 BROADWAY
NEW YORK, NY 10007-1866

JUN 10 2003

Edwin Muñiz
Chief, Antilles Regulatory Section
U.S. Army Corps of Engineers, Jacksonville District
400 Fernandez Juncos Avenue
San Juan, Puerto Rico 00901-3299

Dear Mr. Muñiz:

This letter provides additional guidance for conducting testing in support of the Section 103 assessment of suitability for placement at the Ponce Harbor Ocean Dredged Material Disposal Site (ODMDS) of sediments that are proposed for dredging from areas in Ponce Harbor as part of the Port of Las Americas project. We have received and reviewed the sediment chemistry results for the above referenced project and for the assessment of baseline conditions at the ODMDS. Sediments at both the proposed dredging areas and the ODMDS were found to be typical of near coastal sediments having little anthropogenic contamination.

Due to their confirmed presence in the proposed dredged material, EPA Region 2 has determined that bioaccumulation test organism tissues must be analyzed for PAHs and metals concentrations. Presence of chlorinated organics in the proposed dredged material was not confirmed by the testing results and therefore will not be required analytes in tissue samples. Please instruct the applicant that this testing will be necessary. Following submission of these data, EPA Region 2 and USACE can make a determination regarding the material's suitability for ocean disposal at the Ponce ODMDS.

In addition, EPA Region 2 has prepared a draft of the Site Monitoring and Management Plan for the Ponce ODMDS incorporating the results of the baseline sediment assessment. We anticipate that the draft will be provided to USACE for review in the coming weeks. Following USACE review, it will have to be provided to the public and other interested parties for their review. We look forward to coordinating this effort with you

If you have any questions regarding this correspondence, please do not hesitate to contact Mr. Mark Reiss of my staff at (212) 637-3799. Thank you.

Sincerely,

Ronald Borsellino
Deputy Director
Division of Environmental Planning and Protection

Appendix A-2

Ponce Harbor Tier I Report for Section 103 Ocean Disposal Evaluation

Ponce Harbor Tier I Report
for
Section 103 Ocean Disposal Evaluation

SUBMITTED TO:

Infrastructure Financing Authority
235 Arterial Hostos Avenue
Suite 1601
San Juan, Puerto Rico 00918-1454

SUBMITTED BY:

PPB Environmental Laboratories, Inc.
6821 S.W. Archer Road
Gainesville, Florida 32608
Phone: 352/377-2349

and

Water & Air Research, Inc.
6821 S.W. Archer Road
Gainesville, Florida 32608
Phone: 352/372-1500

Purchase Order Number: C-151
March 2002

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Introduction

This report presents results of a Tier I evaluation of dredged material proposed for disposal at the Ponce, Puerto Rico, Ocean Dredged Material Disposal Site (ODMDS). This evaluation is prescribed in Section 103 of Public Law 92-532, the Marine Protection, Research, and Sanctuaries Act of 1972. According to Section 103, dredged material must be evaluated using criteria promulgated by the U.S. Environmental Protection Agency (EPA) at Title 40 of the *Code of Federal Regulations (CFR)*, Parts 220-228.

To help implement their regulations, EPA published *Evaluation of Dredged Material Proposed for Ocean Disposal* (EPA-503/8-91/001) dated February 1991. The EPA manual prescribes a tiered procedure for evaluating sediments. This report presents details of the Tier I assessment for proposed dredging in Ponce Harbor. The purpose of the Tier I assessment is to determine the need for further evaluation (e.g., Tier II) and, if further evaluation is needed, to prescribe testing parameters.

Ponce is a center of commerce in south Puerto Rico. A harbor and port has existed there since approximately 1911. The current port is operated by the City of Ponce Port Authority. The port provides access to the Caribbean Sea and the ODMDS lies offshore.

Description of Action

The current ocean disposal evaluation of Ponce Harbor will consider potential dredge material from flood control and deepening of the channel work areas to a project depth of 45 feet with 4 feet of possible overdredging. Approximately two million cubic yards of material will be dredged from the harbor. The project plans to use the Ponce ODMDS for disposal of dredged material. This sampling plan and protocol defines that work.

Pollution Sources

Ponce is the second largest city in Puerto Rico. The watershed in the vicinity of Ponce Harbor is an urbanized area with a long history. Several waterways drain the Ponce area and discharge near the harbor. Ponce is a modern city with single- and multiple-family housing, commercial and industrial developments, recreational facilities, and government buildings.

In addition, the waterfront in the vicinity of the port has been used for more than fifty years for commercial and industrial activities. The community of Playa de Ponce is along the north waterfront area. It includes urban commercial and residential activities and was the site of the original port.

Description of Disposal Site

The proposed disposal site is the Ponce ODMDS. This site is located approximately six nautical miles offshore from the Island of Puerto Rico near Ponce Harbor and is designated for the disposal of dredged material. The site is one nautical mile square with

center coordinates of 17°53' 30"N, 66°37' 11"W and is in water that averages 1,400 feet (425 meters) deep. The corners of the ODMDS are located at:

17°54' 00"N, 66°37' 43"W
17°54' 00"N, 66°36' 41"W
17°53' 00"N, 66°36' 41"W
17°53' 00"N, 66°37' 43"W

Section 506 of the Water Resources and Development Act (WRDA) of 1992, which amended the Marine Protection, Research and Sanctuaries Act of 1972 (MPRSA), requires the U.S. Environmental Protection Agency (EPA) and the U.S. Army Corps of Engineers (USACE) to prepare a Site Management and Monitoring Plan (SMMP) for each ocean dredged material disposal site. A site management and monitoring plan will be developed, coordinated, and approved for this site prior to any future dredge material disposal activity. This plan will identify a number of actions, provisions and practices to manage operational aspects of dredging and disposal activities and to perform site monitoring.

Description of Dredged Material

In September 1974, U.S. Army Corps of Engineers prepared a Final Environmental Impact Statement addressing maintenance dredging in Ponce Harbor. The report text indicates that samples of dredge material were tested. Particle size analysis showed an average of approximately 94 percent of the sediment passed a No. 200 sieve. This indicates a relatively fine material. Soil sieve and hygrometer testing reports show sediment to be gray fat clay and gray lean clay.

Sediment quality data in the report include percent volatile solids, percent total volatile solids, and chemical oxygen demand (COD). Data for four samples indicate the following (all on a percent by weight on a dry basis): volatile solids ranged from 7.6 to 9.4 percent; total volatile solids ranged from 4.3 to 7.2 percent; and COD ranged from 3.1 to 6.0 percent. No other sediment chemical quality data are presented in the report.

Environmental Testing Results

In September 1974, U.S. Army Corps of Engineers prepared a Final Environmental Impact Statement addressing maintenance dredging in Ponce Harbor. The report includes results of analyzing "receiving water" from an unidentified location in the harbor area. The following results are presented in the report without units, which are presumed to be in milligrams per liter: arsenic—0.0035; cadmium—0.0003; chromium—0.0500; copper—0.0027; mercury—0.00025; lead—0.011; and, zinc—0.0132. No other chemical quality data are presented in the report.

Contaminants of Concern

Overview

An assessment of current and prior potential sources of contaminants in the vicinity of the port has been conducted to help determine contaminants of concern in port sediments.

Database compilations of regulatory files were ordered and reviewed, and contacts with regulatory agencies were made to identify possible users of chemicals, sources of chemical releases to the environment, and generators of toxic chemical wastes.

Historical aerial photographs were reviewed to identify current and prior land uses that may relate to chemical use. Interviews were conducted with Port of Ponce personnel with direct historical knowledge of current and former industrial activities and facilities in the vicinity of the port.

Database Regulatory Review

A database search of the Ponce Zip Code (00731) was ordered from Environmental Data Resources, Inc. (EDR), to review federal and state databases for records regarding possible contamination sites, hazardous waste generators, and hazardous material spills in the vicinity of the Ponce Port. The U.S. Environmental Protection Agency (EPA) database Envirofacts (accessible through the EPA Web page www.epa.gov) was also used to search for nearby Resource Conservation and Recovery Act (RCRA) facilities.

The Puerto Rico Environmental Quality Board (EQB) Ponce Office was visited to discuss nearby facilities for which significant regulatory files exist. Information also was obtained regarding nearby leaking underground storage tanks (LUSTs) and registered underground storage tanks (USTs). The Puerto Rico Solid Waste Authority (ADS) provided information regarding nearby landfills.

Results of the database search are summarized below:

- No nearby currently listed or delisted NPL sites were identified.

- The following nearby CERCLIS site was identified:
  RECOVERY & RECYCLING CO. PLAYA DE PONCE
  State RD No. 10 Calle Comerio, Ponce, PR
  EPA ID Number: PRSFN0204257
  Site Description: Active facility, engaged in storing, processing, and recycling of scrap metals, batteries, and transformers.

- No nearby sites with RCRA Corrective Action Report (CORRACTS), which identify hazardous waste handlers with RCRA corrective action activity, were found.

- The following RCRA large quantity generators (LQG) or transporters of hazardous waste were identified:
  Caribe Tuna Inc.
  Ave Santiago de los Caballeros, Ponce, PR 00731
  EPA ID No.: PRD982273229
  Classification: Large Quantity Generator
  Violation Status: Violations Exist; achieved compliance 05/08/1989

  PR Marine Management Inc
  Muelle de Ponce, Ponce, PR 00731

EPA ID No.: PRD 000742999
Record Date: 08/18/1980
Classification: Hazardous Waste Transporter
Violation Status: No violations found

- Eight Federal Emergency Response Notification Systems (ERNS) entries were reported in the database review. These indicate facilities for which releases of oil or hazardous substances have been reported. The information is not specific but indicates that releases to the Ponce Harbor have occurred.

- Additional relevant listings were found in the Envirofacts database for the Ponce area (zip code 00734). Details on the listings are as follows:

  Chemex Corp
  State Road 10, Pier 7 Playa, La Guancha Ward, Ponce, PR, 00734
  Database: RCRAInfo
  Handler Type: Transporter
  EPA ID No. PR0002094753, EPA Handler No. PRR000001586

  PRIDCO – Aljoma Lumber Inc.
  Rd 10, Bldg S1497096, Ponce, PR 00734
  Database: RCRAInfo
  Handler Type: Small Quantity Generator
  EPA Facility ID: 000007484492, EPA Handler No.: PRR000009480

- EQB and EDR identified one nearby Leaking Underground Storage Tank (LUST) site: Caribe Tuna facility at Muelle de Ponce. However, on November 2, 1999, EQB determined that no further action was needed. The Caribe Tuna facility is now closed.

- No nearby facilities were identified on the EQB list of Landfill and Solid Waste Disposal Facilities.

EQB reported an underground storage tank used to store gasoline registered to Caribe Tuna Inc. The Caribe Tuna facility is now closed.

Aerial Photograph Review

Aerial photographs from 1959 and 1993 were reviewed to identify land uses. In 1959, development could be seen near the water in the immediate area of the port. Farther inland, land use was predominately agricultural. Attempts were made to identify occupants of buildings larger than typical dwellings, and this is discussed in the interview section below.

A sugar cane processing mill was more than a mile northeast of the current port. The former port was approximately one-half mile north of the current port offices. Remnants of a World War II military base could be seen occupying the Punta Carenero peninsula. The current marina could be seen, but the landmass connecting it to Punta Carenero had

not been constructed. According to Sr. Jose Quiñones, the fill was deposited from dump trucks, i.e., as opposed to hydraulic dredging.

Interview Information

Sr. Jose Quiñones, a long-term port employee and longer-term resident of the port vicinity, was interviewed. He provided information regarding current and former industrial and commercial activity at the port and in the Playa de Ponce area.

Based on this interview, the following former activities were identified:

- Warehousing
- Livestock slaughtering
- Fabric dyeing
- Hide tanning
- Tuna processing
- Liquid motor fuel bulk storage in aboveground tanks
- Motor vehicle fuel processing (blending but not refining)
- Iron working (believed to include a foundry and mills)
- Portland cement bulk storage in piles and transferring to ships
- Electronic component manufacturing
- Industrial wastewater treatment (for one of two tuna processing plants)
- Pleasure boat manufacturing
- Discharge of untreated domestic sewage

The September 1974 U.S. Army Corps of Engineers Final Environmental Impact Statement addressing maintenance dredging in Ponce harbor identified pollution sources in the harbor vicinity. The above information is consistent with information in that report.

The following current industrial activities take place in the port and vicinity.

- Warehousing
- Food processing, cold storage, and dry storage
- Wood preserving using a copper chromate arsenate process
- Domestic wastewater treatment (with ocean disposal)
- Recycling of primarily metal objects (possibility with other materials or wastes)
- Food bulk storage in aboveground tanks (primarily molasses)
- Chemical transferring from railcars to ships
- Propane bulk storage in aboveground tanks
- Coal bulk storage in piles
- Fueling of ships
- Frequent berthing of ocean-going vessels
- Use of heavy hydraulic equipment for loading/unloading and moving materials

In addition, a range of typical urban commercial activities are known to occur and to have occurred in the central business district of Playa de Ponce north of the current port.

Summary of Relevant Constituents

A range of activities has been identified by reviewing regulatory file information, aerial photograph review, and interviews of port personnel. Based on these activities, the following contaminants may have migrated to sediments:

- Polynuclear aromatics associated with fuels
- Chromium compounds associated with tanning hides
- Lead compounds associated with storage and use as a fuel additive
- Persistent (organochlorine) pesticides associated with agricultural land application
- Chromium, copper, and arsenic compounds associated with wood preserving
- Iron and zinc associated with a foundry
- Silver associated with film processing in the urban area
- Cadmium and lead compounds possibly used as dye pigments
- Selenium possibly associated with electronic component manufacturing
- Organotins typically used in ship hull paints to prevent fouling
- Polychlorinated biphenyls (PCBs) possibly used in hydraulic equipment

In addition to contaminants originating from industrial activities, other constituents may be relevant to assess sediment quality. These constituents include:

- Ammonia may be generated naturally in marine sediments and may correlate to toxicity.
- Aluminum content can be compared to content of certain other metals to help characterize sediment.
- Physical characteristics of sediments are important to help predict how sediments will respond to dredging and disposal

No specific contaminant sources or bases for inclusion were identified for the following constituents: antimony, mercury, thallium, ammonia, cyanide, phenolic compounds, phthalate compounds, dioxins, and furans.

Need for Ocean Disposal

Upland disposal sites are not available for the quantity of material to be dredged. The most viable dredged material disposal option is ocean disposal.

Environmental Impacts of Disposal

Aesthetics

The ocean disposal site is relatively remote. Visual and olfactory impacts are not expected. The most important aesthetic issue is expected to be turbidity in the water column. This will be a short-term impact in a remote location. Likewise, any odors will be short-term and not expected to reach any people. Therefore, no adverse long-term impacts are expected. Short-term adverse impacts may occur but are not expected to affect any significant number of persons.

Recreation Resources

The ocean disposal site is relatively remote. No significant recreational activity is known to occur at the disposal site. Any adverse impacts would require significant use by individuals during the actual period of disposal, and this is not expected to occur. Therefore, no adverse impacts are expected.

Commercial Marine Resources

The ocean disposal site is relatively remote. No frequent commercial fishing is known to occur at the disposal site. Any adverse impacts would require significant use during the actual period of disposal, and this is not expected to occur. Therefore, no adverse impacts are expected.

Navigation

The ocean disposal site is relatively remote. No hazards or obstacles are known that would restrict circumnavigation of the disposal site during disposal activities. Therefore, no adverse short-term impacts are expected. At times other than during disposal, no hazards to navigation would exist. Therefore, no adverse long-term impacts are expected.

Mineral Resources

The disposal site is not known to be a mining area. Therefore, no adverse impacts are expected.

Cultural Resources

The disposal site is relatively remote and deep. It has been determined by the U.S. Army Corps of Engineers to be suitable for use as a dredged material disposal site. Therefore, no adverse impacts are expected.

Endangered Species

The extent of the disposal site is limited and is in 1,200 feet of water. Immobile animal and plant life is expected to be limited. The one square nautical mile of disposal site does not represent a significant portion of the bottom area in the immediate vicinity. It has been determined by the U.S. Army Corps of Engineers to be suitable for use as a dredged material disposal site. Therefore, no significant adverse impacts are expected.

Water Quality

Short-term turbidity impacts to the water column can be expected during disposal activities. No long-term turbidity impacts are expected. Impacts from leaching of other dredged sediment constituents will be evaluated during Tier II testing.

Determination and Findings

Existing information does not provide a sufficient basis for making a decision regarding compliance with section 40 *CFR* 227.13 of the regulations. Therefore, further evaluation is needed to characterize the sediments and to determine potential leaching, toxicity, and bioaccumulation characteristics.

Based on this Tier I assessment, sediment quality testing for the following parameters is warranted:

- Physical characteristics
- Aluminum
- Arsenic
- Cadmium
- Copper
- Chromium
- Iron
- Lead
- Selenium
- Silver
- Zinc
- Organochlorine pesticides
- Polychlorinated biphenyls (PCBs)
- Polynuclear aromatic hydrocarbons
- Total organic carbon
- Monobutyltin, dibutyltin, and tributyltin

Acknowledgement

The assistance and cooperation of Sr. Angel Garcia Bonilla, P.E., of CSA Group, and Sr. Jose Quiñones of the Port Authority of Ponce are gratefully acknowledged. Information provided by these persons has been incorporated into this report.

Appendix B

Ponce Harbor Section 103 Sediment Evaluation Work and Quality Assurance Plan

Ponce ODMDS Sediment Evaluation Work and Quality Assurance Plan

Prepared for:

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Purchase Order Number: C-151
December 2002

INTRODUCTION

This plan presents an approach to chemically and physically characterize sediments from the Ponce Ocean Dredged Material Disposal Site (ODMDS) and to provide hydrographic data for application of appropriate fate programs from the collection of ADDAMS modeling modules. This work is being performed under the oversight of the Puerto Rico Infrastructure Financing Authority (AFI).

This document is presented in three sections:

Section 1. Sampling Plan and Protocol. This section provides information about the proposed project area, identifies the contaminants of interest, sampling plan design, field sampling and measurement procedures, and identifies laboratory test methods and detection limits.

Section 2. Project and Field Quality Assurance Plan. This plan provides information on overall project organization, assignment of responsibilities, sample handling and transport, and field-specific aspects of quality assurance (QA).

Section 3. Chemical and Physical Testing Quality Assurance Plan. This section provides laboratory-specific information on laboratory organization, QA objectives, and methods used for the laboratory testing of sediment samples.

Section 1

Ponce ODMDS Sediment Characterization Sampling Plan and Protocol

INTRODUCTION

This plan presents an approach to chemically and physically characterize sediments from the Ponce Ocean Dredged Material Disposal Site (ODMDS) and to provide hydrographic data for application of appropriate fate programs from the collection of ADDAMS modeling modules. This proposed work is independent of any evaluation of Ponce Harbor sediment for 103 certification in accordance with the 1991 "Green Book" ("Evaluation of Dredged Material Proposed for Ocean Disposal - Testing Manual").

DESCRIPTION OF THE DISPOSAL SITE

The proposed disposal site is the Ponce ODMDS. This site is located approximately 6 nautical miles offshore from the Island of Puerto Rico near Ponce Harbor and is designated for the disposal of dredged material. The site is one nautical mile square with center coordinates of 17° 53' 30"N, 66° 37' 11"W and is in water that averages 1,400 feet (425 meters) deep. Using the NAD 27 datum, the corners of the ODMDS are located at:

17° 54' 00" N, 66° 37' 43"W
17° 54' 00" N, 66° 36' 41"W
17° 53' 00" N, 66° 36' 41"W
17° 53' 00" N, 66° 37' 43"W

Section 506 of the Water Resources and Development Act (WRDA) of 1992, which amended the Marine Protection, Research and Sanctuaries Act of 1972 (MPRSA), requires the U.S. Environmental Protection Agency (EPA) and the U.S. Army Corps of Engineers (USACE) to prepare a Site Management and Monitoring Plan (SMMP) for each ocean dredged material disposal site. A site management and monitoring plan will be developed, coordinated, and approved for this site prior to any future dredge material disposal activity. This plan will identify a number of actions, provisions and practices to manage operational aspects of dredging and disposal activities and to perform site monitoring.

EVALUATION NEEDS

Potential Contaminants of Concern

The potential contaminants of concern for this project are listed below. The list includes regulated as well as other common, unregulated contaminants.

The list of analytes suggested by the Regional Implementation Manual, with some modification based on guidance by EPA (Region II) and the Jacksonville District Corps, is proposed. Sediments will be tested for the following parameters:

Section 1

Grain Size Distribution

Percent Solids

Metals

Aluminum (sediment only)	Lead
Arsenic	Mercury
Cadmium	Nickel
Chromium	Silver
Copper	Zinc

Total Organic Carbon (TOC)

Organotin

Chlorinated Hydrocarbon Pesticides

Aldrin	Dieldrin
Chlordane	Endosulfan I
2, 4' –DDD	Endosulfan II
2, 4' –DDE	Endosulfan Sulfate
2, 4' -DDT	Heptachlor
4, 4' -DDD	Heptachlor Epoxide
4, 4' -DDE	Trans-nonachlor
4, 4' -DDT	

Polychlorinated Biphenyls Congeners (IUPAC Numbers)

PCB 8	PCB 18	PCB 28	PCB 44	PCB 49
PCB 52	PCB 66	PCB 87	PCB 101	PCB 105
PCB 118	PCB 128	PCB 138	PCB 153	PCB 170
PCB 180	PCB 183	PCB 184	PCB 187	PCB195
PCB 206	PCB 209			

Polynuclear Aromatic Hydrocarbons (PAHs)

Acenaphthene	Chrysene
Acenaphthylene	Dibenzo (a,h) Anthracene
Anthracene	Fluoranthene
Benzo (a) Anthracene	Fluorene
Benzo (a) Pyrene	Indeno(1,2,3-cd) Pyrene
Benzo (b) Fluoranthene	Naphthalene
Benzo (k) Fluoranthene	Phenanthrene
Benzo (g,h,i) perylene	Pyrene
Biphenyl	

A plan for sampling and testing of representative ODMDS and reference sediment samples is detailed below.

Section 1

ODMDS SAMPLING PLAN DESIGN

Project Coordination and Quality Assurance

Testing for the Ponce ODMDS project will be provided under the direction of PPB Environmental Laboratories, Inc. (PPB; project management; Gainesville, Florida) with the assistance of Continental Shelf Associates, Inc. (CSA; sediment and water sampling; Jupiter, Florida), Harding/ESE (HESE, ADDAMS Modeling; Gainesville, Florida), Battelle (physical and chemical testing; Duxbury, MA and Sequim, WA), and Water and Air Research, Inc. (W&AR; quality assurance; Gainesville, Florida) as major subcontractors.

Before initiating field activities, the approved sampling plan and protocol document will be distributed to project members along with other pertinent information and documentation that relate to the tasks team members perform, e.g., reference station locations, marine charts, names and telephone numbers of people whose involvement is necessary for the successful completion of this project.

A project initiation meeting will be held to ensure that all team members understand their roles and assigned tasks. Representatives of all team member organizations with major roles should be present. At this meeting, the role of each team member will be restated and/or clarified; questions can be asked about logistics, responsibilities, and assignments; agreement will be reached regarding project milestones and timeframes; and contingency plans will be formulated for foreseeable problems (e.g., inclement weather, equipment malfunctions).

Project managers for AFI, US Army Corps of Engineers (Corps), and USEPA Region II (EPA) will review the field and laboratory quality assurance plans and standard operating procedures for project team members. Such plans will be approved by the Commonwealth of Puerto Rico or the state having authority over the business enterprise.

A field quality assurance audit will be conducted to document all aspects of sediment and data collection; sample handling, labeling, transport, and preservation; and documentation of sampling activities and information.

A final report will be submitted documenting field-sampling activities and test results. This report will include information on sediment sampling locations and the physical and chemical test results. Results of fate and transport modeling using the ADDAMS computer modules and collected data will be presented.

Sample Station Selection and Identification

Sampling will occur at the Ponce ODMDS and at two references stations. Before sediment collection begins, sampling locations should be reviewed by EPA. A list of sample station locations (i.e., latitudes and longitudes) will be provided to the sample collection contractor no later than the project initiation meeting.

All sampling locations will be documented using differentially-corrected Global Positioning System (DGPS) receivers. Correction can be based on the US Coast Guard radio beacons or

on the Wide Area Augmentation System (WAAS) signal.

ODMDS Stations. Stations in the square-shaped ODMDS were selected to distribute 18 sampling points almost evenly over the entire area. Thirteen sampling points are distributed at equal distances along two lines forming an "X" that connect the four corners and the center. Five of the points are located at the corners and center of the one square mile ODMDS. Four other stations are positioned as midpoints between the four points on the "X" that are closest to the corner points. A single sampling location is located midway between two points closest to the center point and on different lines of the "X". Figure 1 shows the distribution of these points in the ODMDS. Samples from the ODMDS will be identified as E-PODS03-1 through E-PODS03-18.

The field sampling team leader will select five ODMDS stations for sampling in triplicate. The identifiers for these samples will be modified to reflect an "A", "B", or "C" replicate, e.g., if Station E-PODS03-5 is selected the three samples would be labeled E-PODS03-5A, E-PODS03-5B, and E-PODS03-5C.

Reference Stations. Two reference stations are proposed for use for this project and these are located between the Ponce ODMDS and Ponce Harbor. The proposed coordinates of these reference stations are:

	NAD 27		NAD 83		Station
	Easting	Northing	Easting	Northing	
RS-PH03A	130956	11615	178596	211397	
RS-PH03B	133520	9438	181160	209220	

State plane coordinates were originally specified as NAD 27, Puerto Rico in meters, and will be used by the field team as NAD 83, Puerto Rico in meters.

For comparison with ODMDS sediments, the sediment at each reference station will be collected as three independent samples centered around the same station. These reference sediment samples will be identified as RS-PH03-1A, -1B, and -1C for Station RS-PH03-1 and RS-PH03-2A, -2B, and 2C for Station RS-PH03-2. They will be analyzed for the same parameters as the ODMDS sediment samples, i.e., grain size distribution, TOC, metals, pesticides, PCBs, PAHs, and percent solids. Reference stations are shown of Figure 1.

Field-Related Quality Control Sediment Samples

Field-generated quality control samples will be used during this sediment evaluation. Laboratory generated quality control samples (including standard reference materials, matrix spikes, and others) will be discussed in a separate section of this Work/QA Plan.

Triplicate Samples. Because triplicate samples will be collected for five of the ODMDS stations and both reference stations, no field duplicate samples will be collected for that area.

As stated earlier, both reference stations will be sampled using separate grab sampler deployments for each replicate sample.

Section 1

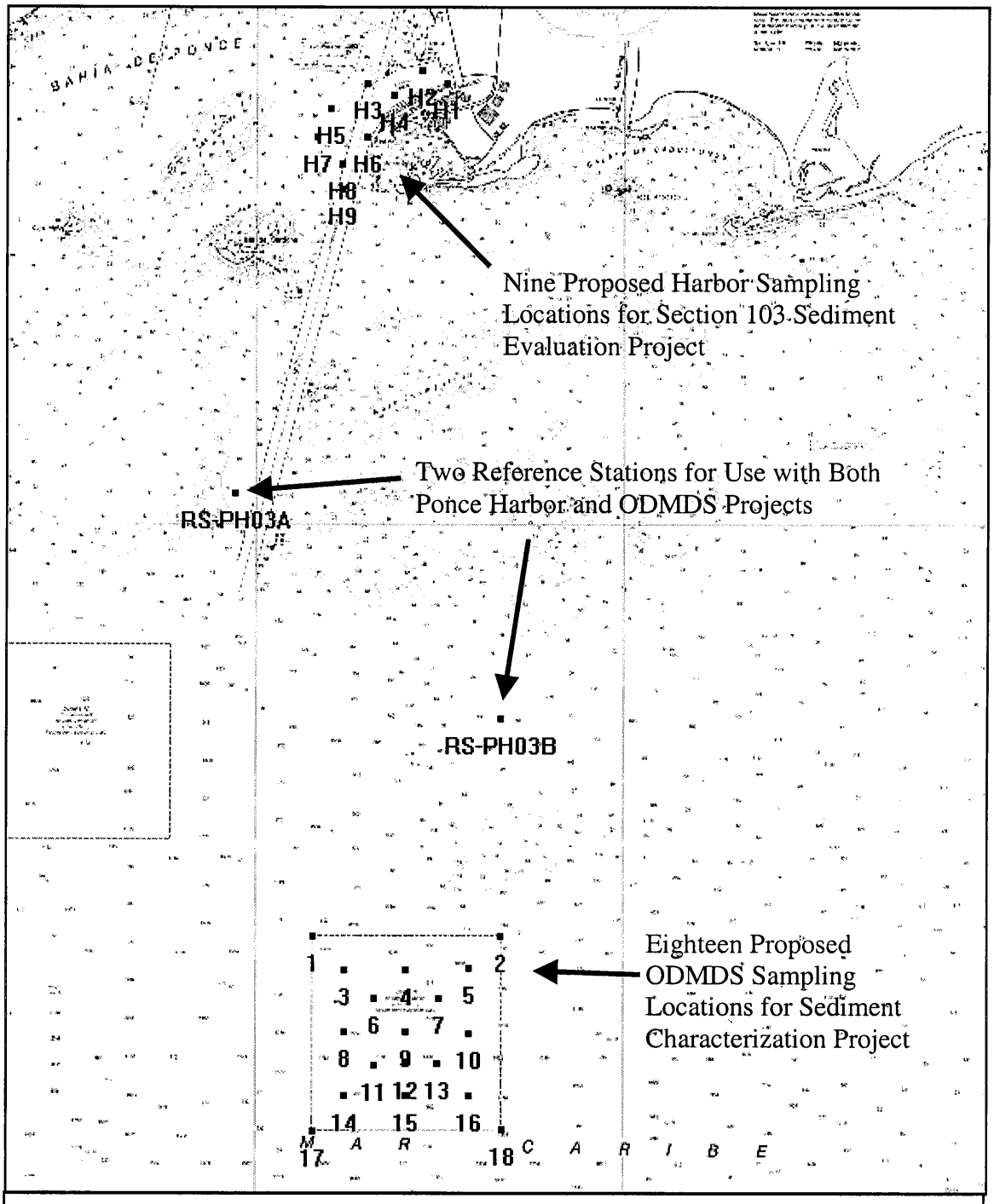

Figure 1.
Proposed Sampling Locations for Reference Stations to Be Used for Both Sediment Testing Projects Associated with Ponce Harbor, Puerto Rico. True north is to the top of the figure. The Ocean Dredged Material Disposal Site (ODMDS) of interest is approximately one nautical mile square. Map project is mercator and the datum is NAD 83.

Section 1

Sampling Methods and Field Procedures.

Field Equipment. Equipment shipped to the Ponce area in preparation for the ODMDS field work should be placed in containers that will protect the items from damage due to rough handling and wet weather conditions. To the extent practical, critical items will be duplicated so that failure of any one item will not stop the field work. Similarly, spare parts and replacement items will be included as appropriate to prevent avoidable down time. When practical, sampling equipment will be pre-cleaned before shipment to the field if it can be ensured that integrity of such equipment will not be compromised during shipment and storage. All sample containers will be supplied by the laboratory and the integrity of all containers must be maintained during shipment to and from the Ponce area and while in storage awaiting use.

Decontamination and Clean Area Procedures. All equipment surfaces that potentially contact sample material will be decontaminated, e.g., stainless steel spoons and bowls. Such equipment will be cleaned using a water/Liqui-nox solution, followed by a tap or clean sea water rinse, isopropanol rinse, and air dried. If not immediately used after cleaning, decontaminated equipment will be wrapped in food-grade aluminum foil with the shiny side facing/contacting the equipment. Field personnel will wear gloves that are non-powdered, talc- and phthalate-free, e.g., Nitrile, and will change them between sampling stations, when cleaning equipment, and when they become "dirty" or become ripped or torn.

After any welding has been finished, the deck area where the sampler is positioned and sample handling occurs will be scrubbed with Liqui-nox solution or other appropriate non-contaminating cleaner to remove petroleum residues, loose debris, and any other materials that might contaminate sampled sediment. If, in the opinion of the field team leader, additional protective measures are needed polyethylene or polypropylene sheeting, e.g., visqueen, will be applied to the deck.

Hydraulic and petroleum-fueled equipment will be maintained in an area away from the sampling area and any drips or leaks of hydraulic fluids, fuels, or lubricants will be cleaned up immediately. To the extent possible, gasoline- and/or diesel-fueled equipment will be positioned downwind to minimize the potential for sample contamination.

Sample collection and handling. All samples will be collected using appropriately cleaned, non-contaminating equipment (e.g., constructed of stainless steel or coated with Teflon®) as required for the analyses of interest.

Triplicate samples from the ODMDS stations will be generated by dividing the exposed (upper) sediment surface in a box core grab sampler into three similar rectangular sections and removing sediment to a sufficient depth from each section to obtain the required one liter. Each replicate sample will be homogenized using an appropriately cleaned stainless steel spoon and bowl before it is containerized.

Reference samples may be collected by grab sampler such as a box corer or van Veen. Collected sediment will be placed in a container made of stainless steel or other material with a protective coating of Teflon® or similar material. Excess water will be decanted before homogenizing and containerizing the sample.

If sample collection problems are encountered, e.g., finding a sediment-free hard bottom or strong currents misaligning the sampler, either the on-site EPA representative will be consulted for guidance or the professional judgement of the field team leader will be used to determine the best course of action, e.g., accepting the sample as collected, relocating to another appropriate sampling location, or taking other corrective actions.

The homogenized sediment of each sample will be used to fill four 250 mL glass jars with Teflon®-lined lids for shipment to the laboratories. Two containers from each station will be shipped overnight on wet ice to Battelle for testing. The remaining two containers from each station will be stored on wet ice and sent to PPB to be archived. Samples will be shipped in ice chests secured with custody seals in a manner that will disclose if any ice chest were opened before delivery to the laboratory.

The attached Table 1 is included to show the numbers and types of samples to be collected or generated and the tests that will be performed. Included on this table are some USEPA Region II-specified, laboratory-generated quality control samples; other laboratory-generated quality control samples are described in the Chemical and Physical Evaluation QAP section.

Hydrographic Measurements

Collection of in situ water quality data, normally collected for 103 certification, is not required for this project.

Conductivity, temperature, and depth (CTD) data will be collected from the ODMDS area. Two CTD profile data sets will be collected every other day, one profile in the morning and one profile in the afternoon, while sampling operations are occurring in the ODMDS area. These data will be used to describe the physical structure of the water column and to support ADDAMS (STFATE) modeling. The specific locations where these data are acquired within the ODMDS are not important, as the hydrographic features are not expected to vary significantly within the site.

All field sheets will be provided to AFI and the Corps upon the completion of fieldwork.

Section 1

Table 1. Number and Type of Sediment Samples to Be Collected for Ponce ODMDS Evaluation.

Sediment Matrix Only	Metals	Pesticides	PCB Congeners	PAHs	TOC	Grain Size
13 ODMDS Station in Singlet	13	13	13	13	13	13
5 ODMDS Stations in Triplicate	15	15	15	15	15	15
2 Reference Stations in Triplicate	6	6	6	6	6	6
Total	34	34	34	34	34	34

Note: Please see Table 1 of Section 3 of this Work/QA Plan for a listing of quality control samples.

Sediment Physical Analyses

The following physical analysis will be conducted on each ODMDS and reference sediment sample including the triplicate samples.

Test Parameter	Test Method	Detection Limit
1. Grain Size Analysis	Plumb 1981	1.0%
2. Percent Solids	SM2540G	1.0 %

Sediment Chemical Analyses.

Sediment chemical analyses (bulk sediment tests) have been assembled into groups and include all elements, compounds or processes using the methods and detection limits listed below. All sediment analyses will be performed on every sediment sample including the triplicate samples.

Test Parameter	Method to be Used	Detection Limit (dry weight basis)
1. Metals		
a. Aluminum	EPA 1638/1640	0.5 mg/kg
b. Arsenic	EPA 1638/1640	0.05 mg/kg
c. Cadmium	EPA 1638/1640	0.01 mg/kg
d. Chromium	EPA 1638/1640	0.04 mg/kg
e. Copper	EPA 1638/1640	0.02 mg/kg
f. Lead	EPA 1638/1640	0.01 mg/kg
g. Mercury	EPA 245.5	0.002 mg/kg
h. Nickel	EPA 245.5	0.03 mg/kg
i. Silver	EPA 1638/1640	0.03 mg/kg
j. Zinc	EPA 1638/1640	0.1 mg/kg

Note: Many of these methods are modified. Refer to Section 3.

Section 1

2. Organic Compounds
   a. Pesticides

1) Aldrin	8081	0.01 ng/g
2) Chlordane	8081	0.01 ng/g
3) 2, 4' –DDT	8081	0.08 ng/g
4) 2, 4' –DDD	8081	0.01 ng/g
5) 2, 4' -DDE	8081	0.07 ng/g
6) 4, 4' –DDT	8081	0.02 ng/g
7) 4, 4' –DDD	8081	0.01 ng/g
8) 4, 4' –DDE	8081	0.02 ng/g
9) Dieldrin	8081	0.03 ng/g
10) Endosulfan I	8081	0.03 ng/g
11) Endosulfan II	8081	0.01 ng/g
12) Endosulfan Sulfate	8081	0.01 ng/g
13) Heptachlor	8081	0.02 ng/g
14) Heptachlor Epoxide	8081	0.02 ng/g
15) Trans nonachlor	8081	0.01 ng/g

Note: Many of these methods are modified. Refer to Section 3.

   b. Polychlorinated Biphenyls Congeners

1) PCB 8	8082	0.1 ng/g
2) PCB 18	8082	0.01 ng/g
3) PCB 28	8082	0.01 ng/g
4) PCB 44	8082	0.02 ng/g
5) PCB 49	8082	0.01 ng/g
6) PCB 52	8082	0.01 ng/g
7) PCB 66	8082	0.02 ng/g
8) PCB 87	8082	0.01 ng/g
9) PCB 101	8082	0.01 ng/g
10) PCB 105	8082	0.02 ng/g
11) PCB 118	8082	0.02 ng/g
12) PCB 128	8082	0.02 ng/g
13) PCB 138	8082	0.02 ng/g
14) PCB 153	8082	0.02 ng/g
15) PCB 170	8082	0.02 ng/g
16) PCB 180	8082	0.02 ng/g
17) PCB 183	8082	0.02 ng/g
18) PCB 184	8082	0.02 ng/g
19) PCB 187	8082	0.02 ng/g
20) PCB 195	8082	0.02 ng/g
21) PCB 206	8082	0.02 ng/g
22) PCB 209	8082	0.02 ng/g

Note: Many of these methods are modified. Refer to Section 3.

   c. Total Organic Carbon 0.1 %
      Note: Analyzed as per EPA Edison, New Jersey, Laboratory procedure, EPA 1986

d. Monobutyltin	Battelle SOP No. 5-196-07	1.0347 ng/g
Dibutyltin	Battelle SOP No. 5-196-07	0.7191 ng/g
Tributyltin	Battelle SOP No. 5-196-07	0.5929 ng/g
Tetrabutyltin	Battelle SOP No. 5-196-07	0.5929 ng/g

e. PAHs

1) Acenaphthene	8270	0.04 ng/g
2) Acenaphthylene	8270	0.04 ng/g
3) Anthracene	8270	0.03 ng/g
4) Benzo (a) Anthracene	8270	0.05 ng/g
5) Benzo (a) Pyrene	8270	0.1 ng/g
6) Benzo (b) Fluoranthene	8270	0.04 ng/g
7) Benzo (k) Fluoranthene	8270	0.03 ng/g
8) Benzo (g,h,i) perylene	8270	0.05 ng/g
9) Biphenyl	8270	0.05 ng/g
9) Chrysene	8270	0.04 ng/g
10) Dibenzo (a,h) Anthracene	8270	0.04 ng/g
11) Fluoranthene	8270	0.04 ng/g
12) Fluorene	8270	0.04 ng/g
13) Indeno(1,2,3-c,d) Pyrene	8270	0.08 ng/g
14) Naphthalene	8270	0.1 ng/g
15) Phenanthrene	8270	0.05 ng/g
16) Pyrene	8270	0.04 ng/g

Note: Many of these methods are modified. Refer to Section 3.

Section 2

Ponce ODMDS Sediment Characterization Project and Field Quality Assurance Plan

Introduction

Section 2 presents information on the field work and overall project organization and field activities for the Ponce ODMDS sediment characterization. A laboratory-specific quality assurance plan appears in Section 3.

Signature Page (for Ponce ODMDS Sediment)

I certify under penalty of law that this document and all attachments were prepared under my direction or supervision. The information submitted is, to the best of my knowledge and brief, true, accurate, and complete. I am aware there are significant penalties for submitting false information, including the possibly of fine and imprisonment for knowing violations.

Ward Dickens 12/19/02	Bryan F. Cotter 12/19/02
Ward Dickens Date	Bryan F. Cotter Date
PPB Project Director	PPB Project Manager
Charles R. Fellows 12/19/02	Paul Berman 12/19/02
Charles R. Fellows Date	Paul Berman Date
Project QA Coordinator	PPB QA Officer

Section 2

Signature Page (For the PONCE ODMDS QAPP)

I certify under penalty of law that this document and all attachments were prepared under my direction or supervision. The information submitted is, to the best of my knowledge and brief, true, accurate, and complete. I am aware there are significant penalties for submitting false information, including the possibly of fine and imprisonment for knowing violations.

Frederick B. Ayer, II – CSA Field Operations Director
(Direct Supervisor for Frank Johnson - Field Team Leader)

12/19/02
Date

Section 2

Signature Page (for Ponce ODMDS Sediment)

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Carole S. Peven McCarthy	12/16/2002	Rosanna Buhl	12-16-02
Carole S. Peven McCarthy	Date	Rosanna Buhl	Date
Battelle Organics Project Manager		Battelle Organics QA Officer	

Section 2

Signature Page (for Ponce ODMDS Sediment)

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Elisabeth S. Barrows 12/19/02
Elisabeth S. Barrows Date
Battelle Metals Project Manager

Deborah Coffey 12/19/02
Deborah Coffey Date
Battelle Metals QA Officer

Section 2

Signature Page (for Ponce ODMDS Sediment)

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12/19/02	12/19/02
Kenneth Davis Date	Jennifer Davis Date
Applied Marine Science Project Manager	Applied Marine Science QA Officer

Section 2

Signature Page (for Ponce ODMDS Sediment)

I certify under penalty of law that this document and all attachments were prepared under my direction or supervision. The information submitted is, to the best of my knowledge and brief, true, accurate, and complete. I am aware there are significant penalties for submitting false information, including the possibly of fine and imprisonment for knowing violations.

Kenneth Davis Date	Jennifer Davis Date
Applied Marine Science Project Manager	Applied Marine Science QA Officer

Section 2

Signature Page (for Ponce ODMDS Sediment)

I certify under penalty of law that this document and all attachments were prepared under my direction or supervision. The information submitted is, to the best of my knowledge and brief, true, accurate, and complete. I am aware there are significant penalties for submitting false information, including the possibly of fine and imprisonment for knowing violations.

Héctor Jiménez Juarbe, Esq. 12/18/02
Executive Director, Port of the Americas

Ferdinand Quiñones 12/18/02
CSA Group Project Manager

Section 2

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Section 2

1 Project Description

This Project and Field Quality Assurance Plan is a portion, Section 2, of the Work/QA Plan for the chemical and physical characterization of sediments from the Ponce Ocean Dredged Material Disposal Site (ODMDS). Hydrographic data also will be collected for application of appropriate fate programs from the collection of ADDAMS modeling modules. This sediment characterization will be performed under the oversight of the Puerto Rico Infrastructure Financing Authority (AFI). This section describes the overall project organization, project assignments for the various team members, and presents the field measurement and sampling quality assurance procedures.

Another Work/QA Plan has been developed in parallel for Ponce Harbor to address the chemical analysis of sediments, waters, and elutriates; water column and sediment bioassays; and bioaccumulation studies to determine if there is significant potential for an unacceptable adverse impact to occur at the Ponce ODMDS.

The proposed disposal site is the Ponce ODMDS. This site is located approximately six nautical miles offshore from the Island of Puerto Rico near Ponce Harbor and is designated for the disposal of dredged material. The site is one nautical mile square with center coordinates of 17° 53' 30"N, 66° 37' 11"W and is in water that averages 1,400 feet (425 meters) deep. Using the NAD 27 datum, the corners of the ODMDS are located at:

17° 54' 00" N,	66° 37' 43" W
17° 54' 00" N,	66° 36' 41" W
17° 53' 00" N,	66° 36' 41" W
17° 53' 00" N,	66° 37' 43" W

The following contaminants of concern have been identified:

Metals
- Aluminum
- Arsenic
- Cadmium
- Chromium
- Copper
- Lead
- Mercury
- Nickel
- Silver
- Zinc

Organotins
Total Organic Carbon (TOC)
Organochlorine Pesticides
Polychlorinated Biphenyls Congeners (PCBs)
Polynuclear Aromatic Hydrocarbons (PAHs)

Section 2

2 Quality Assurance Organization and Responsibilities

This sediment evaluation investigation is being performed for the Infrastructure Financing Authority of Puerto Rico (AFI) by PPB Environmental Laboratories, Inc. (PPB) and subcontractors to PPB. Figure 2-1 presents an organizational chart showing the management structure, personnel assignments, and functional assignment for each corporate participant.

AFI, as the permit applicant, will submit all plans, test results, and other project-related documents to the US Environmental Protection Agency (EPA) and the US Army Corps of Engineers (Corps). CSA Group will assist AFI as its technical representative.

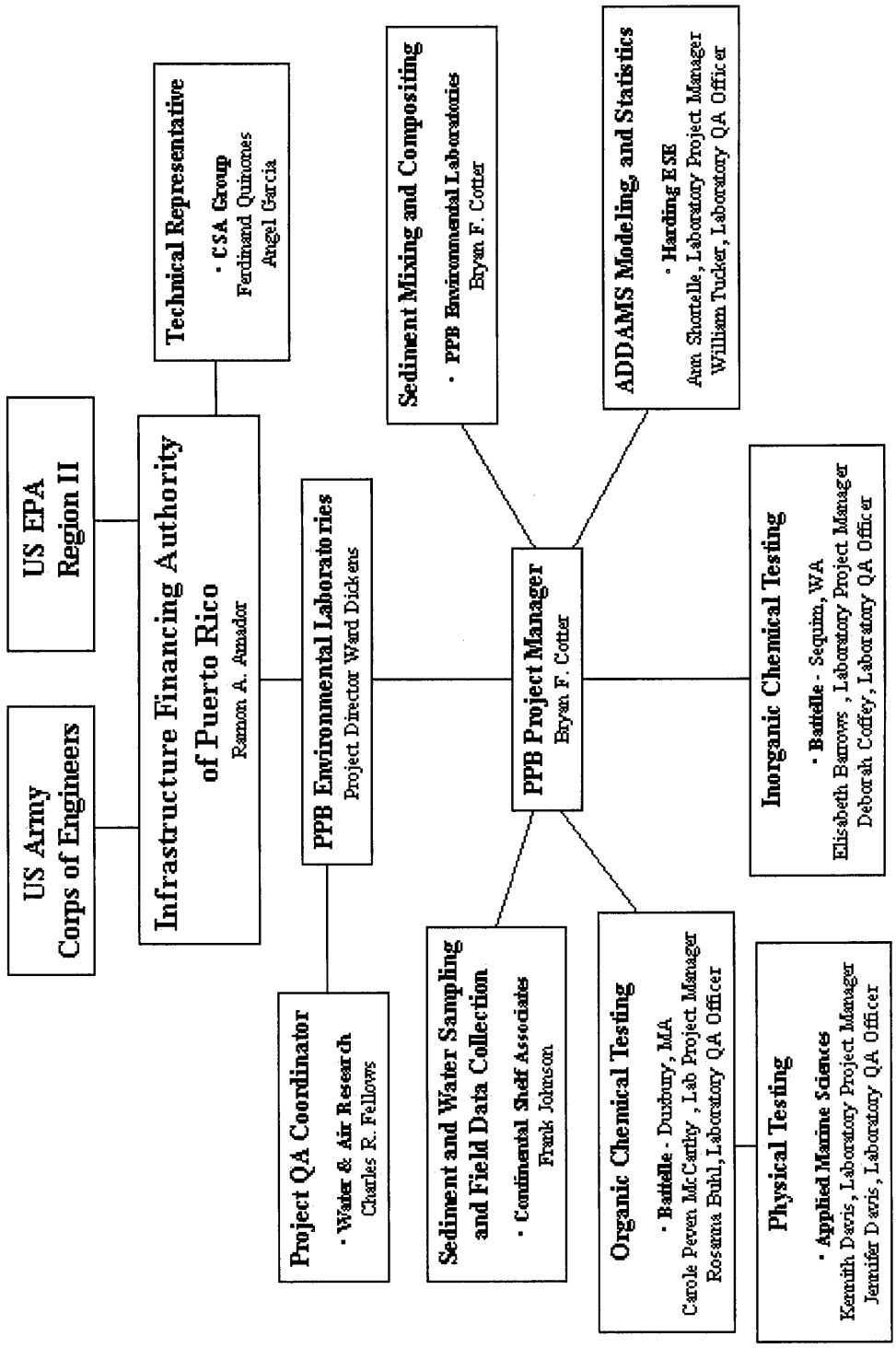
Ward Dickens is the Project Director for PPB and serves as head administrative officer for this project. Mr. Dickens will coordinate all contractual agreements with AFI and the PPB subcontractors and will ensure that all PPB deliverables are sent to AFI in a timely manner. He will submit progress reports at 2-week intervals describing status of work tasks, problems encountered, corrective actions initiated, and work tasks to be undertaken during the next 2-week period.

As the PPB Project Manager, Bryan F. Cotter is responsible for coordinating all project tasks among PPB personnel and subcontractors and has the overall responsibility for assuring data quality. Mr. Cotter will ensure that any appropriate corrective actions are implemented during sample collection, sample analysis, and data assessment. Mr. Paul Berman will have QA oversight for project activities occurring at the PPB laboratory.

The Project QA Coordinator is Charles R. Fellows, who will be relied upon by the Project Manager to review the quality of all collected data before it is reported. He will inform the Project Manager of any needed corrective actions. Mr. Fellows will review sample collection activities and may be asked by the Project Manager to review sample testing activities in any of the analytical laboratories. He will review all laboratory test reports and will evaluate reported data with the reported laboratory QC data as well as the criteria included in this QAPP.

For this project, each physical and chemical testing laboratory used will have an assigned Laboratory Project Manager and Laboratory QA Officer. The names of these individuals, along with their organization, and project role, are shown on Figure 2-1. Each Laboratory Project Manager will be responsible for coordinating with the PPB Project Manager for scheduling and sample receipt. Within their laboratory, the Laboratory Project Manager will follow the progress of sample processing, analysis, and reporting. The Laboratory QA Officer will be responsible for reviewing data quality as they relate to the laboratory QA program and this QAPP. The Laboratory Project Manager will be notified of any test results that are found to be deficient in regard to quality and corrective actions will be developed and implemented. The PPB Project Manager will be notified of all deficiencies requiring corrective actions by laboratory personnel.

Figure 2-1. Organization Chart for the Ponce Harbor Sediment Evaluation



Section 2

The field sampling organization and each testing laboratory has an established Quality Assurance Program that has been approved under appropriate state programs for personnel training, documentation, sample handling, storage, processing, testing, use of laboratory quality control samples, and reporting. Consequently, this project will rely on use of existing and established in-house QA programs.

Initial communications among project personnel will be by telephone and/or e-mail. Additional communications may include the US postal service, Federal Express, and facsimile transmissions. The telephone numbers and e-mail addresses for project personnel follow:

Name	Telephone Number	e-mail Address
Ramon A. Amador	787/641-6801	ramador@afi.gobierno.pr
Ferdinand Quinones	787/763-5757 Ext: 1621	fquinones@csagroup.com
Angel Garcia	787/641-6801 Ext: 708	agarcia@csagroup.com
Ward Dickens	352/377-2349	wdickens@ppb-envlabs.com
Bryan F. Cotter	352/377-2349	bcotter@ppb-envlabs.com
Paul Berman	352/377-2349	pberman@ppb-envlabs.com
Charles R. Fellows	352/372-1500	cfellows@waterandair.com
Frank Johnson	561/746-7946	fjohnson@conshelf.com
Ann Shortelle	352/333-2623	abshortelle@mactec.com
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Elisabeth Barrows	360/681-3643	es.barrows@pnl.gov
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Carole S. Peven McCarthy	781/952-5232	peven@battelle.org
Deborah Coffey	360/681-3645	DeborahCoffey@pnl.gov
Kenneth Davis	281/554-7272	kdavis1ams@aol.com
Jennifer Davis	281/554-7272	jdavis@appliedmarinesciences.com

Section 2

3 Quality Assurance Objectives

Data quality objectives are specified to set performance-based goals for target detection limits, accuracy (defined as bias and precision), precision, and database completeness. Meeting these objectives ensures that the data generated will be of sufficiently good quality to address project decision-making issues and provide data that can be compared with data from other similar projects.

Field data quality objectives are presented on Table 3-1. This table provides information on the field test methods that will be used, the target detection limits and reporting units, accuracy, precision. Data quality objectives for laboratory tests are presented in Section 3 of this Work/QA Plan.

The overall completeness goal for this project is to generate acceptable data for 95 percent of the total data planned. For individual tests, the completeness goal is to generate acceptable data for at least all but three of the sediment samples collected.

Table 3-1. Field Data Quality Objectives.

Matrix and Analyte	Method	Target Detection Limit	Reporting Units	Accuracy Goal	Precision Goal
Conductivity	120.1	0.5	mmhos/cm	---	---
Temperature	170.1	0.5	°C	---	---

Please note these detection limits are "target limits" or, for temperature, the precision to which data will be reported.

4 Standard Operating Procedures

The following procedures will be used during the field activities.

The field team for Continental Shelf Associates and the laboratories at Battelle and Applied Marine Sciences have established and state-approved procedures for all aspects of environmental sample collection and analysis and data handling and reporting. These established, organization-specific procedures will be followed by each corporate team member for the project tasks assigned to them.

Some standard operating procedures (SOPs) are presented in Appendix A of this section to address specific field activities that are important to this sediment evaluation project.

Section 2

5 Sampling Strategy and Procedures

This subsection provides general descriptive information regarding sampling approach and field procedures. Much of this text appeared in Section 1 of this Work/QA Plan. Complete descriptions for many of the procedures discussed in this subsection are presented in Appendix A, Standard Operating Procedures.

Presampling Coordination

Testing for the Ponce ODMDS project will be provided under the direction of PPB Environmental Laboratories, Inc. (PPB; project management; Gainesville, Florida) with the assistance of Continental Shelf Associates, Inc. (CSA; sediment and water sampling; Jupiter, Florida), Harding/ESE (HESE, ADDAMS Modeling; Gainesville, Florida), Battelle (physical and chemical testing; Duxbury, MA and Sequim, WA), and Water and Air Research, Inc. (W&AR; quality assurance; Gainesville, Florida) as major subcontractors.

Before initiating field activities, the approved sampling plan and protocol document will be distributed to project members along with other pertinent information and documentation that relate to the tasks team members perform, e.g., reference station locations, marine charts, names and telephone numbers of people whose involvement is necessary for the successful completion of this project.

A project initiation meeting will be held to ensure that all team members understand their roles and assigned tasks. Representatives of all team member organizations with major roles should be present. At this meeting, the role of each team member will be restated and/or clarified; questions can be asked about logistics, responsibilities, and assignments; agreement will be reached regarding project milestones and timeframes; and contingency plans will be formulated for foreseeable problems (e.g., inclement weather, equipment malfunctions).

Sample Station Locations and Identifiers

Sampling will occur at the Ponce ODMDS and at two references stations. Before sediment collection begins, sampling locations should be reviewed by EPA. A list of sample station locations (i.e., latitudes and longitudes) will be provided to the sample collection contractor no later than the project initiation meeting.

All sampling locations will be documented using differentially-corrected Global Positioning System (DGPS) receivers. Correction can be based on the US Coast Guard radio beacons or on the Wide Area Augmentation System (WAAS) signal.

ODMDS Stations. Stations in the square-shaped ODMDS were selected to distribute 18 sampling points almost evenly over the entire area. Thirteen sampling points are distributed at equal distances along two lines forming an "X" that connect the four corners and the center. Five of the points are located at the corners and center of the one square mile ODMDS. Four other stations are positioned as midpoints between the four points on the "X" that are closest to the corner points. A single sampling location is located midway between two points closest to the center point and on different lines of the "X". Figure 5-1 shows the

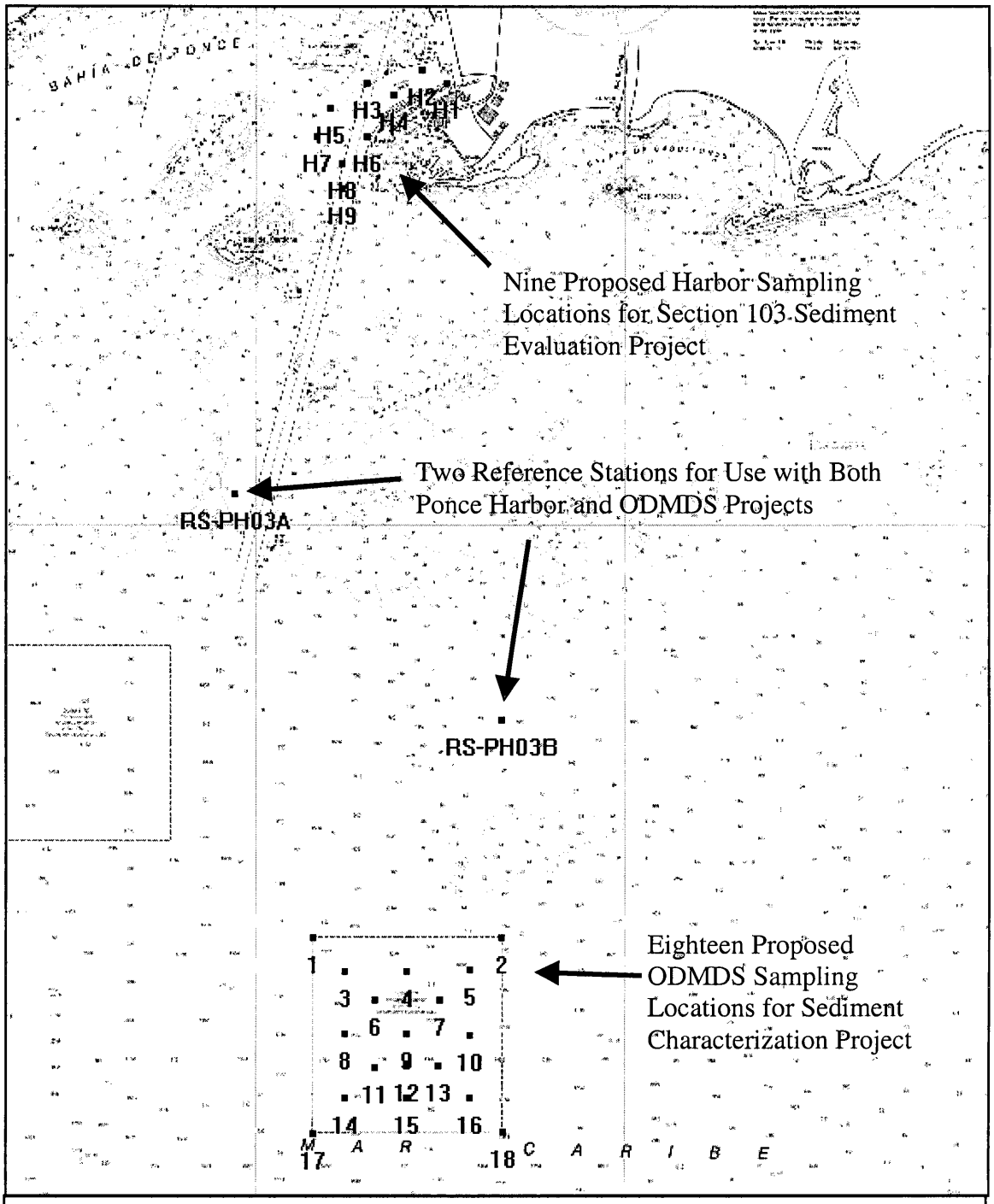

Figure 5-1.
Proposed Sampling Locations for Reference Stations to Be Used for Both Sediment Testing Projects Associated with Ponce Harbor, Puerto Rico. True north is to the top of the figure. The Ocean Dredged Material Disposal Site (ODMDS) of interest is approximately one nautical mile square. Map project is mercator and the datum is NAD 83.

distribution of these points in the ODMDS. Samples from the ODMDS will be identified as E-PODS03-1 through E-PODS03-18.

The field sampling team leader will select five ODMDS stations for sampling in triplicate. The identifiers for these samples will be modified to reflect an "A", "B", or "C" replicate, e.g., if Station E-PODS03-5 is selected the three samples would be labeled E-PODS03-5A, E-PODS03-5B, and E-PODS03-5C.

Reference Stations. Two reference stations are proposed for use for this project and these are located between the Ponce ODMDS and Ponce Harbor. The proposed coordinates of these reference stations are:

	NAD 27		NAD 83	
Station	Easting	Northing	Easting	Northing
RS-PH03A	130956	11615	178596	211397
RS-PH03B	133520	9438	181160	209220

State plane coordinates were originally specified as NAD 27, Puerto Rico in meters, and will be used by the field team as NAD 83, Puerto Rico in meters.

For comparison with ODMDS sediments, the sediment at each reference station will be collected as three independent samples centered around the same station. These reference sediment samples will be identified as RS-PH03-1A, -1B, and -1C for Station RS-PH03-1 and RS-PH03-2A, -2B, and 2C for Station RS-PH03-2. They will be analyzed for the same parameters as the ODMDS sediment samples, i.e., grain size distribution, TOC, metals, pesticides, PCBs, PAHs, and percent solids.

Field Methods and Procedures.

Field Equipment. Equipment shipped to the Ponce area in preparation for the ODMDS field work should be placed in containers that will protect the items from damage due to rough handling and wet weather conditions. To the extent practical, critical items will be duplicated so that failure of any one item will not stop the field work. Similarly, spare parts and replacement items will be included as appropriate to prevent avoidable down time. When practical, sampling equipment will be precleaned before shipment to the field if it can be ensured that integrity of such equipment will not be compromised during shipment and storage. All sample containers will be supplied by the laboratory and the integrity of all containers must be maintained during shipment to and from the Ponce area and while in storage awaiting use.

Decontamination and Clean Area Procedures. All equipment surfaces that potentially contact sample material will be decontaminated, e.g., stainless steel spoons and bowls. Such equipment will be cleaned using a water/Liqui-nox solution, followed by a tap or clean sea water rinse, isopropanol rinse, and air dried. If not immediately used after cleaning, decontaminated equipment will be wrapped in food-grade aluminum foil with the shiny side facing/contacting the equipment. Field personnel will wear gloves that are non-powdered,

talc- and phthalate-free, e.g., Nitrile, and will change them between sampling stations, when cleaning equipment, and when they become "dirty" or become ripped or torn.

After any welding has been finished, the deck area where the sampler is positioned and sampling handling occurs will be scrubbed with Liqui-nox solution or other appropriate non-contaminating cleaner to remove petroleum residues, loose debris, and any other materials that might contaminate sampled sediment. If, in the opinion of the field team leader, additional protective measures are needed polyethylene or polypropylene sheeting, e.g., visqueen, will be applied to the deck.

Hydraulic and petroleum-fueled equipment will be maintained in an area away from the vibracoring area and any drips or leaks of hydraulic fluids, fuels, or lubricants will be cleaned up immediately. To the extent practical, gasoline- and/or diesel-fueled equipment will be positioned downwind to minimize the potential for sample contamination.

Sample collection and handling. All samples will be collected using appropriately cleaned, non-contaminating equipment (e.g., constructed of stainless steel or coated with Teflon®) as required for the analyses of interest.

Triplicate samples from the ODMDS stations will be generated by dividing the exposed (upper) sediment surface in a box core grab sampler into three similar rectangular sections and removing sediment to a sufficient depth from each section to obtain the required one liter. Each replicate sample will be homogenized using an appropriately cleaned stainless steel spoon and bowl before it is containerized.

Reference samples may be collected by grab sampler such as a box corer or van Veen. Collected sediment will be placed in a container made of stainless steel or other material with a protective coating of Teflon® or similar material. Excess water will be decanted before homogenizing and containerizing the sample.

If sample collection problems are encountered, e.g., finding a sediment-free hard bottom or strong currents misaligning the sampler, either the on-site EPA representative will be consulted for guidance or the professional judgement of the field team leader will be used to determine the best course of action, e.g., accepting the sample as collected, relocating to another appropriate sampling location, or taking other corrective actions.

The homogenized sediment of each sample will be used to fill four 250 mL glass jars with Teflon®-lined lids for shipment to the laboratories. Two containers from each station will be shipped overnight on wet ice to Battelle for testing. The remaining two containers from each station will be stored on wet ice and sent to PPB to be archived. Samples will be shipped in ice chests secured with custody seals in a manner that will disclose if any ice chest were opened before delivery to the laboratory.

Table 5-1 is included to show the numbers and types of samples to be collected or generated and the tests that will be performed. Included on this table are some USEPA Region II-

specified, laboratory-generated quality control samples; other laboratory-generated quality control samples are described in the Chemical and Physical Evaluation QAP section.

Hydrographic Measurements

Collection of in situ water quality data, normally collected for 103 certification, is not required for this project.

Conductivity, temperature, and depth (CTD) data will be collected from the ODMDS area. Two CTD profile data sets will be collected every other day, one profile in the morning and one profile in the afternoon, while sampling operations are occurring in the ODMDS area. These data will be used to describe the physical structure of the water column and to support ADDAMS (STFATE) modeling. The specific locations where these data are acquired within the ODMDS are not important, as the hydrographic features are not expected to vary significantly within the site.

All field sheets will be provided to AFI and the Corps upon the completion of fieldwork.

Replicate Samples

Because triplicate samples will be collected for 5 of the ODMDS stations, no field duplicate samples will be collected for that area. As stated previously, both reference stations will be triplicated and each replicate sample will be collected from a separate deployment of the sampling device, unlike ODMDS stations, .

Table 5-1. Number and Type of Sediment Samples to Be Collected and Required Testing for Ponce ODMDS
Evaluation.

Sediment Matrix Only	Metals	Pesticides	PCB Congeners	PAHs	TOC	Grain Size
13 ODMDS Station in Singlet	13	13	13	13	13	13
5 ODMDS Stations in Triplicate	15	15	15	15	15	15
2 Reference Stations in Triplicate	6	6	6	6	6	6
Total	34	34	34	34	34	34

Note: Please see Table 1 of Section 3 of this Work/QA Plan for a listing quality control samples.

Section 2

6 Sample Custody

Samples should remain in the custody of the field team to ensure that they are properly preserved and that sample integrity is not compromised in any way. "Custody" implies that samples or the shipping containers in which they are placed remain in the physical or visual presence of the sample custodian (e.g., a field team member or laboratory personnel) or that the samples are placed in a locked or otherwise protected, restricted-access area (e.g., a locked vehicle or room).

Documentation of sample custody is an important aspect of this project to ensure that samples were secure and properly treated from the time of collection until arrival at each laboratory at which analyses will be performed. Water and sediment sample collection will be documented on field sampling sheets. Within 15 minutes of sample collection, all samples will be placed in laboratory provided containers and chilled with wet ice. Before sample custody is relinquished by the field team that collected them, a PPB Chain of Custody form will be filled out to document: project name, sample identification (which will correspond to sampling location), sample collection time and date, signature(s) of field team leader or his/her representative, sample preservation methods, and time and date the samples were relinquished, and the identity of person or transport service to whom the samples were relinquished. An example of the PPB Custody form is shown in Figure 6-1.

It is planned that two sample containers from each station will be sent by overnight delivery to Battelle for analysis and the remaining two containers will be shipped to PPB to be archived. Samples will be accompanied by signed chain of custody forms that are sealed in water-tight, plastic bags and taped to the inside surface of ice chest lids. Shipping receipts from the courier service will be retained to complement information provided on the chain of custody forms. Because it is impractical to expect courier service personnel to sign custody forms, the field team member will indicate the company name and approximate time of transfer on the custody form before sealing it in the shipping container. Shipping receipts will be retained as part of the project file to document sample transport.

Custody seals will be placed on each sample shipping container (i.e., ice chest) so that the opening of any container will be detected. These seals will be signed and dated by a field team member and placed on each ice chest in such a manner that the container cannot be opened without causing obvious damage to the seal. Condition of the custody seals should be documented upon their arrival at the laboratories.

ppb
ENVIRONMENTAL LABORATORIES, INC.
6821 SW Archer Road, Gainesville, FL 32608
(352) 377-2349 · FAX (352) 395-6639

CHAIN-OF-CUSTODY RECORD

LABTROL CLIENT No.	BATCH NO.	FOR LAB USE ONLY
		Subcontractor ____
		Shipping Method ____

CLIENT NAME

CLIENT PROJECT

SITE NAME & ADDRESS

SAMPLERS: *(Signature)*

LAB REPORT GOES TO (Client contact person):

NUMBER	DATE	TIME	COMP.	GRAB	STATION LOCATION / NUMBER	SAMPLE MATRIX	NUMBER OF CONTAINERS	IDENTIFY PARAMETERS DESIRED AND NO. OF CONTAINERS								Lab I.D. Number

PRESERVATION

CF	Chilled-Filtered
SF	Sulfuric-Filtered
NF	Nitric-Filtered
C	Chilled
S	Sulfuric
N	Nitric
B	Basic/NaOH
Z	Zinc
T	Thiosulfate
H	HCL
Ot	Other (see Remarks)

	Date / Time		Date / Time
Precleaned Containers Relinquished by: *(Signature)*		Received by: *(Signature)*	
Relinquished by: *(Signature)*		Received by: *(Signature)*	
Relinquished by: *(Signature)*		Received by: *(Signature)*	
Relinquished by: *(Signature)*		Received by: *(Signature)*	

Remarks and Observations

Matrix Types S Soil or Sediment WW Wastewater T Animal Tissue SW Surface Water SL Sludge or Solid Waste MW Marine Water GW Ground Water DW Drinking Water P Plant Tissue F Filter A Air Ot Other (See Remarks)

FIGURE 6-1. Example of PPB's Chain-of-Custody Form

Section 2

7 Calibration Procedures and Frequency

Instruments are calibrated with known standards to verify or adjust measurement devices so that an accurate and precise reading from an unknown can be obtained. Normally, instrument calibration occurs before measurement of an unknown and afterward measurement of the standards also occurs to confirm that the instrument continued to function properly within the established acceptance limits.

Instrument calibration and frequency of calibration are important elements of the SOPs for the field and laboratory members of the corporate team members working on this sediment evaluation project. Refer to the relevant SOPs in Section 2 or 3 regarding calibration procedures. Any deviations from or problems encountered during instrument calibration procedures will be documented and reported to the PPB Project Manager and Project QA Coordinator.

8 Analytical Procedures

A list of contaminants of concern has been developed. Table 3-1 presents information on each field test method that will be used for this evaluation. Analytical procedures performed by the testing laboratories are presented in the following sections of this Work/QA Plan.

Sediment Physical Analyses
Physical analyses will be conducted on each of the Ponce ODMDS sediment samples and the reference sediment composite. The tests to be performed are grain size analysis and percent solids. The Ponce ODMDS replicate samples will be tested for these physical parameters.

Sediment Chemical Analyses
Sediment chemical analyses (bulk sediment tests) will include metals, total organic carbon, pesticides, PCB congeners and PAHs. The 13 ODMDS sediment samples, 5 ODMDS stations in triplicate, and 2 ODMDS references in triplicate will be tested for all of these parameters.

9 Data Validation, Reduction, and Reporting

Data validation is a process that is used to deem data acceptable for project needs or to reject some or all data as unacceptable. Analytical data are compared to laboratory control limits for accuracy (bias) and precision; the performance of calibration standards, various other standards, blank samples, and reference materials; and QAPP data quality objectives (DQO'Ss) including target detection limits. Procedures to transform data to another basis for reporting (e.g., a dry or wet weight basis, total content of a group of compounds analyzed separately) are common laboratory calculations. The field team for Continental Shelf Associates and the laboratories at Battelle, Applied Marine Sciences, and Harding ESE have established, state-approved procedures for data validation, reduction, and reporting using the test methods specified for this evaluation. These organization-specific procedures for validating, reducing, and reporting will be followed by each corporate team member.

All laboratory reports presented to the PPB Project Manager will include laboratory QC data generated during analysis of project samples. Data qualifiers will be reported where they are appropriate. Laboratory reports should include QC information on all method blanks, matrix spikes, surrogate spikes, check standards, laboratory replicates, reference materials, and all other information appropriate for the test results being reported. The Project QA Coordinator will review all data with respect to: data quality objectives; consistency of related data from different tests; evaluation of test data from field-derived QC samples (e.g., triplicate samples); and impacts of missing, qualified, or unacceptable data relative to project needs. If needed, the Project QA Coordinator will work with Laboratory Project Managers and QA Officers to resolve any potential problems or data inconsistencies that are identified.

The Project QA Coordinator will provide a written memorandum to the PPB Project Manager discussing the acceptability of data for use in making project related technical decisions.

10 Internal Quality Control Checks

Each project team member will follow the procedures established for their organization that define the use of internal quality control samples and the procedures described for the assessment of the resulting data as stated in their state-approved and established SOPs. Use of field quality control samples will conform to the SOPs appearing in Appendix A.

11 Performance and System Audits

Performance and system audits use various techniques and procedures to monitor the capabilities and performance of field and laboratory systems for sample acquisition and testing.

For many projects with non-repetitive field activities, the one-time field operation may tend to be more complex and have more uncontrolled variables associated with it than the associated routine testing that occurs in the controlled setting of an environmental testing laboratory. Consequently, a field systems audit is planned for this sediment evaluation project. This audit will review the effectiveness with which the field team has implemented their SOPs for collecting, handling, labeling, and documenting sample and field data collection activities. This audit will be the first quality assurance report submitted as stated in Section 16.

No laboratory performance or systems audits are planned at this time.

Each laboratory is expected to continue participating in their routine performance evaluation testing programs. Any deficiencies that are identified that might adversely reflect on the data quality for this project should be brought to the attention of the PPB Project Manger.

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12 Facilities

During field operations, areas are established for the collection and handling of samples so that their integrity is not compromised and to minimize the possibility that they could contaminate the work area or subsequent samples. An SOP for establishing a clean working environment on sampling vessels is included elsewhere in Appendix A.

Normally, laboratories have established areas for sample reception, sample storage, sample preparation, testing, reagent storage, and data reduction and analysis. Having well defined work areas reduces potential for sample contamination and generally improves the quality of data produced. Each testing laboratory performing work on this sediment evaluation project has designed their work areas over a span of many years to best meet the needs of the tests they perform. Each laboratory has had successfully-concluded, on-site inspections/audits performed by their state or for the NELAC approval process.

13 Preventive Maintenance

Preventive maintenance is an essential part of both field work and laboratory testing for the successful and timely production of acceptable quality data. Continental Shelf Associates routinely tests and calibrates all field equipment during mobilization as well as providing back-up systems for each equipment type. For expensive laboratory instruments for which redundancy may not be an option, regularly scheduled maintenance and a large inventory of spare parts are more critical aspects of preventive maintenance planning.

The field team for Continental Shelf Associates and the laboratories at Battelle, Applied Marine Sciences, and Harding-ESE have established and state-approved procedures for preventive maintenance for the instrumentation and equipment that they will use on this sediment evaluation project. These established, organization-specific procedures will be followed by each of these corporate team members for the project tasks assigned to them.

14 Calculation of Field Data Quality Indicators

Although there are no control or warning limits associated with the field data for this project, data quality will be assured by adherence to approved SOPs.

Because multiple laboratories will be performing different tests on the same sediments for this project, precision values will be calculated using the statistic that is used by the laboratory reporting the data.

The field team for Continental Shelf Associates and the laboratories at Battelle, Applied Marine Sciences, and Harding-ESE have established and approved procedures for calculating data quality indicators for the instrumentation and equipment that they will use on this sediment evaluation project. These established, organization-specific procedures will be followed by each of these corporate team members for the project tasks assigned to them.

Percent completeness will be calculated, by constituent, as the ratio of the acceptable data reported divided by the number of data that were planned to be obtained.

Acceptability criteria for data quality for chemical testing of sediment is found in the appropriate subsections of Section 3 of this Work/QA Plan.

15 Corrective Actions

A corrective action will be initiated whenever a "nonconformance event" has been identified. A "nonconformance event" occurs when there is deviation from specified procedures or methods or anytime that the level of data quality may be compromised.

In the field, equipment malfunctions can be a common cause for having to initiate corrective actions. The field team member who first suspects or identifies a potential equipment problem should notify the field team leader immediately. If the cause of the problem is easily identified and can be corrected immediately, the field team leader has the authority to proceed with corrective actions and continue with field operations. For example, if worn equipment parts fail and are replaced from a store of spare parts, sampling might continue while replacement spare parts are shipped should the same failure reoccur. If a problem is encountered that is beyond the control of the field team leader and there is no specified guidance in the scope of work or QAPP, the field team leader should notify the project manager to negotiate an appropriate corrective action or alternative approach that is consistent with project goals. A reliable means of communication between the project manager and the field team should be identified before field operations begin. All problems and corrective actions should be documented on appropriate field sheets or notebooks. Field instrumentation also may require corrective actions similar to those occurring in the analytical laboratory. Corrective actions are addressed in the SOPs for Continental Shelf Associates.

Corrective actions for laboratory analyses or potential data quality problems can be required when test parameters exceed predetermined limits of acceptability. Such problems can be of a routine nature indicating, for example, the need for instrument servicing. However, if the nature of the problem is not routine and if it may require remedies that could compromise data quality, e.g., holding time exceedance, then the Laboratory Project Manager must contact the PPB Project Manager to discuss corrective actions and other options. Corrective actions are specified in the SOPs for each laboratory participating in this project.

Corrective actions for the analytical laboratory are presented in the appropriate subsections of Section 3 of the Work/QA Plan. These corrective actions include those provided in Appendix D of the "Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal", December 18, 1992.

16 Quality Assurance Reports to Management

Three quality assurance reports are planned.

The first QA report will be a field quality assurance audit report that will document all aspects of sediment and field data collection; sample handling, labeling, transport, and preservation; and documentation of sampling activities and information. After field work has been completed, this report will be submitted to the PPB Project Manager. It will include a description of field activities, all findings, and any corrective actions that should be implemented.

The second QA report will be submitted after field and laboratory data are reported to the PPB Project Manager. The Project QA Coordinator will evaluate all field documents and laboratory test data as described in Section 9 and produce a memorandum that will document the utility of the data for project needs.

The final QA report will be submitted discussing the quality of field-sampling activities and laboratory test results. This report will appear as a section of the final report.

17 References

The following references have been consulted in the preparation of this Work/QA Plan and may be cited in it.

Battelle, Quality Assurance Plan for Chemical Evaluation of Dredged Material, Revised October 24, 2001. (See Section 3 of this Work/QA Plan for project-specific information.)

USEPA, Region II and USACE, New York District. Draft dated December 1992 (undated updates attached). "Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal."

USEPA and USACE. April 1995. "QA/QC Guidance for Sampling and Analysis of Sediments, Water, and Tissues for Dredged Material Evaluations - Chemical Evaluations." EPA 823-B-95-001.

USEPA and USACE. February 1991. "Evaluation of Dredged Material Proposed for Ocean Disposal." EPA 503/8-91/001.

USEPA. December 1996. "Test Methods for Evaluating Solid Waste Physical/Chemical Methods." SW-846, Third Edition.

Plumb, R.H., Jr. 1981. "Procedure for Handling and Chemical Analysis of Sediment and Water Samples." Technical Report EPA/CE-81-1.

Appendix A -Standard Operating Procedures

The following procedures will be used during the field activities.

Section 2

SOP for Preparation and Cleaning Vessel Work Area

1.0 Introduction

The work area where sediment and water samples are removed from various sampling devices and initially processed should be properly selected, prepared, and cleaned. Standardized cleaning protocols help reduce the possibility that sampling equipment and utensils come into contact with analytes of interest, or constituents that would interfere with the analytes of interest.

Often, commercial "vessels of opportunity" are utilized for conducting surveys that require water and sediment sampling. Typically, the decks and potential work areas aboard these vessels have some level of contamination from fuel oil, hydraulic fluid, or other contaminants that could adversely affect sample integrity.

2.0 Procedures

2.1 Initiate review of sample types and parameters to determine if cleaning procedures outside of Standard Operating Procedure (SOP) are required or mandated.

2.2 Select work area in the immediate vicinity of the deployment/recovery equipment to facilitate ease of handling and to minimize sample movement.

2.3 To whatever extent possible, the work area should be large enough to store the coolers containing sample containers. If this is not possible, the current sample site containers and coolers should be cleaned and placed within the work area.

2.4 To whatever extent possible, the work area should be located out of the main flow of vessel crew traffic.

2.5 Avoid areas in proximity of fuel storage and handling, engine exhaust, or areas where people congregate to smoke cigarettes.

2.6 Choose a work area with adequate environmental protection as well as adequate ventilation.

2.7 Upon mobilization of deployment/recovery equipment, thoroughly clean work area.

- 2.7.1 Use fresh water hose at highest pressure to remove the largest and most obvious foreign material that may contain any potentially contaminated constituents.
- 2.7.2 Mix appropriate amount of a commercial grade cleanser containing a degreasing agent in appropriate size bucket with fresh water.
- 2.7.3 Scrub work area with cleanser and large hand brush, including deck and bulkheads that may come into contact with sample equipment or personnel.
- 2.7.4 Thoroughly rinse off cleanser with fresh water hose at highest pressure available.
- 2.7.5 Mix appropriate amount of a phosphate-free detergent such as Liqui-Nox, with fresh water.

2.7.6 Thoroughly scrub the work area again with the Liqui-Nox/fresh water mix.

2.7.7 Thoroughly rinse off cleanser with fresh water hose at highest pressure available.

2.7.8 This procedure should be completed at intervals determined by sampling protocol and conditions.

Section 2

CONTINENTAL SHELF ASSOCIATES, INC. STANDARD OPERATING PROCEDURES

USE, DEPLOYMENT, AND RETRIEVAL OF GRAB SAMPLERS FOR SEDIMENT COLLECTION

INTRODUCTION AND SCOPE

Sediments occur in freshwater and marine environments such as streams/rivers, ponds/lakes, canals, ditches, wetlands, lagoons, and estuaries.

The project objectives will define how sampling locations for sediments will be selected, e.g.,

- Sediment samples may be collected as an adjunct to surface water samples;
- A series of sediment samples for compositing to determine water or sediment quality in a system;
- Sediment samples above and below an outfall to document degradation due to a point source discharge; or
- Sediment samples if stressed shore vegetation or visible surface water contamination is evident.

Decisions related to the selection of sampling locations will not be discussed in this document.

If water samples are to be collected from the same stations as sediment, water samples should be collected first to avoid resuspending sediment in the water column that may contaminate the water and bias the analysis. Collect, preserve, and containerize surface water samples prior to collecting sediment samples.

Scope and Application

The document describes the methods for using, deploying, and retrieving grabs samplers for collecting sediment samples from a survey vessel. The following methods are applicable for sediment collection for physical, chemical, and benthic invertebrate sampling and toxicological testing.

Sampler Selection and General Operation

The decision on the specific type of grab sampler to use to collect sediment samples will depend on a number of considerations:

- the bottom conditions (e.g., sediment type);
- volume of sample (or length of core) required;
- station depth;
- the type of sample analysis required;

- the sampling platform, and
- and sampling conditions or criteria, among others.

If undisturbed and uncontaminated sediment samples are required, some samplers are not suitable. If the bottom conditions or if the sediment type is unknown or may vary widely, two or more different types of grab samplers may be used during a survey effort. Regardless of the type selected, one or more backup grab samplers should be available on a survey.

The size of the grab sampler also will need to be considered to balance cost or workability with ease of use or practicability. For example, if only small volumes of sediment are required, small samplers may be used. However, small light-weight samplers may not work in deep stations or where strong currents occur that may cause the sampler to tumble rather than fall to the bottom upright. Small samplers also may not be heavy enough to penetrate the sediment surface in harder substrates. Larger samplers allow larger samples to be taken but may penetrate too deep into the sediment unless the drop speed can be controlled. Some samplers may also create a bow wave that can displace finer sediment from the surface. Larger samplers are also more expensive to ship and difficult to handle. The specific analyses to be done of the sediment will also dictate the type of sampler to be used and other features.

The sampler type or design to select is also an important consideration. Muddy or silty sediments require a different type of sampler compared to coarser sediments. The following types of grab samplers may be used for sediment sampling in estuarine and marine environments:

- Petersen grab
- Smith-McIntyre grab
- Gray-Ohara box core
- Van Veen grab
- Young modified Van Veen grab
- Ponar grab
- Petite Ponar grab

Equipment and Materials

- Grab sampler
- Sampler stand
- Large buckets
- Broad and deep rectangular basins
- Coring tubes
- Sediment splitter
- Hose or tubing
- Seawater hose
- Sieving trays or buckets

Section 2

Procedure

When working on a survey vessel, close coordination between the bridge crew and the deck crew is needed. If possible, a member of the survey crew should act as a navigator posted on the bridge and in communication with the deck crew via radio. When working with larger sediment samplers (e.g., a Van Veen grab sampler or Gray-O'Hara box corer), a deck crew composed of two grab operators and a winch operator is recommended. A third member to act as the data recorder and sample processor also is recommended.

Prior to sampling, the survey team should provide the survey navigator with the specific coordinates and the sequence of stations to sample. Preferably, the stations should be pre-plotted using navigation software that can display location of the sampling sites and the boat position. A circle defining the acceptable distance (e.g., 30 m diameter or 15 m radius) from the planned station location that a sample can be collected should be depicted on the monitor. The Chief Scientist should discuss the procedure for approaching the sampling station and notifying the deck crew to deploy sampler.

When collecting environmental samples that will be analyzed for low levels of analytes, the work area should be free from potential contaminants. Clean the work area on the survey vessel as needed.

Wash the sampler prior to use as required to according to SOP. Position the sampler on its stand and arrange the sampler and other equipment as necessary to facilitate working with the sampler, collecting the sediment samples, and redeploying the sampler.

If necessary, direct the winch operator to raise the sampler to allow cocking the sampler open. Secure top doors of the sampler, cock the sampler open, and set the trigger mechanism.

Upon receiving the signal that the sampler position is on target, the winch operator should release the winch drum brake and allow the sampler to fall to the bottom in a controlled manner. Depending on the sampler, water depth, boat movement, and currents, allowing the sampler to completely free fall may not be recommended as the sampler may tumble or corkscrew on its way to the bottom.

Some grab samplers are triggered shut by messenger or when it hits the bottom while other samplers close when the sampler is winched up. Direct the winch operator to bring the sampler back to the surface.

Direct the winch operator to quickly raise the sampler out of the water and set the sampler on the stand to the deck to avoid sample loss from water escaping from the bottom of the sampler. No sampler seals completely so some sample loss may be expected.

After the grab sampler is lowered to the deck and safely secured on to its stand, open the top doors of the sampler and determine if the sample is acceptable. The general criteria for accepting a sediment grab (sample) are as follows:

- the sampler is not overfilled so that the sediment surface is touching the top of the sampler;
- overlying water is present (indicates minimal leakage). This overlying water will be removed prior to processing and storage by siphoning, not decanting;
- the overlying water is clear or not excessively turbid;
- the sediment-water interface is intact and relatively flat; there is no sign of channeling or sample washout;
- the desired depth of penetration has been achieved, e.g., this depth is 15 cm for the Smith-McIntyre grab; and
- there is no evidence of sediment loss (incomplete closure of the sampler, penetration at an angle, or tilting upon retrieval).

If necessary determine the depth of penetration by either measuring the distance from the top of the sampler to the sediment surface or inserting a ruler into the sediment to the bottom of the sampler.

Near bottom currents and other conditions may confound sediment sampling. If the sampler overpenetrates, remove weights or reduce the drop speed until an acceptable sample is collected.

If the sample is acceptable, signal the bridge crew that a successful drop was made and sediment was collected. The vessel can then be driven to the next station indicated on the sampling schedule.

If overlying water is present, carefully siphon the water off the sediment surface onto the deck or into a bucket if any samples are required or organisms in the overlying water are present.

Insert sample splitter plate and sediment core tubes as required. Remove sediment core tubes from the sampler and process according to SOP.

If the sample is for macroinfauna, remove cores (e.g., for sediment chemistry, grain size, and total organic carbon) if needed, then remove the remaining sediment into large basins. While keeping the collecting basin below the sampler, run a gently stream of water through the inside of the sampler to wash remaining sediment into the collecting basin.

Clean and decontaminate the sampler as required per SOP. Reset the sampler and redeploy as needed.

Surface area of sampler
Penetration depth

Petersen
Smith-McIntyre
Gray-Ohara

Section 2

Van Veen
Young modified Van Veen
Ponar
Petite Ponar

Safety

The deck crew should use hard hats especially when working during rough seas when a wildly swinging grab sampler may cause injury. Safety shoes should be worn to protect feet in case the grab or a weights fall on the deck. Life vests or PFDs also should be worn especially if the deck does not have a gunwale or during rough seas. When working in cold environments where the water temperature is less than 75°C insulated survival gear (e.g. Mustang suits) should be worn because of hypothermia hazards in case one falls into the water.

Operating grab samplers require safety precautions to avoid accidents and injury to the operators. Use of checklists is recommended. The sampler, winch cable, and winch should be inspected for defects prior to use. The grab sampler operators should take care to avoid getting any body parts caught in the sampler especially the jaws of a sampler pinching or severing fingers.

Dredges or Grab Samplers

The three main types of devices used in freshwater are the Ekman, Peterson and Ponar. Heavier oceanographic dredges are used in marine and estuarine waters. The Peterson and Ponar dredges are suitable for hard or rocky substrates or deep water bodies.

The Peterson and Ponar are virtually the same, except that the Ponar has been adapted with a top screen and side plates to prevent sample loss upon ascent. For this reason, the Ponar is the dredge of choice for rocky substrates. These dredges are heavy enough to use in streams with fast currents.

- Open the jaws and place the cross bar into the proper notch.
- Lower the dredge to the bottom, making sure it settles flat.
- When tension is removed from the line, the cross bar will drop, enabling the dredge to close as the line is pulled upward during retrieval.
- Pull the sampler to the surface. Check to make sure the jaws are fully closed and that no sample was lost while lifting the dredge.
- Carefully open the jaws, remove the sample with a clean, non-reactive utensil and transfer the sample into the appropriate sample container(s), label, preserve to 4°C with wet ice and complete field notes.

The Ekman is designed for sampling soft substrates (e.g., sand, silt or mud) in areas with little current.

- Open the spring-loaded jaws and attach the chains to the pegs at the top of the sampler.
- Lower the dredge to the bottom, making sure it settles flat.
- Holding the line taut, send down the messenger to close the jaws of the dredge.
- Pull the sampler to the surface. Check to make sure the jaws are fully closed and that no sample was lost while lifting the dredge.
- Carefully open the jaws, remove the sample with a clean, non-reactive utensil and transfer the sample into the appropriate sample container(s).
- Label, preserve to 4°C with wet ice and complete field notes.

3.4 Equipment Decontamination

For most sampling applications, site water rinse of equipment in between stations is normally sufficient (PSEP, 1997a). However, if one is sampling many stations, including some that could be heavily contaminated, a site water rinse might not be sufficient to minimize cross-contamination of samples among stations. In these cases, it might be necessary to decontaminate all sampling materials in between stations. This would include the sampling device, scoop, spatula, mixing bowls, and any other utensils that come in contact with sediment samples. An approach recommended by ASTM (2000a) for field samples of unknown composition includes: (1) soap and water wash, (2) distilled water rinse, (3) acetone or ethanol rinse, and (4) site water rinse. In general, organic solvents such as methylene chloride should not be used due to the associated health and safety risks. Waste solvents should be collected in labeled hazardous waste containers. If sediment can be collected from the interior of the sampling device, and away from potentially contaminated surfaces of the sampler, it might be adequate to rinse with site water between stations.

If metals or other inorganic compounds are specifically of concern, sampling and handling equipment should be suspended over a tub and rinsed from the top down with 10 percent nitric acid using a pump or squirt bottle (USEPA, 1993; ASTM, 2000a). If organic compounds are a specific concern, sampling equipment can be decontaminated using acetone followed by a site water rinse. Wash water from decontamination should be collected and disposed of properly.

Section 2

SOP for
SBE 19 SEACAT Multiparameter Water Quality Instrument

1.0 INTRODUCTION

The SBE 19 SEACAT Profiler is designed to measure electrical conductivity, temperature, and pressure in marine or fresh water environments. The SEACAT Profiler will operate in two modes, profiling and moored. Profiling mode is designed for applications where vertical profiles of the measured parameters are required. In profiling mode, the sample rate can be varied from twice a second to once every 4 minutes in half-second increments. Moored mode provides a means of acquiring time series measurements at sample rates of once every 15 seconds to once every 8 hours, adjustable in one-second increments.

2.0 SPECIFICATIONS

Measurement Range:
Temperature -5 to +35 °C
Conductivity 0 to 7 S/m (0 to 70 mmho/cm)
Pressure
Strain Gauge Sensor 100, 150, 300, 500, 1000, 1500, 3000, 5000, 10000, or 15000 psia
Digiquartz Sensor 100, 200, 300, 400, 1000, 2000, 3000, 6000, 10000, or 15000 psia

Accuracy:
Temperature 0.01 °C/6 months
Conductivity 0.001 S/m/month
Pressure
Strain Gauge Sensor 0.25% of full scale range (100 - 1500 psia); 0.15% of full scale range (3000 - 15000 psia)
Digiquartz Sensor 0.02% of full scale range

Resolution:
Temperature 0.001 °C
Conductivity 0.0001 S/m
Pressure
Strain Gauge Sensor 0.015% of full scale range (100 - 15000 psia)
Digiquartz Sensor 0.001%

Sensor Calibration:
Temperature -1 to +31 °C (measurements outside this range may be at slightly reduced accuracy due to extrapolation errors)
Conductivity 0 to 7 S/m. Physical calibration over the range 1.4 - 6 S/m. Measurements outside this range may be at slightly reduced accuracy due to extrapolation errors.
Pressure 0 to full scale in 20% steps

Counter time-base:
Quartz TCXO, ±2 ppm per year aging; ±2 ppm vs. temperature (-5 to +30 °C).

Memory:
CMOS static RAM, 1024K or (optional) 2, 4, or 8 Mbyte; battery-backed for minimum 3 years data retention.

Real-time clock:
Watch-crystal type 32,768 Hz; battery-backed for minimum of 1 year operation irrespective of condition of main battery. Corrected for drift and aging by comparison to SEACAT counter time-base.

Batteries:
9 alkaline 'D'-cells provide 8 amp-hours at 20 °C (5 amp-hours at 0 °C) and 3 year data retention reserve.

Materials:
600 Meter Pressure Case, acetal copolymer (plastic)
3400 Meter Pressure Case, 6061-T6 anodized aluminum
6800 Meter Pressure Case, 7075-T6 anodized aluminum
7000 Meter Pressure Case, 3AL-2.5V titanium
10500 Meter Pressure Case, 6AL-4V titanium

3.0 CORROSION PRECAUTIONS

On aluminum-housed units, there is a large zinc anode screwed into the battery end-cap, and a second on the conductivity cell guard is also used on plastic versions. These anodes should be checked from time to time to see that they are securely fastened, and have not been eaten away. All the stainless steel screws that are exposed to salt water have been generously lubricated with NEVER SEEZ Blue Moly (available through marine hardware stores). After a deployment, it is wise to remove these screws and re-lubricate them with a similar compound. This compound is electrically conductive, so care should be used not to get it on circuit boards. SEACAT Profiler should be rinsed with fresh water after use and prior to storage.

4.0 CONDUCTIVITY CELL STORAGE

When possible, keep the conductivity cell in distilled water during periods of non-use by looping a length of water-filled Tygon tubing end to end. If it is not practical to leave the conductivity cell stored with distilled water in it (due to the danger of freezing, for example) merely rinse the cell with distilled water after each use (to remove salt water), then blow lightly through the cell to insure that no water is trapped inside. This is easily done using the Tygon tubes supplied. Loop the tubing end to end of the cell to keep out airborne contaminants. If the cell has been stored dry, wet with a 1% solution of Triton X100 (or

other non-ionic detergent) before use. Triton X100 may be obtained through a medical supply distributor.

5.0 SENSOR CALIBRATION

Sea-Bird sensors are calibrated by subjecting them to known physical conditions, and measuring the sensor responses. Then coefficients are computed which may be used with appropriate algorithms to obtain engineering units. The conductivity and temperature sensors on SEACAT Profiler are supplied fully calibrated, with coefficients as printed on their respective Calibration Certificates. The SEACAT Profiler is returned to Sea-Bird for calibration. Sea-Bird can supply software for coefficient generation. Because temperature is varied to yield a wide range of conductivities from baths of just two salinities, performance of a conductivity calibration on SEACAT Profiler will always yield a temperature calibration as well.

CONTINENTAL SHELF ASSOCIATES, INC. STANDARD OPERATING PROCEDURES

CLEANING OF SEDIMENT SAMPLING AND COLLECTION EQUIPMENT

At a minimum, these procedures apply to all sediment sampling activities where sediment will be analyzed for chemical parameters. Equipment for collecting sediment samples for physical or biological measurements (e.g., grain size distribution or macroinfauna) may be cleaned with soap and water prior to use and with seawater between stations.

All equipment used for collecting and processing sediment chemistry samples will be precleaned prior to sampling operations. Sampling equipment will include the grab sampler, Teflon® and acrylic core tubes, Teflon® and acrylic (e.g., 2-cm) rings, plungers (rubber stoppers with wooden handles for extruding sediment from coring tubes), mixing bowls, utensils (stainless-steel or plastic spoons, scoops, or spatulas), and sample containers.

The grab sampler will be cleaned with phosphate-free soap (e.g., 10% solutions of Liquinox or Alconox), rinsed with seawater, and then rinsed with pesticide-grade isopropanol, acetone, or hexane prior to sampling. Between stations, the sampler will be cleaned with seawater. The plungers used to extract sediment from coring tubes will be washed with seawater between stations as well.

Sample containers need to be treated according to the cleaning procedure if they are not commercially precleaned. Containers need not be cleaned if they are shipped in packs with intact factory seals and contain certified containers.

All equipment used for the collection and processing of sediment chemistry samples will be precleaned prior to every use and between stations. The cleaning procedure for Teflon® core tubes, 2-cm rings, mixing bowls, utensils, and sample containers is as follows:

Initial wash - conducted prior to sampling operations; wash equipment with phosphate-free soap, and scrub with a nylon brush, and rinse with clean freshwater or seawater;

Seawater rinse - after each usage of the equipment, rinse with clean seawater, removing all traces of sediment;

Wash - wash equipment with phosphate-free soap, and scrub with a nylon brush, and rinse with clean freshwater or seawater;

Acid wash - soak all non-metallic equipment thoroughly in a plastic bucket containing a 10%-20% solution of nitric acid (HNO_3);

Organic solvent wash - wash equipment in a stainless steel bucket containing organic solvent (i.e., acetone or hexane) to remove any organic substance (this step may skipped if samples are not analyzed for low level hydrocarbon concentrations); and,

DI water rinse - rinse equipment thoroughly with deionized water.

Acrylic core tubes and 2-cm rings used for samples to be used for grain size or trace metal analysis are washed also with phosphate-free soap and clean seawater, scrubbed with a nylon brush, and rinsed with deionized water.

Section 2

CONTINENTAL SHELF ASSOCIATES, INC. STANDARD OPERATING PROCEDURES

SEDIMENT SAMPLING AND CORE PROCESSING

These procedures apply to collection and processing of subsamples from sediment cores removed from grab samplers. These procedures assume that all equipment used is cleaned according to SOP, and commercially precleaned sample containers are used. The grab sampler is also assumed to be have been deployed and retrieved properly and sample deemed acceptable.

Equipment Required:

Teflon® and acrylic core tubes;
Teflon® and acrylic (e.g., 2-cm) rings;
plungers;
mixing bowls;
utensils (stainless-steel or plastic spoons, scoops, or spatulas), and
sample containers.

This procedure is best done with two persons involved. Subsamples may be collected using acrylic and Teflon® coring tubes of different diameters. The specific diameters and ring sizes will depend on the volume of sediment to be collected. Depending on the size of the grab sampler and needs of the project, a single or multiple cores may be taken from a grab sample. Cores are inserted by hand into the sediment in the grab sampler. After all the required coring tubes have been inserted into the sediment, the inserted cores are carefully removed by hand, capping the bottom of the coring tube with one hand and pulling the core tube out with the other hand.

The following procedure is used to collect the top 2 cm of sediment from a coring tube:

Decant the clear (i.e., sediment free) surface water overlying the subsample. A siphon tube should be used if keeping the upper sediment undisturbed.

If required by the project, pour any fluid, flocculent layer into a precleaned 1-L glass mixing bowl or appropriate container.

Use a rubber plunger to exude the sediment from the coring tubes. Keep the coring tube vertically oriented to prevent mixing the top sediment layer with underlying sediment.

Insert the plunger up into the bottom of the coring tube while holding a precleaned 2-cm high ring (of the same diameter as the tube) firmly in place on the top of the coring tube.

Push the plunger up, until the upper ring fills with sediment. Quickly draw the ring laterally toward the sample container or mixing bowl in one smooth motion, thereby cutting off the portion of sediment in the ring into the collecting container.

If the upper 2 cm of sediment is all that is required, the remaining sediment may be pushed out of the coring tube with the plunger. If further subsamples are required (i.e., deeper levels of sediment), the same technique can be used to bring the deeper sediments up to the top of the core and a subsample collected using another clean 2-cm ring.

The same technique may be used for collecting subsamples from cores of different diameters. Thicker layers of sediment may be sampled using rings of larger dimensions.

After using the core sampling equipment, they are rinsed in seawater and cleaned according to required protocols before being reused.

NOTES:

The use of wide-mouth sample containers is recommended if the sample is to be collected directly into the container. If narrow-mouth containers are used, it will be difficult to fill with sediment from coring tubes of larger diameters. If a sample is collected in a mixing bowl, filling narrow mouth sample containers can be done using a stainless-steel spoon, scoop, or spatula.

Section 3

Ponce ODMDS Sediment
Chemical and Physical Testing Quality Assurance Plan

Introduction

Section 3 presents the laboratory-specific project plan for the sediment evaluation of the Ponce Ocean Dredged Material Disposal Site (ODMDS).

Section 3

QUALITY ASSURANCE PLAN

for

Chemical Evaluation of Dredged Material
from
Ponce Ocean Dredged Material Disposal Site

Prepared for:

PPB Environmental Laboratory, Inc.
Gainesville, Florida

Revision No. 1

December 18, 2002

Battelle Project Manager: _____

Date: _____

Battelle

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Sequim, WA 98382
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Section 3

QUALITY ASSURANCE PLAN

Project Name:	Evaluation of Dredged Material Proposed for Ocean Disposal – Ponce ODMDS
Client:	PPB Environmental Laboratory, Inc.
Project Manager:	Carole Peven McCarthy
Date:	December 18, 2002

Section 3

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Section 3

INTRODUCTION/SCOPE OF WORK

Battelle is providing analytical support to PPB Laboratories, Inc., including sediment physical analysis, and trace organic and metals analytical chemistry services in support of evaluation studies of dredged material from Ponce Harbor, Puerto Rico, proposed for ocean disposal. The testing is conducted according to a guidance manual developed by the U.S. Army Corps of Engineers (USACE) and the U.S. Environmental Protection Agency (EPA), *Evaluation of Dredged Material Proposed for Ocean Disposal (Testing Manual)*, commonly referred to as the "Green Book," as well as the regional manual developed by the USACE New York District (NYD) and EPA Region II, *Guidance for Performing Tests on Dredged Material to be Disposed of in Ocean Waters* (USACE-NYD/EPA Region 2 1992), hereinafter referred to as the "Regional Testing Manual" or "RTM." Under these guidelines, sediment samples are analyzed for chemical contaminants of concern.

Analytical services that will be provided by Battelle and our subcontractors include the following analyses: sediment grain-size, total organic carbon, and percent solids, and measurements of trace levels of selected organic and trace metal contaminants in sediment.

Analytical Activities

Table 1 provides a breakdown, on a sample matrix basis, of the expected numbers of samples, the types of analyses, the numbers of analyses per given analysis type, and the type, frequency, and numbers of quality control samples that will be analyzed. Samples will be extracted in two analytical batches of 20 or fewer field samples. Each batch will include the suite of QC samples, including one SRM (not for TBT – none available), one MS, one LCS (organics only), one blank, and at least one set of triplicate samples (measure of precision).

Table 1. Sample Breakdown - Analytical Chemistry

Matrix	Sample Count/QC Sample Analyses
Sediment Samples	13 ODMDS stations in singlet; Analyses: PCB/Pest, PAH, Metals 5 ODMDS stations in triplicate (total of 15) 2 Reference stations in triplicate (total of 6) 2 SRM 2 MS 2 LCS (organics only - internal check) 2 Procedural Blank

Sample Custody, Receipt, Splitting, and Shipping

Chain-of-Custody Records

Chain-of-custody (COC) records are the administrative records associated with the physical possession and/or storage history of each individual sample from the purchase and preparation of each sample container and sampling apparatus to the final analytical result and sample disposal. Battelle SOPs 6-010 and MSL-A-002 define field and laboratory COC procedures. Sample custody will be documented throughout the life of the sample. Samples should not be left unattended unless properly secured.

Each laboratory (including the contractor responsible for field sampling) must have a formal documented system designed to provide sufficient information to reconstruct the history of each sample, including preparation of sampling containers, sample collection and shipment, receipt, distribution, analysis, storage or disposal, and data reporting within the laboratory. Laboratory documentation must provide a record of custody for each sample (versus a sample batch) throughout processing, analysis, and disposal.

Samples are considered to be in a person's custody if

- The samples are in a person's actual possession;
- The samples are in a person's view after being in that person's possession;

- The samples were in a person's possession and then were locked or sealed up to prevent tampering; or,
- The samples are in a secure area.

The COC record will also be used as a record of the samples collected and analyses requested. Occasionally, multiple coolers will be sent in one shipment to the laboratory. Each cooler should contain a separate COC record for the samples in that cooler.

Sample Receipt

Immediately upon receipt by a laboratory, the condition of samples must be assessed and documented. The contents of the shipping container must be checked against the information on the COC form for anomalies. If any discrepancies are noted, or if laboratory acceptance criteria or project-specific criteria are not met, the laboratory must contact the shipping laboratory. The discrepancy, its resolution, and the identity of the person contacted will be documented in the project file. The following conditions may cause sample data to be unusable and must be communicated to the project manager:

- The integrity of the samples is compromised (*e.g.,* leaks, cracks, grossly contaminated container exteriors or shipping cooler interiors, obvious odors, etc.)
- The identity of the container cannot be verified
- The proper preservation of the container cannot be established
- Incomplete sample custody forms (*e.g.,* the sample collector is not documented or the custody forms are not signed and dated by the person who relinquished the samples)
- The sample collector did not relinquish the samples
- Required sample temperatures were not maintained during transport.

A unique sample identifier must be assigned to each sample container received at the laboratory.

Sample Handling

Table 2 provides information on sample preservation, containers, and holding times. The sample holding times presented are appropriate for the samples of interest, however, the reporting deadlines will drive the extractions for those samples with holding times >30 days. Schedule information is provided in Section 3.0. Holding times are calculated from the time of sample collection.

Table 2. Sample Holding Times, Container Types, and Preservation

Matrix	Preservation	Container Type	Holding Time (from Green Book and RTM)
Sediment	4°C	Organics: Glass with Teflon-lined lid	14 days from collection at 4°C; 1 year frozen; 40 days after extraction
		Metals: Pre-tared, acid-washed, plastic Spex jar	Hg 28 days at 4°C; other metals 6 months at 4°C; all metals 1 year frozen
		Physical Parameters/TOC: Glass with Teflon-lined lid	21 days from collection at 4°C

Field samples will be held for one year from sample collection; sample extracts and digestates will be held for one month. Disposal records for field samples and sample extracts will be sufficient to provide tracking from collection, through laboratory receipt, to sample disposal.

Section 3

Sample Splitting and Distribution

Sediment – Organics. A minimum of 50 g of each sediment composite sample will be subsampled for organics analysis. A duplicate/triplicate sample must be assigned for each batch of 20 samples, and an additional 150 g of sediment included for these analyses. Samples designated for organics analysis will be shipped cold (approximately 4°C with ice or ice substitute) to Battelle's laboratories in Duxbury, MA, to the following address/contact:

Battelle
397 Washington St.
Duxbury, MA 02332
(781) 934-0571
Attn: Carole Peven McCarthy

Sediment – Metals. A minimum of 10 g of each sediment composite sample will be subsampled for metals analysis. A duplicate/triplicate sample must be assigned for each batch of 20 samples, and an additional 30 g of sediment included for these analyses. Samples designated for metals analysis will be shipped cold (approximately 4°C with ice or ice substitute) to Battelle's laboratories in Sequim, WA, to the following address/contact:

Battelle
1529 West Sequim Bay Road
Sequim, WA 98382
(360) 683-4151
Attn: Jill Brandenberger

Sediment – Physical Parameters and Total Organic Carbon (TOC). Approximately 100 g of each individual sediment sample homogenates and 100 g of each of the reference sediment. A duplicate/triplicate sample must be assigned for each batch of 20 samples, and an additional 200 g of sediment included for these analyses. Approximately 600 g of each sediment *composite* will be provided for TOC, grain size, and the associated set of duplicate/triplicate QC samples. Additionally, percent moisture will be determined for each sediment core and composite. (When two or more sediment composites are tested concurrently, only 200 g of the additional samples are required, because replicate analyses are only required on one sample per batch). Samples designated for analysis of physical parameters and TOC will be shipped cold (approximately 4°C with ice or ice substitute) to the following address/contact:

Applied Marine Sciences, Inc.
502 N. Highway 3, Suite B
League City, Texas 77573
(281) 554-7272
Attn: Kenneth S. Davis

An additional 500-g sample of each composite sediment and each individual core sediment (if quantities are available) will be subsampled for archival. Sediments will be archived for 1-year from sample collection.

Sample Preparation

Sediment samples are analyzed for the lists of target analytes specified in the Regional Guidance Manual. Target analytes and Battelle's associated MDLs are presented in Table 3. These MDLs are based on annual MDL studies performed at Battelle following protocols described in the Federal Register (40 CFR, Ch. 1, Part 136, Appendix B, 1990). QC sample requirements for each analytical parameter were presented in Table 1 of this document. Data quality objectives for QC samples are discussed in Section 5 of this QAP.

Table 3. Target Analytes and Associated Detection Limits

Target Constituent	Matrix Sediment
Polycyclic Aromatic Hydrocarbons	*ng/g dry wt.*
Naphthalene	0.1
Biphenyl	0.05
Acenaphthylene	0.04
Acenaphthene	0.04
Fluorene	0.04
Phenanthrene	0.05
Anthracene	0.03
Fluoranthene	0.04
Pyrene	0.04
Benz(a)anthracene	0.05
Chrysene	0.04
Benzo(b)fluoranthene	0.04
Benzo(k)fluoranthene	0.03
Benzo(a)pyrene	0.1
Indeno(1,2,3-c,d)pyrene	0.08
Dibenz(a,h)anthracene	0.04
Benzo(g,h,i)perylene	0.05
Polychlorinated Biphenyls	*ng/g dry wt.*
Cl2(08)	0.1
Cl3(18)	0.01
Cl3(28)	0.01
Cl4(44)	0.02
Cl4(49)	0.01
Cl4(52)	0.01
Cl4(66)	0.02
Cl5(87)	0.01
Cl5(101)	0.01
Cl5(105)	0.02
Cl5(118)	0.02
Cl6(128)	0.02
Cl6(138)	0.02
Cl6(153)	0.02
Cl7(170)	0.02
Cl7(180)	0.02
Cl7(183)	0.02
Cl7(184)	0.02
Cl7(187)	0.02
Cl8(195)	0.02
Cl9(206)	0.02
Cl10(209)	0.02
Chlorinated Pesticides	*ng/g dry wt.*
2,4-DDD	0.01
2,4-DDE	0.07
2,4-DDT	0.08
4,4-DDD	0.01

Target Constituent	Matrix Sediment
4,4-DDE	0.02
4,4-DDT	0.02
a-Chlordane	0.01
Aldrin	0.01
Dieldrin	0.03
Endosulfan I	0.03
Endosulfan II	0.01
Endosulfan sulfate	0.01
Heptachlor	0.02
Heptachlor epoxide	0.02
Trans-nonachlor	0.01
Organotin	*ng/g dry wt.*
Monobutyltin	1.0347 ng/g
Dibutyltin	0.7191 ng/g
Tributyltin	0.5929 ng/g
Tetrabutyltin	0.5929 ng/g

Target Constituent	Matrix Sediment
Inorganics	*µg/g dry*[(a)]
Aluminum	0.5
Arsenic	0.05
Cadmium	0.01
Chromium	0.04
Copper	0.02
Iron	5
Lead	0.01
Mercury	0.002
Silver	0.03
Selenium	0.08
Zinc	0.1

(a) Sediment MDLs determined from analysis of 7-replicate, spiked water, MDL study, and calculated for sediments using representative dry sample mass and instrument dilution factor.

Sediment – Organics. Sediment samples are extracted for chlorinated pesticides, PCBs, and PAHs, by shaker table techniques as described in Battelle SOP 5-192. Prior to extraction the samples are spiked with surrogate internal standards (naphthalene-d_8, phenanthrene-d_{10}, chrysene-d_{12}, Cl_2(14), Cl_3(34), Cl_5(104), Cl_5(112)). After extraction and alumina column cleanup, the extracts are fractionated by HPLC/GPC (Battelle SOP 5-191), and the resulting extract concentrated to 1 mL using Turbo-Vap nitrogen concentration units or K-D, and nitrogen blow down techniques. The samples are then transferred to 4-mL vials (with DCM rinse) and activated copper added to the vials; samples sit on copper for a minimum of 1-h. The RIS (acenaphthene-d_{10}, fluorene-d_{10}, benzo[a]pyrene-d_{12}, Cl_5(96), Cl_5(103), Cl_6(166)) is added and the PIV adjusted to 1 mL. The extract is split qualitatively, with one half submitted for analysis of PAHs by gas chromatography with mass spectrometry (GC/MS), and the other half solvent-exchanged into hexane and submitted for pesticide and PCB analysis by GC/ECD.

Sediment – Butyltins. Sediments will be processed for butyltins following procedures detailed in Battelle SOP 5-196, *Measurement of Butyltin Species in Tissues and Sediment/Soil.* Briefly, an aliquot of sediment will be

spiked with surrogate internal standards to monitor laboratory efficiency, and extracted with hexane and the chelating agent tropolone. Following extraction, the cationic butyltin compounds will be converted to nonpolar *n*-hexyl derivatives with commercially available *n*-hexylmagnesium bromide via a Grignard reaction. The extract will be cleaned through a Florisil gel liquid chromatography column. The butyltins will be analyzed by GC/FPD using a tin-specific photometric filter.

Sediment – Metals. Sediment samples are analyzed for eleven metals: Al, Ag, As, Cd, Cr, Cu, Fe, Hg, Pb, Se, and Zn. To prepare the sediments for analysis, they are first freeze-dried, then blended in a SPEX mixer-mill and ground in a ceramic ball mill. Aliquots of dried, homogeneous sample are digested using aqua regia (nitric and hydrochloric acids) according to Battelle SOP MSL-I-006, *Mixed Acid Sediment Digestion,* and the digestates submitted for analysis. As, Cd, Cr, Cu, Fe, Pb, Ag, Se, and Zn are analyzed by ICP/MS according to Battelle SOP MSL-I-022, *Determination of Elements in Aqueous and Digestate Samples by ICP/MS.* The sediment analysis procedure is based on two methods modified and adapted for analysis of solid sample digestates, EPA Method 1638 *Determination of Trace Elements in Ambient Waters by Inductively Coupled Plasma-Mass Spectrometry* (EPA 1996) and EPA Method 1640 *Determination of Trace Elements in Water by Preconcentration and Inductively Coupled Plasma-Mass Spectrometry* (EPA 1997). Hg is analyzed using cold-vapor atomic absorption spectroscopy (CVAA) according to Battelle SOP MSL-I-016 *Total Mercury in Tissues and Sediments by Cold Vapor Atomic Absorption*, which is based on EPA Method 245.5 *Determination of Mercury in Sediments by Cold Vapor Atomic Absorption Spectrometry* (EPA 1994), with modifications. For analysis of Al, a crustal element, the sediments are digested using a total digestion with boric acid dissolution according to Battelle SOP MSL-I-006, *Mixed Acid Sediment Digestion.* The digestates are analyzed for Al by ICP-MS.

If results of analysis by ICP-MS do not meet data quality objectives, metals are present in high concentrations (*e.g.*, crustal elements), or if different detection limits are required, individual elements may be reanalyzed by GFAA; or hydride generation atomic absorption (HGAA) spectroscopy with flow injection analysis system (FIAS). GFAA analysis is conducted according to Battelle SOP MSL-I-029, *Determination of Metals in Aqueous and Digestate Samples by GFAA*, based on EPA Method 200.9 (EPA 1991b). HGAA/FIAS analysis is conducted according to Battelle SOP MSL-I-030, *Determination of Metals in Aqueous and Digestate Samples by HGAA-FIAS*, which conforms to specifications in the EPA Method 200 series and EPA SW-846 Method 7000 series.

Sediment – Physical Parameters and Total Organic Carbon (TOC). Grain size is measured following two methods described by Plumb (1981)(AMS SOP AMS-PGS88). The wet sieve method is used to determine the size distribution of sand or coarser-grained particles larger than a U.S. No. 230 standard sieve (62.5-μm mesh). The size distribution of particles smaller than an U.S. No. 230 sieve is determined using the pipet method. Grain size is reported as percentages within four general size classes:

gravel	≥2000 μm diameter
sand	≥ 62.5-μm and <2000 μm diameter
silt	≥ 3.9-μm diameter and < 62.5-μm diameter
clay	< 3.9-μm diameter.

Percentage of moisture is obtained using the Plumb (1981) method for determining total solids. The procedure involves drying a sediment sample at 100°C until a constant weight is obtained. Percentage of moisture is calculated by subtracting the percentage of total solids from 100%.

Sediment samples are analyzed for TOC according to the EPA Edison, New Jersey, Laboratory procedure (EPA 1986; AMS SOP AMS-9901). Inorganic carbon is removed from the sample by acidification. The sample is combusted and the evolved carbon dioxide is quantitated using a carbon-hydrogen-nitrogen (CHN) analyzer. TOC is reported as a percentage of the dry weight of the unacidified sample.

Instrumental Analysis, Calibration, and Maintenance

All calibration standards and standard solutions are traceable to the source. Logbooks are maintained which include the certificates of analysis from the vendor, lot numbers, and purity information. Additionally, all calibration solutions are quantitatively checked using standards secured from a second source.

Organics – GC/ECD Analysis for Pesticides/PCBs. Samples will be quantified based on recovery internal standards added to the extracts just prior to analysis. Surrogate recoveries will be quantified and reported for each sample. GC/ECD analysis for pesticides and PCBs will be determined according to Battelle SOP 5-128, *Identification and Quantification of Polychlorinated Biphenyls (by Congener and Aroclor) and Chlorinated Pesticides by Gas Chromatography/Electron Capture Detection.*

Instrument methods and maintenance applicable to GC/ECD analysis of samples for PCB/pesticides will conform to guidance presented in Battelle SOP No. 3-116. Dual-column analysis will be performed. The data collected from the confirmatory analysis will be consulted when necessary; this additional analysis is not required. The Battelle method uses a 5-point (minimum) calibration curve with 22 individual PCB congeners, and the linear regression correlation coefficient must be 0.995 or better; this is a modification of EPA Method 8081. A mid-level calibration check standard will be analyzed no less frequently than every 10 samples. This check standard will be quantified in the same manner as field and QC samples. The concentrations of the target analytes must be within 25% of true concentration, and the average percent difference between the detected concentrations and true concentrations must be below 15%. If the percent difference between the detected and true concentrations of representative pesticides and PCB congeners is greater than the acceptability criteria, remedial maintenance will be performed on the instrument, and a new calibration check standard analyzed. If the check standard meets the acceptance criteria, then the affected samples will be reanalyzed. If the check standard does not meet the acceptance criteria then a new initial calibration will be performed, and the affected batch of samples will be reanalyzed, at the discretion of the analyst and project management. Because GC/ECD analysis is a multicomponent analysis, it may not be necessary to reanalyze all samples. For example, if only certain analytes are detected in a sample, and the calibration is acceptable for those particular analytes, the sample should not require reanalysis. Reanalyses will be performed at the discretion of the Project or Task Manager. Deviations from calibration or data objectives will be documented in the project files.

Organics – GC/MS Analysis for PAHs. Samples will be quantified based on recovery internal standards added to the extracts just prior to analysis. Surrogate recoveries will be quantified and reported for each sample.

Instrumental methods, maintenance, and QC procedures for the GC/MS analysis of samples for PAH will be performed according to Battelle SOP No. 5-157, the NOAA Status and Trends methodology, a modification of EPA Method 8270. Analytical instruments will be calibrated before sample analysis. The GC/MS will be tuned with perfluorotributylamine (PFTBA) before the initiation of the sample sequence. Response factors (RF) for GC/MS analysis will be generated for each PAH target analyte. At a minimum, five concentrations of standard solutions that encompass the expected range in sample concentrations will be analyzed. Initial calibrations will be acceptable if the relative standard deviations (RSD) are within 25% of the mean for each individual analyte, and the mean of all analyte RSD is 15%. Any initial calibration that does not meet these criteria will be reviewed by the task leader and reanalyzed. Any exceptions will be documented and justified by the Project or Task Manager.

At a minimum, the GC/MS system calibrations will be verified in the middle and at the end of each sample batch, using a mid-range calibration check. Using the mean RF of each analyte from the initial calibration, the percent difference between those mean values and the RFs from the midrange calibration checks will be calculated. The calibration checks will be acceptable under the same criteria as the initial calibration (*i.e.*, 25% for individual analytes, 15% for average RF). If the percent difference between the RFs is greater than the acceptability criteria, remedial maintenance will be performed on the instrument, and a new calibration check standard analyzed. If the check standard meets the acceptance criteria, then the affected samples will be reanalyzed. If the check standard does not meet the acceptance criteria then a new initial calibration will be performed, and the affected batch of samples will be reanalyzed, at the discretion of the Task Leader. Because GC/MS analysis is a multicomponent analysis, it may not be necessary to reanalyze all samples. For example, if only certain analytes are detected in a sample, and the calibration is acceptable for those particular analytes, the sample should not require reanalysis. Reanalyses will be performed at the discretion of the Task Leader. Deviations from calibration or data objectives will be documented in the project files.

Samples analyzed by GC/MS will be bracketed by two acceptable calibrations, initial or check. Analytes will be quantified by using the average RFs for that individual PAH analyte generated from the initial calibration.

Butyltins – GC/FPD. Sample extracts will be analyzed for butyltins using capillary gas chromatography with flame photometric detection (GC/FPD) following procedures described in Battelle SOP 3-100, *Operation of the Hewlett-Packard Flame Photometric Detector (FPD).* Butyltins will be quantified using the method of internal standards. A minimum of a three-point calibration curve will be performed for each butyltin compound relative to the SIS TPT. A calibration check standard will be analyzed after every 10 samples to ensure that the GC/FPD system remains in calibration over the course of sample analysis. All samples will be bracketed by two passing calibrations.

Because the analysis of butyltin compounds requires many steps – including a derivitization and cleanup procedure where it is possible for significant losses of the target compounds to occur – quantification will be carried out using the method of internal standards. The surrogate internal standard tripropyltin chloride (TPT) will be added at the beginning of the extraction procedure, and the concentration of the analytes in the samples calculated relative to TPT. The overall recovery efficiency of the method will be measured by calculating the recovery of TPT relative to the recovery internal standard dipropylpentyltin, added just prior to GC analysis.

Metals – ICP-MS, GFAA, ICP-AES, and CVAA/CVAF Analysis. For each of the instruments used in metals analyses, a three- or four-point calibration (minimum) is performed just prior to sample analysis. A calibration check standard is analyzed after every 10 authentic samples to ensure that the instrument remains in calibration over the course of sample analysis. If the percent difference is greater than the acceptability criteria, remedial maintenance will be performed on the instrument, a new initial calibration will be performed, and the affected batch of samples will be reanalyzed at the discretion of the analyst and project management. Deviations from calibration or data objectives will be documented in the project files.

The ICP-MS is tuned as described in Battelle SOP MSL-I-022, based on EPA Method 200.8. The tuning solution used includes ^{24}Mg, ^{103} Rh, and ^{208} Pb to determine the resolution of the instrument for low, medium and high masses. Routine maintenance is performed by the manufacturer twice yearly or as needed, and any daily maintenance is recorded in a logbook.

Calibration procedures for the GFAA are described in Battelle SOP MSL-I-029. Routine maintenance is recorded in a logbook.

Instrument methods, calibration procedures and maintenance applicable to CVAA analyses follow Battelle SOP MSL-I-016. Mercury quantification will be performed by the method of external standards, using a linear regression curve or a response factor approach.

Documentation, Data Reduction, and Reporting

All laboratory records will be stored in dedicated project files and on project specific data forms. Battelle will provide reports containing sampling information, testing results and QA/QC information. The standard turnaround time for water/elutriate and sediment data is 60 days from sample collection. Tissue data will be delivered within 90 days of sample receipt. Draft data can also be delivered (faxed) within 24 hours of availability as determined by the Project Manager. Data will be maintained and archived for at least three years following submission of final report.

ORGANIZATION

The following individuals are key personnel assigned to each of the required tasks of an individual Work Order:

Management and Reporting:	Carole Peven McCarthy, Program Manager Elisabeth S. Barrows, Deputy Program Manager
Analytical Chemistry:	Carole Peven McCarthy, Analytical Chemistry Project Manager - Elisabeth S. Barrows, Metals Analysis Project Manager - Jill Brandenberger, Metals Analysis Task Leader

- Carole Peven McCarthy, Organics Analysis Project Manager
- Kenneth Davis, Applied Marine Sciences, Inc., Physical Characterizations

Quality Assurance: Mark Guilmain, Battelle Duxbury Operations
Deborah Coffey, Battelle Marine Sciences Laboratory

SCHEDULE

Standard turnaround time (TAT) as defined in the RTM for delivery of sediment chemical analysis results is 60 days from sample collection,

PROJECT ORGANIZATION AND COMMUNICATION PLAN

Carole Peven McCarthy will be the project manager for the Ponce Harbor dredge material project. She has the ultimate responsibility for client communication and data reporting. Elisabeth S. Barrows will be the deputy project manager, and will act on Ms. McCarthy's behalf when necessary. She will also oversee the metals analysis task. A task leader has been assigned responsibility for each analytical task, as follows:

Organics Task Leader: Carole Peven McCarthy
Metals Task Leader: Jill Brandenberger

These task leaders will be responsible for the project-specific QAPPs, oversight of the day-to-day progress of the project, and delivery of audited data to the PM in a timely manner. The task leader will provide schedule information to the PM on a regular basis.

Quality Assurance

The Quality Assurance Officer will remain independent of all work activities pertaining to this project. The QA Officer will monitor the project according to existing Battelle SOP's to ensure the accuracy, integrity, and completeness of the data. The QA Program scope includes system inspections, data audits, and document review. Data will be audited (targeted) prior to delivery to the client.

Sampling and Analytical Quality Control Procedures

Project staff will be responsible for ensuring that sample tracking, sample preparation, and analytical instrument operation all meet the QC criteria detailed in applicable analytical SOPs. A list of applicable SOPs is presented in Table 4 of this document. The type and frequency of analysis of QCl samples for this study is specified in Table 1 of the project-specific lab plan. Data quality objectives and acceptance criteria for each type of QC sample analysis are provided in Table 5. Corrective actions associated with the QC samples are also described in Table 5. Data qualifiers will be applied to the data summary tables as defined in Table 6. A list of standard reference materials (SRM) used in chemistry analyses associated with this project and the certified, reference or consensus values for each analyte are provided in appendix A.

Table 4. Pertinent Standard Operating Procedures

SOP No.	Title
3-160	Use of Electronic Balances
4-009	Quality Assurance Facilities Inspections
4-015	Quality Assurance Audits of Reported Data
5-128	Identification and Quantification of Polychlorinated Biphenyls (by Congener and Aroclor) and Chlorinated Pesticides by Gas Chromatography/Electron Capture Detection
5-157	Identification and Quantification of Polynuclear Aromatic Hydrocarbons by Gas Chromatography/Mass Spectrometry
5-191	HPLC Cleanup of Samples for Semivolatile Organic Pollutants
5-192	Sediment Extraction for Trace Level Semivolatile Organic Contaminants
5-216	Cleaning of Organic Chemistry Labware
6-007	Chemistry Laboratory Sample Identification
6-010	Chemistry Laboratory Sample Control
6-017	Data Recording
7-029	Reporting Quality Control Data in the Organic Chemistry Laboratory
5-196-07	Measurement of Butyltin Species in Tissues and Sediment/Soil
MSL-A-001	Sample Log In Procedure
MSL-A-002	Sample Chain of Custody
MSL-A-005	Deviations from Established Requirements
MSL-C-009	Use and Performance Checks of Balances
MSL-C-010	Calibration and Use of Pipettes
MSL-C-013	Glassware and Equipment Cleaning Procedures
MSL-A-014	Sample Container Requests
MSL-Q-002	Quality Assurance Inspections of MSL System and Study Activities
MSL-Q-003	Quality Assurance Audits of Reports
MSL-Q-005	Quality Assurance Data Audits
MSL-Q-007	Procedures for Determining Method Detection Limits
MSL-I-006	Mixed Acid Sediment Digestion
MSL-I-025	Methods of Sample Preconcentration: Cobalt/APDC Coprecipitation and Borohydride Reductive Precipitation for Trace Metals Analysis in Water
MSL-I-016	Total Mercury in Tissues and Sediments by Cold Vapor Atomic Absorption
MSL-I-022	Determination of Elements in Aqueous and Digestate Samples by ICP-MS
MSL-I-027	Determination of Metals in Aqueous and Digestate Samples by ICP-AES
MSL-I-029	Determination of Metals in Aqueous and Digestate Samples by GFAA
MSL-I-030	Determination of Metals in Aqueous and Digestate Samples by HGAA/FIAS
AMS-9509	Preventative Maintenance Requirements UIC Model 150 Coulometric Carbon Analyzer
AMS-9510	Calibration Procedures UIC Model 150 Coulometric Carbon Analyzer
AMS-PGS88	Determination of Particle Size Distribution (Percent Cobble, Gravel, Sand, Silt and Clay Fractions) in Sediment Samples
AMS-9901	Determination of Total Organic Carbon, Total Inorganic Carbon, and Total Organic Carbon Content of Sediment Samples by Coulometric Detection

Table 5. Data Quality Objectives

QC Sample Type	Acceptance Criteria	Corrective Action
Procedural Blank	Organics - < 3 x MDL Butyltins - < 3 x MDL Metals - < 3 x MDL	Apply data qualifiers to affected data. Reextraction, reanalysis, and/or blank subtraction - determined by PM or Task Leader; all corrective actions documented
SRM	Evaluated for analytes >10x the MDL. Organics - 30% difference vs. certified values using upper and lower 95% confidence interval values on average, not to exceed 30% difference for more than 35% of individual analytes; no recovery >50% difference Butyltins – NA (none available) Metals - ≤20% of certified value using upper and lower 95% confidence interval values; no more than 35% can exceed 20% difference of certified value; no recoveries >50% difference from certified value TOC - <20% difference from established value	Apply data qualifiers to affected data. Results examined by PM or Task Leader. Corrective action (reextraction, reanalysis) or justification documented.
Matrix Spike	Organics, TBT and DBT - 50 to 120% recovery for at least 80% of analytes Metals – 75 to 125% recovery	Apply data qualifiers to affected data. Results examined by PM or Task Leader. Corrective action (reextraction, reanalysis) or justification documented.
Triplicate	Evaluated for analytes >10x the MDL. Organics and Butyltins - 30% RSD for analytes >10 x MDL; no RSD >70% for analytes >10x MDL; <35% of analytes >30% RSD Metals – 20% RSD for analytes >10x MDL; no RSD >70% for analytes >10x MDL; <35% of analytes >20% RSD. TOC – 10% RSD Physical Measurements – 20% RSD	Apply data qualifiers to affected data. Document deviations.
SIS	Organics - 30-150% recovery Butyltins – 15 – 120% recovery	Apply data qualifiers to affected data. Results examined by PM or Task Leader. Corrective action (reextraction, reanalysis) or justification documented.
Calibrations: Initial	Organics and Butyltins - 25% RSD individual analyte 15% RSD average of all analytes Correlation coeff. > 0.995 Metals - <15% difference from true value Correlation coeff. >0.995	Analysts reanalyze or document and justify
Initial Calibration Check	Organics and Butyltins - <15% recovery Metals -- <15% recovery	Analysts reanalyze or document and justify
Continuing Calibration Check	Organics - <20% difference from initial calibration Metals - <20% difference from initial calibration	Remedial maintenance, new initial calibration, reanalyze samples at discretion of analyst and Task Leader. Decision documented and/or justified.

Section 3

Measurement Quality Objectives

Accuracy is defined as the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations. Accuracy is evaluated through analysis and recovery of analytes from blank spike or laboratory control samples, matrix spike samples, and SRMs.

Precision is defined as degree to which a set of observations or measurements of the same property, obtained under similar conditions, conforms to itself. Precision is usually expressed as standard deviation, variance, or range, in either absolute or relative terms. Precision is evaluated through the analysis of replicate samples.

Completeness is the amount of data collected as compared to the amount needed to ensure that the uncertainty or error is within acceptable limits. The goal for data completeness is 100%.

Comparability is a measure of the confidence with which one data set can be compared to another. This is a qualitative assessment and is addressed primarily in sampling design through use of comparable sampling procedures or, for monitoring programs, through accurate re-sampling of stations over time. In the laboratory, comparability is assured through the use of comparable analytical procedures and ensuring that project staff are trained in the proper application of the procedures. Within-study comparability will be assessed through analytical performance (quality control samples).

Representativeness is the degree to which data accurately and precisely represent a characteristic of a population. This is a qualitative assessment and is addressed primarily in the program design, through the selection of sampling sites, and procedures that reflect the project goals and environment being sampled. It is ensured in the laboratory through (1) the proper handling, homogenizing, compositing, and storage of samples and (2) analysis within the specified holding times so that the material analyzed reflects the material collected as accurately as possible.

Data Validation

A series of reviews by technical personnel will be implemented to ensure that the data generated for this project meet the data quality objectives. These reviews will include the following:

- Data will be reviewed by the laboratory personnel periodically to ensure that sample analytical activities are completely and adequately documented.
- Reviews of analytical results and supporting documentation will be the responsibility of the Lab Managers. The Lab Manager will review sample holding times, sample preservation, equipment calibration, and sample integrity. The results of QC measurements will be compared to pre-established criteria as a measure of data acceptability.
- All calculations performed manually will be checked for accuracy by someone other than who performed the original calculation. Checking will be performed by qualified persons who did not participate in performing the calculations. Checking will be documented, by signature and date on the calculation worksheet. Separate documentation is acceptable, provided traceable records are maintained.
- For verification of data input into spreadsheets and/or data bases, the staff member entering data will ensure correct entry into the software by comparing data with the hard copy of the data listing. If errors are discovered, the errors will be corrected and a new data listing generated. When data are correctly entered, the staff member entering the data will sign and date the correct data listing and submit it as a project record.

A final data audit by the Quality Assurance Officer will be performed prior to submission of the data and report to the client. This audit ensures that the data are accurate, traceable, defensible, and complete, as compared to the planning documents. The audit procedure (SOPs 4-015, *Quality Assurance Audits of Reported Data* and MSL-Q-005, *Quality Assurance Data Audits*) is a statistical, randomized check that involves comparing

selected reported values to the original data. This procedure is designed to ensure a 95 percent chance of detecting whether one percent or more reported values disagree with the original data.

Corrective Action

A prompt and thorough correction of non-conformance conditions that can affect data quality minimizes the possibility of questionable data or documentation. Immediate corrective actions include correction of documentation deficiencies or errors, repair of inaccurate instrumentation, or correction of inadequate procedures. Internal laboratory corrective actions to be implemented if analytical results exceed data quality objectives are defined in Table 5. Responsibilities of analysts, task leaders, and project managers are identified in Table 5 as well. Where corrective action is not feasible, appropriate qualifiers will be added to the data reported (see Table 6).

Quality problems and non-conformance conditions, including any deviations to this WP/QAPP will be documented to provide a complete record of QA activities. Corrective action reporting and documentation procedures are defined in SOPs 4-035, *Corrective Action*, and MLS-A-005, *Deviations from Established Requirements*.

Data Reporting

At the completion of all analyses, data package reports will be compiled for submittal to the client. Data reports will include, but are not limited to, the following items:

- Data report tables summarizing analytical results for each sample. Results of sample analyses are reported in the following units:
  Sediment: Organics, ng/g dry weight; Metals, μg/g dry weight
- All raw and reduced data for all analyses (*e.g.*, laboratory worksheets/benchsheets, instrument printouts)
- Names of laboratories and personnel performing analyses
- Description and results of QC checks and data validation. Results of QC sample analyses will be provided in the data report summary tables and additionally described in QC narratives and/or QC sample checklists
- Analytical methods and observations (described in narratives)
- Clear mechanisms for correlating sample collection and sample analytical results (sample tracking, sample and project identification)
- Sample custody and log-in forms
- Problems encountered or deviations prescribed laboratory protocols and corrective actions taken

In addition to the hard-copy data package, analytical results (data report summary tables) will be provided in electronic (Microsoft® *Excel* spreadsheet) format, either via e-mail or on diskette.

Sample and Data Disposition

Sample material will be archived on-site for up to 1 year from sample collection; archived sediment will be stored at -20 °C. Data and project records will be stored for at least 3 years from test start date. After this period of time, Battelle will contact the client for authorization to dispose of the samples and records, or will arrange for extended storage/archival with the client.

Table 1. Data Qualifiers

Qualifier	Use
J	Detected, but below the MDL (applies to organics data).
B	Analyte detected in the method or procedural blank at level greater than 3× MDL. The qualifier is entered for the blank and affected field samples.
ME	Estimate, significant Matrix Interference.
U	Undetected at or above MDL; MDL reported
&	QC value outside the accuracy or precision data quality objective.
#	Value from confirmation column (applies to organics data).

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National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1944

New York/New Jersey Waterway Sediment

Standard Reference Material (SRM) 1944 is a mixture of marine sediment collected near urban areas in New York and New Jersey. SRM 1944 is intended for use in evaluating analytical methods for the determination of selected polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners, chlorinated pesticides, and trace elements in marine sediment and similar matrices. Reference values are also provided for selected dibenzo-*p*-dioxin and dibenzofuran congeners, total organic carbon, total extractable material, and particle-size characteristics. All of the constituents for which certified, reference, and information values are provided in SRM 1944 were naturally present in the sediment material before processing. A unit of SRM 1944 consists of a bottle containing 50 g of radiation sterilized, freeze-dried sediment material.

Certified Concentration Values: Certified values for concentrations, expressed as mass fractions, for 24 PAHs, 35 PCB congeners (some in combination), four chlorinated pesticides, and nine trace elements are provided in Tables 1-4. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST. The certified values for the PAHs, PCB congeners, and chlorinated pesticides are based on the agreement of results obtained at NIST from two or more chemically independent analytical techniques. The certified values for the trace elements are based on NIST measurements by one technique and additional results from several collaborating laboratories.

Reference Concentration Values: Reference values for concentrations, expressed as mass fractions, are provided for 32 additional PAHs (some in combination) in Table 5, seven additional chlorinated pesticides in Table 6, and 19 additional inorganic constituents in Tables 7 and 8. Reference values are provided in Table 9 for the 17 2,3,7,8-substituted polychlorinated dibenzo-*p*-dioxin and dibenzofuran congeners and total tetra-, penta-, hexa-, and hepta-congeners of polychlorinated dibenzo-*p*-dioxin and dibenzofuran. Reference values for particle-size characteristics are provided in Table 10. Reference values for total organic carbon and percent extractable mass are provided in Table 11. Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods. Explanations in support of each reference value are given as notes in Tables 5-11.

Information Concentration Values: Information values for concentrations, expressed as mass fractions, are provided in Table 12 for eight additional trace elements. An information value is considered to be a value that will be of interest and use to the SRM user, but insufficient information is available to assess the uncertainty associated with the value or only a limited number of analyses were performed.

Expiration of Certification: The certification of this SRM lot is valid until **31 March 2009**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification is nullified if the SRM is damaged, contaminated, or modified.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by B.S. MacDonald.

Willie E. May, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 14 May 1999

Thomas E. Gills, Director
Office of Measurement Services

Table 3. Certified Concentrations for Selected Chlorinated Pesticides in SRM 1944

Chlorinated Pesticides	Mass Fractions in µg/kg (dry-mass basis)[a,b]
Hexachlorobenzene[e,f,g,h,i,j]	6.03 ± 0.35
cis-Chlordane (α-Chlordane)[c,d,e,f,g,h,i,j]	16.51 ± 0.83
trans-Nonachlor[c,d,e,f,g,h,i,j]	8.20 ± 0.51
4,4'-DDT[c,d,e,f,g,h,i,j]	119 ± 11

[a] Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.
[b] The results are expressed as the certified value ± the expanded uncertainty. Each certified value is a mean of the means from two or more analytical methods, weighted as described in Paule and Mandel [23]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [24], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty within each analytical method as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.
[c] GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.
[d] GC-ECD (IB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IA).
[e] GC-ECD (IIA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.
[f] GC-ECD (IIB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).
[g] GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone.
[h] GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with 50 % hexane/50 % acetone.
[i] GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).
[j] Results from 19 laboratories participating in an interlaboratory comparison exercise.

Table 4. Certified Concentrations for Selected Inorganic Constituents in SRM 1944

Elements	Degrees of Freedom	Mass Fractions in percent (dry-mass basis)[a,b]
Aluminum[c,d,e]	4	5.33 ± 0.49
Iron[c,d,e]	6	3.53 ± 0.16
		Mass Fractions in mg/kg (dry-mass basis)[a,b]
Arsenic[c,d,e,f,g]	10	18.9 ± 2.8
Cadmium[c,f,h,i]	6	8.8 ± 1.4
Chromium[c,d,f,g,i]	9	266 ± 24
Lead[c,h,i]	5	330 ± 48
Manganese[c,d,e]	8	505 ± 25
Nickel[c,g,h,i]	6	76.1 ± 5.6
Zinc[c,d,e,g,i]	9	656 ± 75

[a] The results are expressed as the certified value ± the expanded uncertainty. The certified value is the mean of four results: (1) the mean of NIST INAA or ID-ICPMS analyses, (2) the mean of two methods performed at NRCC, and (3) the mean of results from seven selected laboratories participating in the NRCC intercomparison exercise, and (4) the mean results from INAA analyses at IAEA. The expanded uncertainty in the certified value is equal to $U = ku_c$, where u_c is the combined standard uncertainty and k is the coverage factor, both calculated according to the ISO Guide [24]. The value of u_c is intended to represent at the level of one standard deviation the combined effect of all the uncertainties in the certified value. Here u_c accounts for both possible method biases, within-method variation, and material inhomogeneity. The coverage factor, k, is the Student's t-value for a 95 % prediction interval with the corresponding degrees of freedom. Because of the material inhomogeneity, the variability among the measurements of multiple samples can be expected to be greater than that due to measurement variability alone.
[b] Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.
[c] Results from five to seven laboratories participating in the NRCC interlaboratory comparison exercise.
[d] Measured at NIST using INAA.
[e] Measured at NRCC using ICPAES.
[f] Measured at NRCC using GFAAS.
[g] Measured at IAEA using INAA.
[h] Measured at NIST using ID-ICPMS.
[i] Measured at NRCC using ID-ICPMS.

Table 5. Reference Concentrations for Selected PAHs in SRM 1944

Table 6. Reference Concentrations for Selected Chlorinated Pesticides in SRM 1944

NOTE: These concentrations are provided as reference values because either the results have not been confirmed by an independent analytical technique as required for certification or the agreement among results from multiple methods was insufficient for certification. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures.

Chlorinated Pesticides	Mass Fractions in µg/kg (dry-mass basis)[a,b]
α-HCH[c,d,e,f]	2.0 ± 0.3
trans-Chlordane (γ-Chlordane)[c,d,e,f,g,h,i,j]	8 ± 2
cis-Nonachlor [d,e,f,i,j]	3.7 ± 0.7
2,4'-DDE [c,d,e,f,g,h,i,j]	19 ± 3
2,4'-DDD [e,f,g,h,i,j]	38 ± 8
4,4'-DDE [c,d,e,f,g,h,i,j]	86 ± 12
4,4'-DDD [c,d,e,f,g,h,i,j]	108 ± 16

[a] Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.
[b] The reference value for each analyte is the equally-weighted mean of the means from two or more analytical methods or the mean from one analytical technique. The uncertainty in the reference value defines a range of values that is intended to function as an interval that contains the true value at a level of confidence of 95 %. This uncertainty includes sources of uncertainty within each analytical method, among methods, and from the drying study.
[c] GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.
[d] GC-ECD (IB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IA).
[e] GC-ECD (IIA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.
[f] GC-ECD (IIB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).
[g] GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone.
[h] GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with 50 % hexane/50 % acetone.
[i] GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts anlayzed as in GC-ECD (IIA).
[j] Results from 19 laboratories participating in an interlaboratory comparison exercise.

Table 7. Reference Concentrations for Selected Inorganic Constituents in SRM 1944 as Determined by Multiple Laboratories

NOTE: These concentrations are provided as reference values because either the results have not been confirmed by an independent analytical technique as required for certification, the agreement among results from multiple methods was insufficient for certification, or insufficient analyses have been performed at NIST to confirm the results of the outside laboratories.

Elements	Degrees of Freedom	Mass Fraction in percent (dry-mass basis)[a,b]
Silicon[c,d]	81	31 ± 3
		Mass Fraction in mg/kg (dry-mass basis)[a,b]
Beryllium[c,h]	17	1.6 ± 0.3
Copper[c,d,f]	101	380 ± 40
Mercury[c,i]	18	3.4 ± 0.5
Selenium[c,e,f]	24	1.4 ± 0.2
Silver[c,d,e,g]	8	6.4 ± 1.7
Thallium[c,f]	12	0.59 ± 0.1
Tin[c,f]	22	42 ± 6

[a] The results are expressed as the reference value ± the expanded uncertainty. The reference value is the equally weighted mean of available results from: (1) NIST INAA analyses, (2) two methods performed at NRCC, (3) results from seven selected laboratories participating in the NRCC intercomparison exercise, and (4) results from INAA analyses at IAEA. The expanded uncertainty in the reference value is equal to $U = ku_c$ where u_c is the combined standard uncertainty and k is the coverage factor, both calculated according to the ISO Guide [24]. The value of u_c is intended to represent at the level of one standard deviation, the uncertainty in the value. Here u_c accounts for both possible method differences, within-method variation, and material inhomogeneity. The coverage factor, k, is the Student's t-value for a 95 % prediction interval with the corresponding degrees of freedom. Because of material inhomogeneity, the variability among the measurements of multiple samples can be expected to be greater than that due to measurement variability alone.

Certified Reference Material

National Research Council Canada | Conseil national de recherches Canada

HISS-1, MESS-3, PACS-2

Marine Sediment Reference Materials for Trace Metals and other Constituents

The following tables show those constituents for which certified and information values have been established. Certified values are based on the results of determinations by at least two independent methods of analysis. The uncertainties represent 95% confidence limits for an individual sub-sample of 250 mg or greater.

Trace Metals
(milligrams per kilogram)

	HISS-1	MESS-3	PACS-2
Antimony	(0.13)*	1.02 ± 0.09	11.3 ± 2.6
Arsenic	0.801 ± 0.099	21.2 ± 1.1	26.2 ± 1.5
Beryllium	0.129 ± 0.023	2.30 ± 0.12	1.0 ± 0.2
Cadmium	0.024 ± 0.009	0.24 ± 0.01	2.11 ± 0.15
Chromium	30.0 ± 6.8†	105 ± 4	90.7 ± 4.6
Cobalt	(0.65)*	14.4 ± 2.0	11.5 ± 0.3
Copper	2.29 ± 0.37	33.9 ± 1.6	310 ± 12
Lead	3.13 ± 0.40	21.1 ± 0.7	183 ± 8
Lithium	2.83 ± 0.54	73.6 ± 5.2	32.2 ± 2.0
Manganese	66.1 ± 4.2	324 ± 12	440 ± 19
Mercury	(0.01)*	0.091 ± 0.009	3.04 ± 0.20
Molybdenum	(0.13)*	2.78 ± 0.07	5.43 ± 0.28
Nickel	2.16 ± 0.29	46.9 ± 2.2	39.5 ± 2.3
Selenium	0.050 ± 0.007	0.72 ± 0.05	0.92 ± 0.22
Silver	0.016 ± 0.002	0.18 ± 0.02	1.22 ± 0.14
Strontium	96.9 ± 11.2	129 ± 11	276 ± 30
Thallium	(0.06)*	0.90 ± 0.06	(0.6)*
Tin	(0.11)*	2.50 ± 0.52	19.8 ± 2.5
Uranium	(0.26)*	(4)*	(3.)*
Vanadium	6.80 ± 0.78	243 ± 10	133 ± 5
Zinc	4.94 ± 0.79	159 ± 8	364 ± 23
Tributyltin (as Sn)	---	---	0.98 ± 0.13
Dibutyltin (as Sn)	---	—	1.09 ± 0.15
Monobutyltin (as Sn)	---	---	0.45 ± 0.05

*information value only
† see page 3

NRC · CNRC

Matrix and Minor Constituents - Percent

		HISS-1	MESS-3	PACS-2
Al	(f,i,n,x)	0.73 ± 0.05	8.59 ± 0.23	6.62 ± 0.32
C	(e)	---	(2)*	(3.3)*
Ca	(f,i,n,x)	1.14 ± 0.10	1.47 ± 0.06	1.96 ± 0.18
Cl	(n,x)	(0.35)*	---	(3.)*
Fe	(f,i,n,x)	0.246 ± 0.009	4.34 ± 0.11	4.09 ± 0.06
K	(f,n,x)	0.332 ± 0.013	(2.6)*	1.24 ± 0.05
Mg	(f,i,x)	0.075 ± 0.016	(1.6)*	1.47 ± 0.13
Na	(f,i,n)	0.373 ± 0.026	(1.6)*	3.45 ± 0.17
P	(i,x)	---	(0.12)*	0.096 ± 0.004
S	(i,x)	---	(0.19)*	1.29 ± 0.13
Si	(i,x)	(44.)*	(27.)*	(28.)*
Ti	(f,i,n,x)	0.076 ± 0.004	0.44 ± 0.06	0.443 ± 0.032

Methods of Determination for Trace Metals

Antimony (b,g,i,n,q)
Arsenic (b,g,h,i,n,x)
Beryllium (b,g,i)
Cadmium (b,g,i,q)
Chromium (b,g,i,n,q,x)
Cobalt (b,g,i,n,x)
Copper (b,g,i,n,q,x)
Lead (b,g,i,q,x)
Lithium (b,g,i,q)
Manganese (b,g,i,n,x)
Mercury (a,c,q)
Molybdenum (g,i,q)
Nickel (b,g,i,q,x)
Selenium (b,g,h)
Silver (b,g,i,q)
Strontium (f,i,n,q,x)
Thallium (b,q)
Tin (b,g,i,q)
Uranium (q)
Vanadium (b,g,i,n,x)
Zinc (b,g,i,n,q,x)
organotin species (l,m)

Coding

a - Atomic fluorescence spectrometry
b - Inductively coupled plasma mass spectrometry (ICPMS)
c - Cold vapour atomic absorption spectrometry
e - Coulometry
f - Flame atomic absorption spectrometry
g - Graphite furnace atomic absorption spectrometry
h - Hydride generation atomic absorption spectrometry
i - Inductively coupled plasma atomic emission spectrometry
l - High-performance liquid chromatography ICPMS
m - Gas chromatography microwave induced plasma atomic emission
n - Instrumental neutron activation analysis
q - Isotope dilution inductively coupled plasma mass spectrometry
r - Infrared spectrometry
x - X-ray fluorescence spectrometry

Not all the methods listed above were applied to all three certified reference materials.

These reference materials are primarily intended for use in the calibration of procedures and the development of methods used for the analysis of marine sediments and materials with similar matrices.

Preparation of material

HISS-1 was collected from the Hibernia Shelf, off the coast of Newfoundland. MESS-3 is from the Beaufort Sea. PACS-2 was collected in the harbour of Esquimalt, B.C. They were all freeze dried, screened to pass a No. 120 (125 µm) screen, blended and bottled by Institute staff using the facilities of the Canada Centre for Mineral and Energy Technology in Ottawa. After bottling, the samples were radiation sterilized with a minimum dose of 2.5 Mrad by Nordion International Inc. to minimize any effects from biological activity.

Instructions for drying

Although initially free from moisture following the freeze drying, the materials, which contain sea salt, have picked up moisture during subsequent operations. They should be dried to a constant weight before use. Drying for several hours at 105°C has proved to be a relatively simple method to achieve a dry weight for most purposes.

Storage

Repeated analysis of these materials has demonstrated that elemental concentrations are stable for at least 10 years when bottles are stored well sealed, and in a cool place. Each bottle is packaged in a trilaminate foil pouch as an impermeable barrier to mercury vapour. Experiments have shown that, under conditions of high ambient levels of mercury vapour, mercury is able to penetrate the plastic cap of the bottle, thereby potentially contaminating the contents.

Storage of PACS-2 for Organotin Stability

To ensure the stability of the organotin species in PACS-2 it is necessary to store the material at a temperature of 4°C or lower.

Information values

Information values are considered less reliable than certified values because they are not based on the results of at least two independent methods, there were insufficient analyses performed or inhomogeniety is suspected. These numbers are given for information only and care should be excised not to attribute more reliability to these numbers than they warrant.

Homogeneity

Randomly selected bottles were used for the analytical determinations. Results from different bottles showed no significant differences compared to results from sub-samples within bottles. Nor was there any correlation between values obtained and bottle sequence. Thus, it is assumed that all bottles of each of these materials have essentially the same composition. One exception is Co in HISS-1 where results indicated sample inhomogeniety.

†Chromium in HISS-1

It became apparent during the certification of HISS-1 that there is a significant fraction of Cr that is not easily solubilized. The certified value of 30 mg/kg was obtained using solid sampling techniques or prolonged digestion with hydrofluoric, sulphuric and perchloric acids. Less vigorous acid dissolution techniques (including microwave heating using closed vessels at high pressure) result in Cr values between 10 and 13 mg/kg.

Acknowledgements

These materials were prepared following the advice of the NRC Committee on Marine Analytical Chemistry. The guidance of the members is much appreciated.

The following staff of the Institute for National Measurement Standards, National Research Council of Canada, participated in the analysis of at least one of these sediment certified reference materials:
S. Berman, V.J. Boyko, V.P. Clancy, J. Lam, P. Maxwell, J.W. McLaren, B. Methven, C. Scriver, K.W.M. Siu, R. Sturgeon, S. Willie and L. Yang.

The cooperation of the following during the certification of one or more of these sediments is gratefully acknowledged:

Nordion International Inc., Ville de Laval, Quebéc.

E. Crecelius, B. Lasorsa, C.W. Apts, O.A. Cotter, R.W. Sanders and T. Gilfoil, Battelle Pacific Northwest, Sequim, Washington.

R. Presley, P. Boothe, R. Taylor and T. Wade, Texas A & M University, College Station, Texas.

M. Leaver, R. Beaudoin and H. Steger, Canada Centre for Mineral and Energy Technology, Natural Resources Canada, Ottawa, Ontario.

W. May, D.A. Becker, Rolf Zeisler, R.R. Greenberg and S. Wise, National Institute of Standards and Technology, Gaithersburg, Maryland.

R. Parker, Department of Geology, University of Auckland, New Zealand

C. Davies, Z. Huang and P. Moore Brooks Rand Ltd., Seattle, Washington.

Alan Jeffrey, John Stewart, Peter Lynch, Helen Walker, John Drinnan and Glenn Barry, Department of Natural Resources Indooroopilly, Queensland, Australia.

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Issac Pereiro and Ryszard Lobinski, Laboratoire de Chimie Bio-Inorganique et Environnement, Pau, France.

HISS-1 issued February 1997
MESS-3 issued January 2000
PACS-2 issued August 1997

last revision January 2000

Visit our WWW site
http://www.cm.inms.nrc.ca

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Metals in Sediment
Standard Reference Material Recovery
MESS-3
(Concentration in µg/g dry weight)

ICP-MS Data Set ID	Central File No.	Project	Cr
032702-5000a	CF 1768	ASI-American Sugar Refining	78.9
101501-6100A	CF 1698	Kill van Kull	103
112001-5000A	CF 1692	Boston Harbor	87.9
112001-5000A	CF 1721	Three Sites in CT	83.5
072501-5000A	CF 1677	Jacksonville-Yabucoa	94.7
072501-5000A	CF 1677	Jacksonville-Yabucoa	102
101501-6100A	CF 1693	ASI-Navy-Maguire (Earle)	106
MEAN LAB-ACHIEVED CONCENTRATION			**93.7**
STANDARD DEVIATION			10.6
SRM MESS-3 Certified Value			105
Range			±4.00
Percent Difference			**11%**

Metals in Sediment
Standard Reference Material Recovery
PACS-2
(Concentration in µg/g dry weight)

ICP-MS Data Set ID	Central File No.	Project	Cr
032702-5000a	CF 1768	ASI-American Sugar Refining	56.7
101501-6100A	CF 1698	Kill van Kull	71.8
112001-5000A	CF 1692	Boston Harbor	63.7
112001-5000A	CF 1721	Three Sites in CT	59.3
072501-5000A	CF 1677	Jacksonville-Yabucoa	69.3
072501-5000A	CF 1677	Jacksonville-Yabucoa	71.4
101501-6100A	CF 1693	ASI-Navy-Maguire (Earle)	78.8
MEAN LAB-ACHIEVED CONCENTRATION			**67.3**
STANDARD DEVIATION			7.78
SRM PACS-2 Certified Value			90.7
Range			±4.6
Percent Difference			**26%**

Certified Reference Material

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CASS-4

Nearshore Seawater Reference Material for Trace Metals

The following table shows the twelve metals for which certified values have been established. Certified values are based on the results of determinations by at least two independent methods of analysis. Each uncertainty is based on a 95 percent confidence limit for the mean.

Trace Metal Concentrations
(micrograms/litre)

Arsenic (f,h,j,r)	1.11	±	0.16
Cadmium (c,i,p,r,s)	0.026	±	0.003
Chromium (i,p)	0.144	±	0.029
Cobalt (f,i,q)	0.026	±	0.003
Copper (i,p,r,s)	0.592	±	0.055
Iron (i,p,s)	0.713	±	0.058
Lead (c,i,p,s)	0.0098	±	0.0036
Manganese (f,i,q,s)	2.78	±	0.19
Molybdenum (d,p)	8.78	±	0.86
Nickel (c,i,p,r,s)	0.314	±	0.030
Uranium* (p)	3.0		
Vanadium(f,q)	1.18	±	0.16
Zinc (f,i,p,s)	0.381	±	0.057

*information value only

c - Chelation-solvent extraction separation, inductively coupled plasma mass spectrometry (ICPMS)
d - Direct determination by graphite furnace atomic absorption spectrometry (GFAAS)
f - Reductive precipitation separation, ICPMS determination
h - hydride generation, electrothermal vaporization ICPMS determination
i - Immobilized ligand separation, GFAAS determination
j - Hydride generation, atomic absorption determination
p - Immobilized ligand separation, isotope dilution ICPMS
q - Immobilized ligand separation, ICPMS
r - Reductive precipitation separation, GFAAS determination
s - Chelation-solvent extraction separation, GFAAS determination

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DETERMINATION OF LABORATORY CONSENSUS VALUES
Metals in SRM CASS-4

(concentrations in µg/L - not blank corrected)

	RUN ID		Cd	Cr	Cu	Ni	Pb	Zn
	Navy-Earle (ASI/Maguire)							
1.	CASS-4 R1	ICP-MS: 091201-5000A; GFAA: T092301A-Ag	0.0216	0.248	0.787	0.484	0.0186	0.412
2.	CASS-4 R2	ICP-MS: 091201-5000A; GFAA: T092301A-Ag	0.0208	0.257	0.798	0.509	0.0144	0.433
	USACE-NED Boston Harbor							
3.	CASS 4-1	ICP-MS: 091301-5000A	0.0322	0.193	0.888	0.362	0.0163	0.467
4.	CASS 4-2	ICP-MS: 091301-5000A	0.0329	0.192	0.816	0.368	0.016 U	0.466
	USACE-NYD Kill van Kull - CF 1698							
5.	CASS-4 R1	ICP-MS: 102901-5000A; GFAA: T110201A-Ag	0.0214	0.256	0.657	0.374	0.0122	0.410
6.	CASS-4 R2	ICP-MS: 102901-5000A; GFAA: T110201A-Ag	0.0221	0.241	0.645	0.352	0.0113	0.391
	USACE-NED Bridgeport - CF 1720							
7.	CASS-4 r1	ICP-MS: 111901-5000A	0.0236	0.201	0.655	0.423	0.016 U	0.390
8.	CASS-4 r2	ICP-MS: 111901-5000A	0.0209	0.199	0.659	0.331	0.016 U	0.386
	USACE-NED Three Sites in Connecticut - CF 1721							
9.	CASS-4 r1	112001-5000A	0.0256	0.234	0.706	0.414	0.016 U	0.430
10.	CASS-4 r2	112001-5000A	0.0253	0.227	0.689	0.368	0.016 U	0.438
11.	CASS-4 r3	112701-6100A	0.0258	0.201	0.651	0.352	0.0146	0.381
12.	CASS-4 r4	112701-6100A	0.0218	0.216	0.681	0.35	0.0132	0.420
	Rhode Island Sound Disposal Site Study - CF 1722							
13.	CASS-4 r1	ICP-MS: 121201-5000A; GFAA-Ag	0.0341	0.232	0.594	0.351	0.0397	0.477
14.	CASS-4 r2	ICP-MS: 121201-5000A; GFAA-Ag	0.0393	0.221	0.624	0.349	0.0399	0.447
	American Sugar Refining (ASI) - CF 1768							
15.	CASS-4 r1	ICP-MS: 032002-5000A; GFAA-Ag	0.0386	0.234	0.616	0.490	0.0398	0.322
16.	CASS-4 r2	ICP-MS: 032002-5000A; GFAA-Ag	0.0406	0.226	0.619	0.464	0.0401	0.321

		Cd	Cr	Cu	Ni	Pb	Zn
MEAN	**(Laboratory Consensus Value)**	**0.0279**	**0.224**	**0.693**	**0.396**	**0.0214**	**0.412**
STD DEVIATION	**±**	**0.007**	**0.02**	**0.08**	**0.06**	**0.01**	**0.05**

CASS-4 Certified (or Reference) Value	0.026	0.144	0.592	0.314	0.0098	0.381
Range	±0.003	±0.029	±0.055	±0.03	±0.0036	±0.057
Percent Difference from Certified Value	**7%**	**55%**	**17%**	**26%**	**118%**	**8%**

(Laboratory-achieved results over 16 analyses of CASS-4)

Determination of Laboratory Consensus Value for Concentration of Ag in SRM CASS-4

GFAA Run ID	Sample ID	Concentration Ag (µg/L)
T022201A	CASS-4 R1	0.0193
T022201A	CASS-4 R2	0.0161
T061801A	CASS-4	0.0194
T062501A	CASS-4	0.0303
T081501A	CASS-4	0.0240
T110201A	CASS-4 R1	0.0274
T110201A	CASS-4 R2	0.0204
		0.0224 MEAN
		0.00504 STDEV

Attachment to Miscellaneous Documentation
42836-D-003

1640 Trace elements in Natural Water

Statistical analysis of the experimental data was performed by W.F. Guthrie of the NIST Statistical Engineering Division.

Coordination of the NIST technical measurements was under the direction of J.R. Moody of the NIST Analytical Chemistry Division.

The overall coordination of measurements performed by the U.S. Geological Survey National Water Quality Laboratory, Arvada, CO, and by laboratories that participate in the Standard Reference Water Program was under the direction of K. Long.

Recognizing contamination at the microgram per kilogram level can be a serious problem, labware should be scrupulously cleaned and only high purity reagents employed. Sampling and manipulations, such as evaporations, should be done in a clean environment, such as a Class-100 clean hood.

Table 1. Certified Mass Fractions

Element	µg/kg			Element	µg/kg		
Aluminum	52.0	±	1.5	Iron	34.3	±	1.6
Antimony	13.79	±	0.42	Lead	27.89	±	0.14
Arsenic	26.67	±	0.41	Manganese	121.5	±	1.1
Barium	148.0	±	2.2	Molybdenum	46.75	±	0.26
Beryllium	34.94	±	0.41	Selenium	21.96	±	0.51
Boron	301.1	±	6.1	Silver	7.62	±	0.25
Cadmium	22.79	±	0.96	Strontium	124.2	±	0.7
Chromium	38.6	±	1.6	Vanadium	12.99	±	0.37
Cobalt	20.28	±	0.31				

Table 2. Reference Mass Fractions

Element	µg/kg			Element	mg/kg		
Copper	85.2	±	1.2	Calcium	7.045	±	0.089
Lithium	50.7	±	1.4	Magnesium	5.819	±	0.056
Nickel	27.4	±	0.8	Silicon	4.73	±	0.12
Potassium	994	±	27	Sodium	29.35	±	0.31
Rubidium	2.00	±	0.02				
Zinc	53.2	±	1.1				

Table 3. Information Mass Fraction

Thallium	<0.1 µg/kg

Source and Preparation of Material: A sample of about 3500 L of natural (fresh) water was obtained by the USGS at Clear Creek, CO. It was filtered through a 0.1 µm ultra filter and acidified with nitric acid. Analysis of the water by inductively coupled plasma mass spectrometry (ICPMS), before and after the stabilization process, showed that arsenic, beryllium, cobalt, selenium, and zinc were decreased in concentration during the stabilization process. These elements were adjusted to their original concentration levels by the addition of salts of the decreased elements. The stabilized solution was then pumped through an ultra filter, past a UV light source (for sterilization purposes), and then to a bottling station. At the bottling station, the bottles were rinsed with the sample and then filled.

Certified Reference Material

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Recertified Hg

DORM-2 DOLT-2

Dogfish Muscle and Liver Certified Reference Materials for Trace Metals

The following table shows those elements for which certified values have been established for the two dogfish (*Squalus acanthias*) reference materials. Certified values are based on results of determinations by at least two independent methods of analysis. The uncertainties represent 95 percent tolerance limits for an individual sub-sample of 250 mg or greater.

Trace Elements - (milligrams/kilogram)

	DORM-2			DOLT-2		
Aluminum (d,g,i)†	10.9	±	1.7	25.2	±	2.4
Arsenic (d,g,h,x)	18.0	±	1.1	16.6	±	1.1
Cadmium (g,p)	0.043	±	0.008	20.8	±	0.5
Cobalt (d,g)	0.182	±	0.031	0.24	±	0.05
Chromium (g,i,p)	34.7	±	5.5	0.37	±	0.08
Copper (g,i,p,x)	2.34	±	0.16	25.8	±	1.1
Iron (g,i,p,x)	142	±	10	1103	±	47
Lead (g,p)	0.065	±	0.007	0.22	±	0.02
Manganese (d,g,i)	3.66	±	0.34	6.88	±	0.56
Mercury (c,p)	4.64	±	0.26	2.14	±	0.28
Nickel (g,i,p)	19.4	±	3.1	0.20	±	0.02
Selenium (g,p)	1.40	±	0.09	6.06	±	0.49
Silver (g,p)	0.041	±	0.013	0.608	±	0.032
Thallium (p)	(0.004)*				---	
Tin (p)	(0.023)*			(0.13)*		
Zinc (f,g,i,p)	25.6	±	2.3	85.8	±	2.5
Methylmercury (as Hg) (e,t)	4.47	±	0.32	0.693	±	0.053

† - see next page for key to coding.
* - information value only.

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National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1566b

Oyster Tissue

This Standard Reference Material (SRM) is intended primarily for use in evaluating analytical methods and instruments used for the determination of the concentrations of selected elements and proximates, selected fatty acids, total dietary fiber, as well as the caloric content in marine bivalve tissue, foods, or similar materials. A unit of SRM 1566b contains approximately 25 g of freeze-dried oyster tissue.

Certified Concentrations of Constituent Elements: Certified values for 22 elements and for methylmercury are provided in Table 1. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been fully investigated or accounted for by NIST. Certified values are based on results obtained by a single primary method with confirmation by other methods, or with two or more critically evaluated independent methods [1]. The certified value for sulfur is the result of a single NIST method. All other certified values are weighted means of results obtained using two or more independent methods.

Reference Concentration Values: Reference values are noncertified values that are the best estimates of the true values; however, the values do not meet NIST criteria for certification. Such values are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods. Reference values for elemental concentrations are provided in Table 2. Reference values for proximates, nitrogen, total dietary fiber, selected fatty acids, and caloric content are provided in Table 3.

Information Concentration Values: Information concentration values for selected fatty acids are provided in Table 4. These are noncertified values with no reported uncertainties as there is insufficient information to assess uncertainties. The information values are given to provide additional characterization of the material, and are not recommended for use to monitor or assess analytical performance.

Expiration of Certification: The certification of this SRM lot is valid until **01 June 2010**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification is nullified if the SRM is contaminated or modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of certification. If substantive changes occur that affect the certification prior to the expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

The coordination of the technical measurements leading to the certification of this SRM was performed by R.R. Greenberg of the NIST Analytical Chemistry Division. Coordination of technical measurements of organic compounds and methylmercury leading to certified, reference, and information values was performed by K.E. Sharpless and S.A. Wise of the NIST Analytical Chemistry Division.

The NIST analysts and cooperating laboratories that participated in the characterization of this SRM are listed in Appendix A.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by J.C. Colbert.

Willie E. May, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 17 January 2001

Nancy M. Trahey, Chief
Standard Reference Materials Program

Preparation of the oyster tissue material was performed by B.J. Porter of the NIST Analytical Chemistry Division and M.P. Cronise, C.N. Fales, and D.G. Friend of the NIST Standard Reference Materials Program.

The statistical analysis of the data was performed by J.H. Yen, L.M. Gill, and M.S. Levenson of the NIST Statistical Engineering Division.

NOTICE AND WARNING TO USERS

Storage: The material should be kept in its original bottle, tightly closed, and stored in a desiccator over magnesium perchlorate $Mg(ClO_4)_2$, at temperatures between 10 °C to 30 °C. It should **NOT** be exposed to intense sources of radiation, including ultraviolet light or sunlight.

Use: A minimum sample mass of 250 mg of material is necessary for any certified value in Table 1 to be valid within the stated uncertainty. This amount of material should be on a dry-mass basis (see Instructions for Drying). The contents of the bottle should be shaken well before each use, closed tightly **immediately** after use, and stored as described above.

Instructions for Drying: Prior to removal of subsamples for elemental analysis, the contents of the bottle should be thoroughly mixed. Before the mass determination, samples of SRM 1566b must be dried to constant mass by one of the following procedures:

1. Drying at room temperature for *at least* 5 d over $Mg(ClO_4)_2$ in a desiccator.
2. Vacuum drying at room temperature for *at least* 24 h at a pressure of approximately 30 Pa (0.2 mm Hg) using a cold trap.
3. Freeze drying for *at least* 5 d at a pressure of approximately 30 Pa (0.2 mm Hg).

The analyst should ascertain that the material has indeed reached constant mass. Although the above procedures have been generally sufficient, in a few instances the time needed to reach constant mass was longer than listed above. If the constituents of interest are volatile, a separate subsample of the oyster tissue should be removed from the bottle at the time of analysis and dried to determine the concentration on a dry-mass basis.

SOURCE, PREPARATION, AND ANALYSIS[1]

Source and Preparation of Material: The oysters (*Crassostrea virginica*, an American Eastern oyster) used for the preparation of SRM 1566b were purchased from Bon Secour Fisheries, Inc., Bon Secour, AL. The oysters were collected from the Gulf of Mexico, shucked, rinsed twice to remove sediment and shells, packed and sealed in polyethylene bags, and frozen. The frozen oysters and fluids were shipped in styrofoam coolers containing dry ice to NIST. At NIST, the oysters and fluids were ground in a Robot-Coupe Vertical Cutter Mixer that was equipped with a stainless steel bowl and titanium blades. The oyster tissue was blended for 100 s into a slurry; approximately 5 kg of slurry was poured into each of 40 specially cleaned aluminum trays outfitted with temperature probes, and frozen at -20 °C. The trays were taken to a large freeze-drying facility at the Frederick Cancer Research and Development Center, Natural Products Group in Frederick, MD. The freeze-dryer's initial temperature was -45 °C and gradually increased to a temperature of 10 °C over a period of five days. The freeze-dried material was stored at -20 °C, then broken into smaller pieces, blended in the Robot-Coupe Mixer, jet milled, and homogenized in a V-blender for 30 min to 40 min. The material was radiation sterilized (^{60}Co) at Neutron Products, Inc., Dickerson, MD, for approximately 5 h at 3 Mrad and then aliquoted into amber bottles.

Description of Calculations Used in Value Assignment

A. Certified Values and Their Uncertainties

Sulfur

The certified value of sulfur is the result of a single NIST method, thermal ionization mass spectrometry (TIMS), with confirmation by a second NIST method. Its uncertainty is expressed as an expanded uncertainty, U, and is calculated as $U = ku_c$. The quantity, u_c, is the combined standard uncertainty calculated according to the ISO Guide [2] that accounts for the combined components of uncertainty for the method at one standard deviation. The coverage factor, k, is determined from the Student's t-distribution corresponding to five degrees of freedom and a 95 % prediction interval.

[1]Certain commercial equipment, instrumentation, or materials are identified in this certificate to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Methylmercury

The results for methylmercury are expressed as mg/kg mercury. The certified value is the mean of results from four different laboratory analyses of SRM 1566b using four different analytical methods. The expanded uncertainty in the certified value is equal to $U = ku_c$ where u_c is the combined standard uncertainty calculated according to the ISO Guide [2] and k is the coverage factor. The value, u_c, is intended to represent, at the level of one standard deviation, the combined effect of all the uncertainties in the certified value. Here, u_c is given by the standard error of the mean of the four analyses. The coverage factor, k, is determined from the Student's t-distribution corresponding to three degrees of freedom and 95 % confidence for each analyte.

All Other Elements

All other certified values are weighted means of results from two or more analytical methods. For these certified values, the uncertainty is calculated as $U = ku_c + B$. The quantity, u_c, is the combined standard uncertainty calculated according to the ISO Guide [2], which accounts for the combined effect of the within variance for all methods at one standard deviation. The coverage factor, k, is determined from the Student's t-distribution corresponding to the appropriate associated degrees of freedom and 95 % confidence for each element. The term, B, is a bias adjustment for the difference between methods which is the maximum difference between the certified value and the method means [3]. Because of heterogeneity, the uncertainty associated with calcium and thorium takes the form of a prediction interval.

Table 1. Certified Concentration Values of SRM 1566b[1]

Element	Mass Fraction (%)			Element	Mass Fraction (%)		
Calcium[c,g]	0.0838	±	0.0020	Potassium[g,i,k]	0.652	±	0.009
Chlorine[g,i]	0.514	±	0.010	Sodium[b,g]	0.3297	±	0.0053
Magnesium[c,g]	0.1085	±	0.0023	Sulfur[d,j]	0.6887	±	0.0140

Element	Mass Fraction (mg/kg)			Element	Mass Fraction (mg/kg)		
Aluminum[g,l]	197.2	±	6.0	Mercury[a,n] (total)	0.0371	±	0.0013
Arsenic[g,j]	7.65	±	0.65	Methylmercury[o,p,q,r] (as mercury)	0.0132	±	0.0007
Cadmium[e,h]	2.48	±	0.08	Nickel[e,j,k]	1.04	±	0.09
Cobalt[e,g]	0.371	±	0.009	Rubidium[e,g]	3.262	±	0.145
Copper[e,g,h]	71.6	±	1.6	Selenium[g,j]	2.06	±	0.15
Iron[g,k,m]	205.8	±	6.8	Silver[e,g]	0.666	±	0.009
Lead[c,e]	0.308	±	0.009	Thorium[c,g]	0.0367	±	0.0043
Manganese[e,g]	18.5	±	0.2	Vanadium[e,g]	0.577	±	0.023
				Zinc[g,k]	1 424	±	46

[1] Dry-mass basis

[2] Analytical Methods:

[a] Cold vapor atomic absorption spectrometry at NIST
[b] Flame atomic emission spectrometry at NIST
[c] Isotope dilution inductively coupled plasma mass spectrometry at NIST
[d] Isotope dilution thermal ionization mass spectrometry at NIST
[e] Inductively coupled plasma mass spectrometry at NIST
[f] Inductively coupled plasma optical emission spectrometry at NIST
[g] Neutron activation analysis (instrumental) at NIST
[h] Neutron activation analysis (radiochemical) at NIST
[i] Prompt gamma activation analysis at NIST
[j] Electrothermal atomic absorption spectrometry at National Research Council of Canada (NRCC)
[k] Isotope dilution inductively coupled plasma mass spectrometry at NRCC
[l] Inductively coupled plasma mass spectrometry at NRCC
[m] Inductively coupled plasma optical emission spectrometry at NRCC
[n] Neutron activation analysis (radiochemical) at Jo ef Stefan Institute, Ljubljana, Slovenia
[o] Gas chromatography with atomic emission detection at NIST
[p] Gas chromatography with atomic fluorescence detection at Jo ef Stefan Institute, Ljubljana, Slovenia
[q] Cold vapor atomic absorption spectrometry at Jo ef Stefan Institute, Ljubljana, Slovenia
[r] Cold vapor atomic absorption spectrometry at Research Centre Jülich, Jülich, Germany

Table 2. Reference Concentration Values of SRM 1566b[1]

These concentrations are provided as reference values because the results have not been confirmed by an independent analytical technique as required for certification; therefore unrecognized bias may exist for some measurands in this matrix.

Element	Mass Fraction (%)
Nitrogen[a]	7.6 ± 0.4

Element	Mass Fraction (mg/kg)	Element	Mass Fraction (mg/kg)
Antimony[b]	0.011 ± 0.002	Strontium[c]	6.8 ± 0.2
Barium[d]	8.6 ± 0.3	Tin[d]	0.031 ± 0.008
Boron[a]	4.5 ± 1.9	Uranium[d]	0.2550 ± 0.0014
Hydrogen[a]	7.2 ± 0.4		

[1] Dry-mass basis

[2] Analytical Methods:

[a] Prompt gamma activation analysis at NIST
[b] Neutron activation analysis (instrumental) at NIST
[c] Inductively coupled plasma mass spectrometry at NIST
[d] Isotope dilution inductively coupled plasma mass spectrometry at NRCC, Ottawa, Canada

Table 3. Reference Concentration Values of Proximates, Nitrogen, Total Dietary Fiber, Selected Fatty Acids (as Triglycerides) and Calories in SRM 1566b

These concentrations are provided as reference values because the results have not been confirmed by an independent analytical technique as required for certification; therefore unrecognized bias may exist for some measurands in this matrix.

Constituent	Mass Fraction as received (%)[a]	Mass Fraction dry-mass basis (%)[a]
Moisture	4.6 ± 3.6	0 (by definition)
Solids	95.4 ± 3.6	100 (by definition)
Carbohydrates	43.4 ± 3.2	45.4 ± 1.7
Ash	3.87 ± 0.09	4.05 ± 0.15
Fat	5.5 ± 1.2	5.8 ± 1.1
Protein[b]	42.6 ± 1.3	44.7 ± 2.6
Protein Nitrogen[b]	6.82 ± 0.20	7.16 ± 0.42
Total Dietary Fiber	6.5 ± 1.6	6.8 ± 1.4
Caloric Content[c]	(394 ± 20) kcal/100 g	(412.7 ± 5.9) kcal/100 g
Tetradecanoic Acid (C14:0) (Myristic Acid)	0.403 ± 0.075	0.421 ± 0.057
Pentadecanoic Acid (C15:0)	0.079 ± 0.016	0.083 ± 0.013
Hexadecanoic Acid (C16:0) (Palmitic Acid)	2.16 ± 0.20	2.27 ± 0.15
(Z)-9-Hexadecenoic Acid (C16:1) (Palmitoleic Acid)	0.362 ± 0.063	0.379 ± 0.064
Heptadecanoic Acid (C17:0) (Margaric Acid)	0.168 ± 0.022	0.178 ± 0.023
Octadecanoic Acid (C18:0) (Stearic Acid)	0.424 ± 0.054	0.444 ± 0.041
(Z)-9-Octadecenoic Acid (C18:1) (Oleic Acid)	0.86 ± 0.20	0.90 ± 0.21
(all-Z)-5,8,11,14, 17-Eicosapentaenoic Acid (C20:5) (Timnodonic Acid)	0.065 ± 0.012	0.068 ± 0.014

[a] Each reference value, expressed as a mass fraction of the material on an as-received or dry-mass basis, for the above measurands is an equally weighted mean of results from the cooperating laboratories listed in Appendix A. Each of four laboratories analyzed one portion from each of three bottles of SRM 1566b using their routine methods. Determinations were performed on the material "as received," with conversion of results to a dry mass basis using moisture values determined by the four laboratories on separate subsamples taken from each of the three bottles. The uncertainty in the reference values is expressed as an expanded uncertainty, U, at the 95 % level of confidence, and is calculated according to the method described in the ISO Guide [2]. The expanded uncertainty is calculated as, $U = ku_c$, where, u_c, is intended to represent, at the level of one standard deviation, the combined effect of between-laboratory and within-laboratory components of uncertainty. The coverage factor, k, is determined from the Student's t-distribution corresponding to the appropriate associated degrees of freedom and 95 % confidence for each analyte. Analytical methodology information is provided in Table 5.

[b] The protein concentration was calculated from the nitrogen values reported by the laboratories using a conversion factor of 6.25. The value for protein is the mean of the individual protein calculations. If the mean nitrogen values above are used for calculation, the mean protein concentrations are 42.6 % and 44.7 % on a wet mass and dry mass basis, respectively.

[c] The value for caloric content is the mean of the individual caloric calculations. If the mean proximate values are used for calculation, with caloric equivalents of 9, 4, and 4 for fat, protein, and carbohydrate, respectively, the mean caloric content is 394 kcal/100 g and 412.7 kcal/100 g on an as-received and dry-mass basis, respectively.

Table 4. Information Concentration Values of Selected Fatty Acids (as Triglycerides)

These concentrations are provided as information values only because the disagreement among the results was greater than expected for reference values or because results were reported by a limited number of laboratories. The data for these information values are not of sufficient quality or quantity to adequately assign uncertainties.

Fatty Acid	Mass Fraction, as received (%)[a]	Mass Fraction, dry-mass basis (%)[b]
(E)-9-Octadecenoic Acid (C18:1) (Elaidic Acid)	0.16	0.17
(Z,Z)-9,12-Octadecadienoic Acid (C18:2) (Linoleic Acid)	0.097	0.10
(all Z)-5,8,11,14-Eicosatetraenoic Acid (C20:4) (Arachidonic Acid)	0.029	0.031
Docosanoic Acid (C22:0) (Behenic Acid)	0.017	0.018
Tetracosanoic Acid (C24:0) (Lignoceric Acid)	0.022	0.023

[a] Each information value, reported on an as-received and dry-mass basis, is the equally weighted mean of results reported by the laboratories shown in Appendix A. These values are based on results from determinations by two to four of the laboratories and are included to provide additional characterization of the material; no uncertainties are provided. Analytical methodology information is provided in Table 5.

[b] Determinations were performed on the material "as received," with conversion of results to a dry-mass basis using moisture values determined by the four laboratories on separate subsamples taken from each of three bottles.

Table 5. Analytical Methods Used by Collaborating Laboratories for the Determination of Proximates, Fatty Acids, Calories, and Total Dietary Fiber in SRM 1566b

Ash	mass loss after ignition in a muffle furnace
Calories	calculated; [(9 × fat) + (4 × protein) + (4 × carbohydrate)]
Carbohydrate	calculated; [solids – (protein + fat + ash)]
Fat	sum of individual fatty acids
Fatty acids	hydrolysis followed by gas chromatography
Moisture	mass loss after drying in a vacuum oven (3 laboratories) or forced-air oven (1 laboratory)
Protein nitrogen	Dumas (1 laboratory); modified Dumas (1 laboratory); Kjeldahl (2 laboratories)
Protein	calculated from nitrogen using a factor of 6.25
Solids	calculated; (sample mass – moisture)
Total dietary fiber	enzymatic gravimetry

REFERENCES

[1] May, W., Parris, R., Beck, C., Fassett, J., Greenberg, R., Guenther, F., Kramer, G., Wise, S., Gills, T., Colbert, J., Gettings, R., and MacDonald, B., "Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements," NIST Special Publication 260-136, January 2000; available at http://www.cstl.nist.gov/nist 839/srminfo.html.

[2] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland, 1993; see also Taylor, B.N. and Kuyatt, C.E., Guidelines for Evaluating and Expressing Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington DC, (1994); available at http://physics.nist.gov/Pubs/.

[3] Schiller, S. and Eberhardt, K., "Combining Data from Independent Chemical Analysis Methods," Spectrochimica Acta, 46B, No. 12, pp. 1607-1613, (1991).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.

Appendix A

NIST Analysts, Analytical Chemistry Division, National Institute of Standards and Technology

B. Buehler
T.A. Butler
R. Demiralp
M.S. Epstein
M.E. Howard
W.R. Kelly
R.M. Lindstrom
S.E. Long
E.A. Mackey
J.L. Mann
K.E. Murphy
M.S. Rearick
S. Tutschku
R.D. Vocke, Jr.
L.L. Yu

Collaborating Laboratories and Analysts for Elemental Determinations

Analytical Chemistry Division, National Research Council of Canada, Ottawa, Canada, V. Boyko, J. Lam, S. Willie, and L. Yang
Jo ef Stefan Institute, Ljubljana, Slovenia - H. Akagi, V. Fajon, M. Horvat, K. Jereb, and M. Logar
Research Center Jülich, Jülich, Germany - H. Emons and K. May

Collaborating Laboratories for Proximates, Fatty Acids, Total Dietary Fiber, and Calorie Determinations

Covance Laboratories, Madison, WI, USA
Lancaster Laboratories, Lancaster, PA, USA
Medallion Laboratories, Minneapolis, MN, USA
Southern Testing and Research Laboratories, Wilson, NC, USA

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1944

020111-1

New York/New Jersey Waterway Sediment

Standard Reference Material (SRM) 1944 is a mixture of marine sediment collected near urban areas in New York and New Jersey. SRM 1944 is intended for use in evaluating analytical methods for the determination of selected polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners, chlorinated pesticides, and trace elements in marine sediment and similar matrices. Reference values are also provided for selected dibenzo-*p*-dioxin and dibenzofuran congeners, total organic carbon, total extractable material, and particle-size characteristics. All of the constituents for which certified, reference, and information values are provided in SRM 1944 were naturally present in the sediment material before processing. A unit of SRM 1944 consists of a bottle containing 50 g of radiation sterilized, freeze-dried sediment material.

Certified Concentration Values: Certified values for concentrations, expressed as mass fractions, for 24 PAHs, 35 PCB congeners (some in combination), four chlorinated pesticides, and nine trace elements are provided in Tables 1-4. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST. The certified values for the PAHs, PCB congeners, and chlorinated pesticides are based on the agreement of results obtained at NIST from two or more chemically independent analytical techniques. The certified values for the trace elements are based on NIST measurements by one technique and additional results from several collaborating laboratories.

Reference Concentration Values: Reference values for concentrations, expressed as mass fractions, are provided for 32 additional PAHs (some in combination) in Table 5, seven additional chlorinated pesticides in Table 6, and 19 additional inorganic constituents in Tables 7 and 8. Reference values are provided in Table 9 for the 17 2,3,7,8-substituted polychlorinated dibenzo-*p*-dioxin and dibenzofuran congeners and total tetra-, penta-, hexa-, and hepta-congeners of polychlorinated dibenzo-*p*-dioxin and dibenzofuran. Reference values for particle-size characteristics are provided in Table 10. Reference values for total organic carbon and percent extractable mass are provided in Table 11. Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods. Explanations in support of each reference value are given as notes in Tables 5-11.

Information Concentration Values: Information values for concentrations, expressed as mass fractions, are provided in Table 12 for eight additional trace elements. An information value is considered to be a value that will be of interest and use to the SRM user, but insufficient information is available to assess the uncertainty associated with the value or only a limited number of analyses were performed.

Expiration of Certification: The certification of this SRM lot is valid until **31 March 2009**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification is nullified if the SRM is damaged, contaminated, or modified.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by B.S. MacDonald.

Willie E. May, Chief
Analytical Chemistry Division

Thomas E. Gills, Director
Office of Measurement Services

Gaithersburg, MD 20899
Certificate Issue Date: 14 May 1999

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

The coordination of the technical measurements leading to the certification was under the leadership of S.A. Wise of the NIST Analytical Chemistry Division.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by M.G. Vangel and M.S. Levenson of the NIST Statistical Engineering Division.

The sediment material was collected with the assistance of the New York District of the U.S. Army Corp of Engineers (ACENYD), who provided the expertise in the site selection, the ship, sampling equipment, and personnel. L. Rosman of ACENYD and R. Parris (NIST) coordinated the collection of this sediment material. Collection and preparation of SRM 1944 were performed by R. Parris, M. Cronise, and C. Fales (NIST); L. Rosman and P. Higgins (ACENYD); and the crew of the *Gelberman* from the ACE Caven Point facility in Caven Point, NJ.

Analytical measurements for the certification of SRM 1944 were performed at NIST by E.S. Beary, D.A. Becker, R. Demiralp, R.R. Greenberg, M. Lopez de Alda, K.E. Murphy, B.J. Porter, D.L. Poster, L.C. Sander, M.M. Schantz, and L. Walton of the Analytical Chemistry Division. Measurements for percent total organic carbon measurements were provided by three commercial laboratories and T.L. Wade of the Geochemical and Environmental Research Group, Texas A&M University (College Station, TX). The particle-size distribution data were provided by Honeywell, Inc. (Clearwater, FL).

Analytical measurements for the polychlorinated dibenzo-*p*-dioxins and dibenzofurans were the results of an interlaboratory comparison study among 14 laboratories (see Appendix A) coordinated by S.A. Wise of the NIST Analytical Chemistry Division and R. Turle and C. Chiu of Environment Canada, Environmental Technology Centre, Analysis and Air Quality Division (Ottawa, Ontario, Canada). Analytical measurements for selected trace elements were provided by the International Atomic Energy Agency (IAEA, Seibersdorf, Austria) by M. Makarewicz and R. Zeisler. Results were also used from seven laboratories (see Appendix B) that participated in an intercomparison exercise coordinated by S. Willie of the Institute for National Measurement Standards, National Research Council Canada (NRCC, Ottawa, Ontario, Canada).

NOTICE AND WARNING TO USERS

Storage: SRM 1944 must be stored in its original bottle at temperatures less than 30 °C away from direct sunlight.

Handling: This material is naturally occurring marine sediment from an urban area and may contain constituents of unknown toxicities; therefore, caution and care should be exercised during its handling and use.

Instructions for Use: Prior to removal of subsamples for analysis, the contents of the bottle should be mixed. The concentrations of constituents in SRM 1944 are reported on a dry-mass basis. The SRM, as received, contains approximately 1.3 % moisture. The sediment sample should be dried to a constant mass before weighing for analysis, or if the constituents of interest are volatile, a separate subsample of the sediment should be removed from the bottle at the time of analysis and dried to determine the concentration on a dry-mass basis.

PREPARATION AND ANALYSIS[1]

Sample Collection and Preparation: The sediment used to prepare this SRM was collected from six sites in the vicinity of New York Bay and Newark Bay in October 1994. Site selection was based on contaminant levels measured in previous samples from these sites and was intended to provide relatively high concentrations for a variety of chemical classes of contaminants. The sediment was collected using an epoxy-coated modified Van Veen-type grab sampler designed to sample the sediment to a depth of 10 cm. A total of approximately 2100 kg

[1]Certain commercial equipment, instrumentation, or materials are identified in this report to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

of wet sediment was collected from the six sites. The sediment was freeze-dried, sieved (nominally 250 µm to 61 µm), homogenized in a cone blender, radiation sterilized (^{60}Co), and then packaged in screw-capped amber glass bottles.

Conversion to Dry-Mass Basis: The results for the constituents in SRM 1944 are reported on a dry-mass basis; however, the material "as received" contains residual moisture. The amount of moisture in SRM 1944 was determined by measuring the mass loss after freeze-drying subsamples of 1.6 g to 2.5 g for five days at 1 Pa with a -10 °C shelf temperature and a -50 °C condenser temperature. The moisture content in SRM 1944 at the time of the certification analyses was 1.25 % ± 0.03 % (95 % confidence level).

Polycyclic Aromatic Hydrocarbons: The general approach used for the value assignment of the PAHs in SRM 1944 was similar to that reported for the recent certification of several environmental matrix SRMs [1-5] and consisted of combining results from analyses using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques. This approach consisted of Soxhlet extraction and pressurized fluid extraction (PFE) using dichloromethane (DCM) or a hexane/acetone mixture, cleanup of the extracts using solid phase extraction (SPE) or normal-phase liquid chromatography (LC), followed by analysis using the following techniques: (1) reversed-phase liquid chromatography with fluorescence detection (LC-FL) for analysis of the total PAH fraction, (2) reversed-phase LC-FL analysis of isomeric PAH fractions isolated by normal-phase LC (i.e., multidimensional LC), (3) gas chromatography/mass spectrometric (GC/MS) analysis of the PAH fraction on three stationary phases of different selectivity, i.e., a 5 % (mole fraction) phenyl-substituted methylpolysiloxane phase, a 50 % (mole fraction) phenyl-substituted methylpolysiloxane phase, and a smectic liquid crystalline stationary phase.

Six sets of GC/MS results, designated as GC/MS (I), GC/MS (II), GC/MS (III), GC/MS (IV), GC/MS (V), and GC/MS (Sm), were obtained using three columns with different selectivities for the separation of PAHs. For GC/MS (I) analyses, duplicate subsamples of 1 g from eight bottles of SRM 1944 were Soxhlet extracted for 24 h with DCM. Copper powder was added to the extract to remove elemental sulfur. The concentrated extract was passed through a silica SPE cartridge and eluted with 2 % DCM in hexane. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. x 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-5 MS, J&W Scientific, Folsom, CA). The GC/MS (II) analyses were performed using 1 g to 2 g subsamples from three bottles of SRM 1944 and 2 g to 3 g subsamples from three bottles of SRM 1944 that had been mixed with a similar amount of water (i.e., a wetted sediment). These samples were Soxhlet extracted with DCM and processed through the silica SPE as described above; however, the extract was further fractionated using normal-phase LC on a semi-preparative aminopropylsilane column to isolate the PAH fraction [6-9]. The PAH fraction was then analyzed using the same column as described above for GC/MS (I); however, the subsamples were extracted, processed and analyzed as part of three different sample sets at different times using different calibrations for each set. For the GC/MS (III), 1 g to 2 g subsamples from six bottles of SRM 1944 were Soxhlet extracted for 18 h with 250 mL of a mixture of 50 % hexane/50 % acetone (volume fractions). The extracts were then processed and analyzed as described for GC/MS (II). For GC/MS (IV) analyses, 1 g to 2 g subsamples from six bottles of SRM 1944 were extracted using PFE with a mixture of 50 % hexane/50 % acetone as described by Schantz et al. [10], and the extracts were processed as described above for GC/MS (II). The GC/MS (V) results were obtained by analyzing three of the same PAH fractions that were analyzed in GC/MS (III) and three of the PAH fractions that were analyzed in GC/MS (IV) using a 50 % phenyl-substituted methylpolysiloxane stationary phase (0.25 mm i.d. x 60 m, 0.25 µm film thickness) (DB-17MS, J&W Scientific, Folsom, CA). For GC/MS (Sm) 1 g to 2 g subsamples from six bottles of SRM 1944 were Soxhlet extracted for 24 h with 250 mL of DCM. The extracts were processed as described above for GC/MS (I) using an aminopropylsilane SPE cartridge followed by GC/MS analysis using 0.2 mm i.d. x 25 m (0.15 µm film thickness) smectic liquid crystalline phase (SB-Smectic, Dionex, Lee Scientific Division, Salt Lake City, UT).

Two sets of LC-FL results, designated as LC-FL (Total) and LC-FL (Fraction), were used in the certification process. Subsamples of approximately 1 g from six bottles of SRM 1944 were Soxhlet extracted for 20 h using 200 mL of 50 % hexane/50 % acetone (volume fractions). The extracts were concentrated and then processed through two aminopropylsilane solid phase extraction (SPE) cartridges connected in series to obtain the total PAH fraction. A second 1 g subsample from the six bottles was Soxhlet extracted and processed as described above; the PAH fraction was then fractionated further on a semi-preparative aminopropylsilane column (µBondapak NH_2, 9 mm i.d. x 30 cm, Waters Associates, Milford, MA) to isolate isomeric PAH fractions as described previously [6-9]. The total PAH fraction and the isomeric PAH fractions were analyzed using a 5-µm particle-size polymeric octadecylsilane (C_{18}) column (4.6 mm i.d. x 25 cm, Hypersil-PAH, Keystone Scientific, Inc., Bellefonte, PA) with wavelength programmed fluorescence detection [7,8]. For all of the GC/MS and LC-FL measurements

described above, selected perdeuterated PAHs were added to the sediment prior to solvent extraction for use as internal standards for quantification purposes.

Homogeneity Assessment for PAHs: The homogeneity of SRM 1944 was assessed by analyzing duplicate samples of 1 g from eight bottles selected by stratified random sampling. Samples were extracted, processed, and analyzed as described above for GC/MS (I). No statistically significant differences among bottles were observed for the PAHs at the 1 g sample size.

PCBs and Chlorinated Pesticides: The general approach used for the determination of PCBs and chlorinated pesticides in SRM 1944 was similar to that reported for the recent certification of several environmental matrix SRMs [2,4,11,12,13], and consisted of combining results from analyses using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques. This approach consisted of Soxhlet extraction and PFE using DCM or a hexane/acetone mixture, cleanup/isolation using SPE or LC, followed by analysis using GC/MS and gas chromatography with electron capture detection (GC-ECD) on two columns with different selectivity.

Eight sets of results were obtained designated as GC-ECD (I) A and B, GC-ECD (II) A and B, GC/MS (I), GC/MS (II), GC/MS (III), and QA Exercise. For the GC-ECD (I) analyses, 1 g subsamples from four bottles of SRM 1944 were Soxhlet extracted with DCM for 18 h. Copper powder was added to the extract to remove elemental sulfur. The concentrated extract was passed through a silica SPE cartridge and eluted with 10 % DCM in hexane. The concentrated eluant was then fractionated on a semi-preparative aminopropylsilane column to isolate two fractions containing: (1) the PCBs and lower polarity pesticides, and (2) the more polar pesticides. GC-ECD analyses of the two fractions were performed on two columns of different selectivities for PCB separations: 0.25 mm x 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-5, J&W Scientific, Folsom, CA) and a 0.32 mm x 100 m fused silica capillary column with a 50 % (mole fraction) octadecyl (C-18) methylpolysiloxane phase (0.1 µm film thickness) (CPSil 5 C18 CB, Chrompack International, Middelburg, The Netherlands). The results from the 5 % phenyl phase are designated as GC-ECD (IA) and the results from the C-18 phase are designated as GC-ECD (IB). A second set of samples was also analyzed by GC-ECD (i.e., GC-ECD IIA and IIB). Subsamples of 1 g to 2 g from three bottles of SRM 1944 and 2 g to 3 g subsamples from three bottles of SRM 1944 that had been mixed with a similar amount of water (i.e., a wetted sediment) were extracted, processed, and analyzed as described above for GC-ECD (I); however, the subsamples were extracted, processed and analyzed as part of three different sample sets at different times using different calibrations for each set.

Three sets of results were obtained by GC/MS. For GC/MS (I), 1 g to 2 g subsamples from six bottles were Soxhlet extracted with a mixture of 50 % hexane/50 % acetone. Copper powder was added to the extract to remove elemental sulfur. The concentrated extract was passed through a silica SPE cartridge and eluted with 10 % DCM in hexane. The extract was then analyzed by GC/MS using a 0.25 mm x 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness). The GC/MS (II) results were obtained in the same manner as the GC/MS (I) analyses except that the six subsamples were extracted using PFE as described by Schantz et al. [10]. The GC/MS (III) analyses were performed on the same extract fractions analyzed in GC-ECD (II) using the 5 % phenyl-substituted methylpolysiloxane phase describe above for GC/MS (I). For both the GC-ECD and GC/MS analyses, two PCB congeners that are not significantly present in the sediment extract (PCB 103 and PCB 198 [14,15]), and 4,4'-DDT-d_8 were added to the sediment prior to extraction for use as internal standards for quantification purposes.

In addition to the analyses performed at NIST, SRM 1944 was used in an interlaboratory comparison exercise in 1995 as part of the NIST Intercomparison Exercise Program for Organic Contaminants in the Marine Environment [16]. Results from 19 laboratories that participated in this exercise were used as the eighth data set in the determination of the certified values for PCB congeners and chlorinated pesticides in SRM 1944. The laboratories participating in this exercise used the analytical procedures routinely used in their laboratories to measure PCB congeners and chlorinated pesticides.

Polychlorinated Dibenzo-*p*-dioxins and Dibenzofurans: Value assignment of the concentrations of the 17 2,3,7,8-substituted polychlorinated dibenzo-*p*-dioxin and dibenzofuran congeners and the total tetra- through hepta-substituted polychlorinated dibenzo-*p*-dioxins and dibenzofurans was accomplished by combining results from the analysis of SRM 1944 by 14 laboratories that participated in an interlaboratory comparison study (see Appendix A). Each laboratory analyzed three subsamples (typically 1 g) of SRM 1944 using their routine analytical procedures and gas chromatography with high resolution mass spectrometric detection (GC-HRMS).

The analytical procedures used by all of the laboratories included spiking with ^{13}C-labeled surrogates (internal standards); Soxhlet extraction with toluene; sample extract cleanup with acid/base silica, alumina, and carbon columns; and finally analysis of the cleaned up extract with GC-HRMS. Most of the laboratories used a 5 % phenyl-substituted methylpolysiloxane phase capillary column (DB-5), and about half of the laboratories confirmed 2,3,7,8-tetrachlorodibenzofuran using a 50 % (mole fraction) cyanopropylphenyl-substituted methylpolysiloxane capillary column (DB-225, J&W Scientific, Folsom, CA).

Analytical Approach for Inorganic Constituents: Value assignment for the concentrations of selected trace elements was accomplished by combining results of the analyses of SRM 1944 from NIST, NRCC, IAEA, and seven selected laboratories that participated in an interlaboratory comparison exercise coordinated by the NRCC [17] (see Appendix B). A similar approach was recently used to provide certified and reference concentration values for trace elements in two mussel tissue materials [18-20]. The analytical methods used for the determination of each element are summarized in Table 13. For the certified concentration values listed in Table 4, results were combined from: (1) analyses at NIST using isotope dilution inductively coupled plasma mass spectrometry (ID-ICPMS) or instrumental neutron activation analysis (INAA), (2) analyses at NRCC using ID-ICPMS, graphite furnace atomic absorption spectrometry (GFAAS), and/or inductively coupled plasma atomic emission spectroscopy (ICPAES), (3) analyses at IAEA using INAA, and (4) the mean of the results from seven selected laboratories that participated in the NRC interlaboratory comparison exercise. The reference concentration values in Table 7 were determined by combining results from (1) analyses performed at NIST using INAA; (2) analyses at NRCC using ID-ICPMS, GFAAS, ICPAES, and/or cold vapor atomic absorption spectroscopy (CVAAS); (3) analyses at IAEA using INAA; and (4) the mean of the results from five to seven laboratories that participated in the NRCC interlaboratory comparison exercise. The information concentration values in Table 12 were determined by INAA at NIST and IAEA.

NIST Analyses using ID-ICPMS: Lead, cadmium, and nickel were determined by ID-ICPMS [21]. Subsamples (0.4 g to 0.5 g) from six bottles of the SRM were spiked with ^{206}Pb, ^{111}Cd, and ^{62}Ni and wet ashed using a combination of nitric, hydrochloric, hydrofluoric, and perchloric acids. Lead and cadmium were determined in the same sample; nickel was determined in a second sample set. A small amount of crystalline material remained after the acid dissolution. Lithium metaborate fusion was performed on this residue to confirm that the residue contained insignificant amounts of the analytes. Cadmium and nickel were separated from the matrix material to eliminate the possibility of spectral interferences, and concentrations were determined from the measurement of the $^{112}Cd/^{111}Cd$ and $^{62}Ni/^{60}Ni$ ratios, respectively. The $^{208}Pb/^{206}Pb$ ratios were measured directly because interferences at these masses are negligible.

NIST Analyses using INAA: Analyses were performed in two steps [22]. Elements with short-lived irradiation products (Al, Ca, Cl, K, Mg, Mn, Na, Ti, and V) were determined by measuring duplicate 300 mg samples from each of 10 bottles of SRM 1944. The samples, standards, and controls were packaged in clean polyethylene bags and were individually irradiated for 15 s in the NIST Reactor Pneumatic Facility RT-4. Reactor power was 20 megawatts which corresponds to a neutron fluence rate of about 8×10^{13} $cm^{-2} \cdot s^{-1}$. After irradiation, the samples, controls, and standards were repackaged in clean polyethylene bags and counted (gamma-ray spectrometry) three times at different decay intervals. A sample to detector distance (counting geometry) of 20 cm was used. Elements with long-lived irradiation products (Ag, As, Br, Co, Cr, Cs, Fe, Rb, Sb, Sc, Se, Th, and Zn) were determined by measuring one 300 mg sample from each of nine bottles of SRM 1944. The samples, standards, controls, and blank polyethylene bags were irradiated together for a total of 1 h at a reactor power of 20 megawatts. Approximately four days after irradiation, the polyethylene bags were removed, and each sample, standard, control, and blank was counted at 20 cm from the detector. The samples were then recounted at 10 cm from another detector. After an additional decay time of about one month, the samples, standards, controls, and blanks were counted a third time (at 10 cm) from the second detector.

Particle-Size Information: Dry particle-size distribution measurements for SRM 1944 were obtained as part of a collaborative effort with Honeywell's Particle and Components Measurements Laboratory (Clearwater, FL). A Microtrac particle analyzer, which makes use of light-scattering techniques, was used to measure the particle-size distribution of SRM 1944. Briefly, a reference beam is used to penetrate a field of particles and the light that scatters in the forward direction from the field is measured and the particle size as a volume distribution is derived via a computer-assisted analysis. From these data, the total volume, average size, and a characteristic width of the particle-size distribution are calculated. The system has a working range from 0.7 μm to 700 μm.

Total Organic Carbon and Percent Extractable Mass: Four laboratories provided results for Total Organic Carbon (TOC) using similar procedures. Briefly, subsamples of approximately 200 mg were reacted with 6 N

hydrochloric acid and rinsed with deionized water prior to combustion in a gas fusion furnace. The carbon monoxide and carbon dioxide produced were measured and compared to a blank for calculation of the percent TOC. Each laboratory analyzed subsamples from six bottles of SRM 1944. For the determination of percent extractable mass, six subsamples of approximately 1 g to 2 g of SRM 1944 were extracted using Soxhlet extraction for 18 h with DCM. The extraction thimbles were allowed to air dry. After reaching constant mass, the difference in the mass before and after extraction was determined.

Table 1. Certified Concentrations for Selected PAHs in SRM 1944

PAHs	Mass Fractions in mg/kg (dry-mass basis)[a,b]		
Naphthalene[c,d,e,f,g]	1.65	±	0.31
Phenanthrene[c,d,e,f,g]	5.27	±	0.22
Anthracene[c,d,e,f,g]	1.77	±	0.33
Fluoranthene[c,d,e,f,g]	8.92	±	0.32
Pyrene[c,d,e,f,g]	9.70	±	0.42
Benzo[*c*]phenathrene[c,d,e,f,h]	0.76	±	0.10
Benz[*a*]anthracene[c,d,e,f,g,h]	4.72	±	0.11
Chrysene[h,k]	4.86	±	0.10[i]
Triphenylene[h,k]	1.04	±	0.27
Benzo[*b*]fluoranthene[g,h,j]	3.87	±	0.42
Benzo[*j*]fluoranthene[h,j]	2.09	±	0.44
Benzo[*k*]fluoranthene[c,d,e,f,g,h,j]	2.30	±	0.20
Benzo[*a*]fluoranthene[c,d,e,f,h,,j]	0.78	±	0.12
Benzo[*e*]pyrene[c,d,e,f,h,j]	3.28	±	0.11
Benzo[*a*]pyrene[c,d,e,f,g,h,j]	4.30	±	0.13
Perylene[c,d,e,f,g,h,j]	1.17	±	0.24
Benzo[*ghi*]perylene[c,d,e,f,j,k]	2.84	±	0.10
Indeno[1,2,3-*cd*]pyrene[c,d,e,f,j,k]	2.78	±	0.10
Dibenz[*a,j*]anthracene[c,d,e,f,j,k]	0.500	±	0.044
Dibenz[*a,c*]anthracene[j,k]	0.335	±	0.013
Dibenz[*a,h*]anthracene[j,k]	0.424	±	0.069
Pentaphene[c,d,e,f,j,k]	0.288	±	0.026
Benzo[*b*]chrysene[c,d,e,f,j,k,h]	0.63	±	0.10
Picene[c,d,e,f,j,k]	0.518	±	0.093

[a] Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

[b] The results are expressed as the certified value ± the expanded uncertainty. Each certified value is a mean of the means from two or more analytical methods, weighted as described in Paule and Mandel [23]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [24], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty within each analytical method as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.

[c] GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

[d] GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

[e] GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone.

[f] GC/MS (IV) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with 50 % hexane/50 % acetone.

[g] LC-FL of total PAH fraction after Soxhlet extraction with 50 % hexane/50 % acetone.

[h] GC/MS (Sm) using a smectic liquid crystalline phase after Soxhlet extraction with DCM.

[i] The uncertainty interval for chrysene was widened based on expert consideration of the analytical methods and analysis of the data for all PAHs, which suggests that the half-widths of the expanded uncertainties should not be less than 2 %.

[j] GC/MS (V) on 50 % phenyl-substituted methylpolysiloxane phase of extracts from GC/MS (III) and GC/MS (IV).

[k] LC-FL of isomeric PAH fractions after Soxhlet extraction with 50 % hexane/50 % acetone.

Table 2. Certified Concentrations for Selected PCB Congeners[a] in SRM 1944

PCB Congeners		Mass Fractions in µg/kg (dry-mass basis)[b,c]		
PCB 8	(2,4'-Dichlorobiphenyl)[d,e,f,g,h,i,j,k]	22.3	±	2.3
PCB 18	(2,2',5-Trichlorobiphenyl)[d,e,f,g,h,i,j,k]	51.0	±	2.6
PCB 28	(2,4,4'-Trichlorobiphenyl)[d,e,f,g,j,k]	80.8	±	2.7
PCB 31	(2,4',5-Trichlorobiphenyl)[d,e,f,g,j]	78.7	±	1.6[l]
PCB 44	(2,2',3,5'-Tetrachlorobiphenyl)[d,e,f,g,h,i,j,k]	60.2	±	2.0
PCB 49	(2,2',4,5'-Tetrachlorobiphenyl)[d,e,f,g,h,i,j,k]	53.0	±	1.7
PCB 52	(2,2',5,5'-Tetrachlorobiphenyl)[d,e,f,g,h,i,j,k]	79.4	±	2.0
PCB 66	(2,3',4,4'-Tetrachlorobiphenyl)[e,g,h,i,j]	71.9	±	4.3
PCB 87	(2,2',3,4,5'-Pentachlorobiphenyl)[d,e,f,g,h,i,j]	29.9	±	4.3
PCB 95	(2,2',3,5',6-Pentachlorobiphenyl)[e,g,h,i,j]	65.0	±	8.9
PCB 99	(2,2',4,4',5-Pentachlorobiphenyl)[d,e,f,g,h,i,j,k]	37.5	±	2.4
PCB 101	(2,2',4,5,5'-Pentachlorobiphenyl)[d,e,f,g,h,i,j,k]	73.4	±	2.5
90	(2,2',3,4',5-Pentachlorobiphenyl)			
PCB 105	(2,3,3',4,4'-Pentachlorobiphenyl)[e,f,g,h,i,j,k]	24.5	±	1.1
PCB 110	(2,3,3',4',6-Pentachlorobiphenyl)[g,h,i,j]	63.5	±	4.7
PCB 118	(2,3',4,4',5-Pentachlorobiphenyl)[d,e,f,g,h,i,j,k]	58.0	±	4.3
PCB 128	(2,2',3,3',4,4'-Hexachlorobiphenyl)[d,e,f,g,h,i,j,k]	8.47	±	0.28
PCB 138	(2,2',3,4,4',5'-Hexachlorobiphenyl)[d,e,f,g,h,i,j,k]	62.1	±	3.0
163	(2,3,3',4',5,6-Hexachlorobiphenyl)			
164	(2,3,3',4',5',6-Hexachlorobiphenyl)			
PCB 149	(2,2',3,4',5',6-Hexachlorobiphenyl)[d,e,f,g,h,i,j,k]	49.7	±	1.2
PCB 151	(2,2',3,5,5',6-Hexachlorobiphenyl)[d,e,f,g,h,i,j,k]	16.93	±	0.36
PCB 153	(2,2',4,4',5,5'-Hexachlorobiphenyl)[d,e,f,g,h,i,j,k]	74.0	±	2.9
PCB 156	(2,3,3',4,4',5-Hexachlorobiphenyl)[d,e,f,g,h,i,j]	6.52	±	0.66
PCB 170	(2,2',3,3',4,4',5-Heptachlorobiphenyl)[d,e,f,g,h,i,j,k]	22.6	±	1.4
190	(2,3,3',4,4',5,5'-Heptachlorobiphenyl)			
PCB 180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl)[d,e,f,g,h,i,j,k]	44.3	±	1.2
PCB 183	(2,2',3,4,4',5',6-Heptachlorobiphenyl)[d,e,f,g,h,i,j]	12.19	±	0.57
PCB 187	(2,2',3,4',5,5',6-Heptachlorobiphenyl)[d,e,f,g,h,i,j,k]	25.1	±	1.0
159	(2,3,3',4,5,5'-Hexachlorobiphenyl)			
182	(2,2',3',4,4',5,6'-Heptachlorobiphenyl)			
PCB 194	(2,2',3,3',4,4',5,5'-Octachlorobiphenyl)[d,e,f,g,h,i,j]	11.2	±	1.4
PCB 195	(2,2',3,3',4,4',5,6-Octachlorbiphenyl)[d,e,f,g,h,i,j,k]	3.75	±	0.39
PCB 206	(2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl)[d,e,f,g,h,i,j,k]	9.21	±	0.51
PCB 209	Decachlorobiphenyl [d,e,f,g,h,i,j,k]	6.81	±	0.33

[a] PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [14] and later revised by Schulte and Malisch [15] to conform with IUPAC rules; for the specific congeners mentioned in this SRM, the Ballschmiter-Zell numbers correspond to those of Schulte and Malisch. When two or more congeners are known to coelute under the conditions used, the congener listed first is the major component; additional congeners may be present as minor components.

[b] Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

[c] The results are expressed as the certified value ± the expanded uncertainty. Each certified value is a mean of the means from two or more analytical methods, weighted as described in Paule and Mandel [23]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [24], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty within each analytical method as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.

[d] GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

[e] GC-ECD (IB) on the 50 % C-18 dimethylpolysiloxane phase; same extracts analyzed as in GC-ECD (IA).

[f] GC-ECD (IIA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

[g] GC-ECD (IIB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

[h] GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone.

[i] GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with 50 % hexane/50 % acetone.

[j] GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

[k] Results from 19 laboratories participating in an interlaboratory comparison exercise.

[l] The uncertainty interval for PCB 31 was widened based on expert consideration of the analytical methods and analysis of the data for all PCB congeners, which suggests that the half-widths of the expanded uncertainties should not be less than 2 %.

Table 3. Certified Concentrations for Selected Chlorinated Pesticides in SRM 1944

Chlorinated Pesticides	Mass Fractions in µg/kg (dry-mass basis)[a,b]
Hexachlorobenzene[e,f,g,h,i,j]	6.03 ± 0.35
cis-Chlordane (α-Chlordane)[c,d,e,f,g,h,i,j]	16.51 ± 0.83
trans-Nonachlor[c,d,e,f,g,h,i,j]	8.20 ± 0.51
4,4'-DDT[c,d,e,f,g,h,i,j]	119 ± 11

[a] Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.
[b] The results are expressed as the certified value ± the expanded uncertainty. Each certified value is a mean of the means from two or more analytical methods, weighted as described in Paule and Mandel [23]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [24], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty within each analytical method as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.
[c] GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.
[d] GC-ECD (IB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IA).
[e] GC-ECD (IIA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.
[f] GC-ECD (IIB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).
[g] GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone.
[h] GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with 50 % hexane/50 % acetone.
[i] GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).
[j] Results from 19 laboratories participating in an interlaboratory comparison exercise.

Table 4. Certified Concentrations for Selected Inorganic Constituents in SRM 1944

Elements	Degrees of Freedom	Mass Fractions in percent (dry-mass basis)[a,b]
Aluminum[c,d,e]	4	5.33 ± 0.49
Iron[c,d,e]	6	3.53 ± 0.16
		Mass Fractions in mg/kg (dry-mass basis)[a,b]
Arsenic[c,d,e,f,g]	10	18.9 ± 2.8
Cadmium[c,f,h,i]	6	8.8 ± 1.4
Chromium[c,d,f,g,i]	9	266 ± 24
Lead[c,h,i]	5	330 ± 48
Manganese[c,d,e]	8	505 ± 25
Nickel[c,g,h,i]	6	76.1 ± 5.6
Zinc[c,d,e,g,i]	9	656 ± 75

[a] The results are expressed as the certified value ± the expanded uncertainty. The certified value is the mean of four results: (1) the mean of NIST INAA or ID-ICPMS analyses, (2) the mean of two methods performed at NRCC, and (3) the mean of results from seven selected laboratories participating in the NRCC intercomparison exercise, and (4) the mean results from INAA analyses at IAEA. The expanded uncertainty in the certified value is equal to $U = ku_c$, where u_c is the combined standard uncertainty and k is the coverage factor, both calculated according to the ISO Guide [24]. The value of u_c is intended to represent at the level of one standard deviation the combined effect of all the uncertainties in the certified value. Here u_c accounts for both possible method biases, within-method variation, and material inhomogeneity. The coverage factor, k, is the Student's t-value for a 95 % prediction interval with the corresponding degrees of freedom. Because of the material inhomogeneity, the variability among the measurements of multiple samples can be expected to be greater than that due to measurement variability alone.
[b] Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.
[c] Results from five to seven laboratories participating in the NRCC interlaboratory comparison exercise.
[d] Measured at NIST using INAA.
[e] Measured at NRCC using ICPAES.
[f] Measured at NRCC using GFAAS.
[g] Measured at IAEA using INAA.
[h] Measured at NIST using ID-ICPMS.
[i] Measured at NRCC using ID-ICPMS.

Table 5. Reference Concentrations for Selected PAHs in SRM 1944

NOTE: These concentrations are provided as reference values because either the results have not been confirmed by an independent analytical technique as required for certification or the agreement among results from multiple methods was insufficient for certification. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures.

PAHs	Mass Fractions in mg/kg (dry-mass basis)[a,b]		
1-Methylnaphthalene[c,d,e,f]	0.52	±	0.03
2-Methylnaphthalene[c,d,e,f]	0.95	±	0.05
Biphenyl[c,d,e,f]	0.32	±	0.07
Acenaphthene[c,d,e,f]	0.57	±	0.03
Fluorene[c,d,e,f]	0.85	±	0.03
Dibenzothiophene[d,e,f]	0.62	±	0.01[g]
1-Methylphenanthrene[c,d,e,f]	1.7	±	0.1
2-Methylphenanthrene[c,d,e,f]	1.90	±	0.06
3-Methylphenanthrene[c,d,e,f]	2.1	±	0.1
4-Methylphenanthrene and 9-Methylphenanthrene[c,d,e,f]	1.6	±	0.2
2-Methylanthracene[c,d,e,f]	0.58	±	0.04
3,5-Dimethylphenanthrene[c]	1.31	±	0.04
2,6-Dimethylphenanthrene[c]	0.79	±	0.02[g]
2,7-Dimethylphenanthrene[c]	0.67	±	0.02[g]
3,9-Dimethylphenanthrene[c]	2.42	±	0.05[g]
1,6-, 2,9-, and 2,5-Dimethylphenanthrene[c]	1.67	±	0.03[g]
1,7-Dimethylphenanthrene[c]	0.62	±	0.02[g]
1,9- and 4,9-Dimethylphenanthrene[c]	1.20	±	0.03[g]
1,8-Dimethylphenanthrene[c]	0.24	±	0.01[g]
1,2-Dimethylphenanthrene[c]	0.28	±	0.01[g]
8-Methylfluoranthene[c]	0.86	±	0.02[g]
7-Methylfluoranthene[c]	0.69	±	0.02
1-Methylfluoranthene[c]	0.66	±	0.02[g]
3-Methylfluoranthene[c]	2.46	±	0.07
2-Methylpyrene[c]	1.81	±	0.04[g]
4-Methylpyrene[c]	1.44	±	0.03[g]
1-Methylpyrene[c]	1.29	±	0.03
Anthanthrene[h]	0.9	±	0.1

[a] Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

[b] The reference value for each analyte is the equally-weighted mean of the means from two or more analytical methods or the mean from one analytical technique. The uncertainty in the reference value defines a range of values that is intended to function as an interval that contains the true value at a level of confidence of 95 %. This uncertainty includes sources of uncertainty within each analytical method, among methods, and from the drying study.

[c] GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

[d] GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

[e] GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone.

[f] GC/MS (IV) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with 50 % hexane/50 % acetone.

[g] The uncertainty interval for this compound was widened in accordance with expert consideration of the analytical procedures, along with the analysis of the data as a whole, which suggests that the half-widths of the expanded uncertainties should not be less than 2 %.

[h] LC-FL of isomeric PAH fractions after Soxhlet extraction with 50 % hexane/50 % acetone.

Table 6. Reference Concentrations for Selected Chlorinated Pesticides in SRM 1944

NOTE: These concentrations are provided as reference values because either the results have not been confirmed by an independent analytical technique as required for certification or the agreement among results from multiple methods was insufficient for certification. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures.

Chlorinated Pesticides	Mass Fractions in µg/kg (dry-mass basis)[a,b]
α-HCH[c,d,e,f]	2.0 ± 0.3
trans-Chlordane (γ-Chlordane)[c,d,e,f,g,h,i,j]	8 ± 2
cis-Nonachlor [d,e,f,i,j]	3.7 ± 0.7
2,4'-DDE [c,d,e,f,g,h,i,j]	19 ± 3
2,4'-DDD [e,f,g,h,i,j]	38 ± 8
4,4'-DDE [c,d,e,f,g,h,i,j]	86 ± 12
4,4'-DDD [c,d,e,f,g,h,i,j]	108 ± 16

[a] Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.
[b] The reference value for each analyte is the equally-weighted mean of the means from two or more analytical methods or the mean from one analytical technique. The uncertainty in the reference value defines a range of values that is intended to function as an interval that contains the true value at a level of confidence of 95 %. This uncertainty includes sources of uncertainty within each analytical method, among methods, and from the drying study.
[c] GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.
[d] GC-ECD (IB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IA).
[e] GC-ECD (IIA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.
[f] GC-ECD (IIB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).
[g] GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone.
[h] GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with 50 % hexane/50 % acetone.
[i] GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts anlayzed as in GC-ECD (IIA).
[j] Results from 19 laboratories participating in an interlaboratory comparison exercise.

Table 7. Reference Concentrations for Selected Inorganic Constituents in SRM 1944 as Determined by Multiple Laboratories

NOTE: These concentrations are provided as reference values because either the results have not been confirmed by an independent analytical technique as required for certification, the agreement among results from multiple methods was insufficient for certification, or insufficient analyses have been performed at NIST to confirm the results of the outside laboratories.

Elements	Degrees of Freedom	Mass Fraction in percent (dry-mass basis)[a,b]
Silicon[c,d]	81	31 ± 3
		Mass Fraction in mg/kg (dry-mass basis)[a,b]
Beryllium[c,h]	17	1.6 ± 0.3
Copper[c,d,f]	101	380 ± 40
Mercury[c,i]	18	3.4 ± 0.5
Selenium[c,e,f]	24	1.4 ± 0.2
Silver[c,d,e,g]	8	6.4 ± 1.7
Thallium[c,f]	12	0.59 ± 0.1
Tin[c,f]	22	42 ± 6

[a] The results are expressed as the reference value ± the expanded uncertainty. The reference value is the equally weighted mean of available results from: (1) NIST INAA analyses, (2) two methods performed at NRCC, (3) results from seven selected laboratories participating in the NRCC intercomparison exercise, and (4) results from INAA analyses at IAEA. The expanded uncertainty in the reference value is equal to $U = ku_c$ where u_c is the combined standard uncertainty and k is the coverage factor, both calculated according to the ISO Guide [24]. The value of u_c is intended to represent at the level of one standard deviation, the uncertainty in the value. Here u_c accounts for both possible method differences, within-method variation, and material inhomogeneity. The coverage factor, k, is the Student's t-value for a 95 % prediction interval with

the corresponding degrees of freedom. Because of material inhomogeneity, the variability among the measurements of multiple samples can be expected to be greater than that due to measurement variability alone.

[b] Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

[c] Results from five to seven laboratories participating in the NRCC interlaboratory comparison exercise.

[d] Measured at NRCC using GFAAS.

[e] Measured at NIST using INAA.

[f] Measured at NRCC using ID-ICPMS.

[g] Measured at IAEA using INAA.

[h] Measured at NRCC using ICPAES.

[i] Measured at NRCC using CVAAS.

Table 8. Reference Concentrations for Selected Inorganic Constituents in SRM 1944 as Determined by INAA

NOTE: These concentrations are provided as reference values because the results have not been confirmed by an independent analytical technique as required for certification; therefore, unrecognized bias may exist for some analytes in this matrix.

Element	Effective Degrees of Freedom	Mass Fraction in percent (dry-mass basis)[a,b]		
Calcium	21	1.0	±	0.1
Chlorine	21	1.4	±	0.2
Potassium	21	1.6	±	0.2
Sodium	25	1.9	±	0.1
		Mass Fraction in mg/kg (dry-mass basis)[a,b]		
Bromine	10	86	±	10
Cesium	11	3.0	±	0.3
Cobalt	10	14	±	2
Rubidium	14	75	±	2
Scandium	37	10.2	±	0.2
Titanium	21	4300	±	300
Vanadium	21	100	±	9

[a] The results are expressed as the reference value ± the expanded uncertainty. The reference value is based on the results from an INAA study. The associated uncertainty accounts for both random and systematic effects, but because only one method was used, unrecognized bias may exist for some analytes in this matrix. The expanded uncertainty in the reference value is equal to $U = ku_c$, where u_c is the combined standard uncertainty and k is the coverage factor, both calculated according to the ISO Guide [24]. The value of u_c is intended to represent at the level of one standard deviation, the uncertainty in the value. Here u_c accounts for within-method variation and material inhomogeneity. The coverage factor, k, is the Student's t-value for a 95 % prediction interval with the corresponding degrees of freedom. Because of material inhomogeneity, the variability among the measurements of multiple samples can be expected to be greater than that due to measurement variability alone.

[b] Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

Table 9. Reference Concentrations for Selected Dibenzo-*p*-dioxin and Dibenzofuran Congeners in SRM 1944

NOTE: These concentrations are provided as reference values because the results have not been confirmed by an independent analytical technique as required for certification. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures.

Dibenzo-*p*-dioxin and Dibenzofuran Congeners	Mass Fraction in µg/kg (dry-mass basis)[a,b]		
2,3,7,8-Tetrachlorodibenzo-*p*-dioxin	0.133	±	0.009
1,2,3,7,8-Pentachlorodibenzo-*p*-dioxin	0.019	±	0.002
1,2,3,4,7,8-Hexachlorodibenzo-*p*-dioxin	0.026	±	0.003
1,2,3,6,7,8-Hexachlorodibenzo-*p*-dioxin	0.056	±	0.006
1,2,3,7,8,9-Hexachlorodibenzo-*p*-dioxin	0.053	±	0.007
1,2,3,4,6,7,8-Heptachlorodibenzo-*p*-dioxin	0.80	±	0.07
Octachlorodibenzo-*p*-dioxin	5.8	±	0.7
2,3,7,8-Tetrachlorodibenzofuran[c]	0.039	±	0.015[d]
1,2,3,7,8-Pentachlorodibenzofuran	0.045	±	0.007
2,3,4,7,8-Pentachlorodibenzofuran	0.045	±	0.004
1,2,3,4,7,8-Hexachlorodibenzofuran	0.22	±	0.03
1,2,3,6,7,8-Hexachlorodibenzofuran	0.09	±	0.01
2,3,4,6,7,8-Hexachlorodibenzofuran	0.054	±	0.006[e]
1,2,3,7,8,9-Hexachlorodibenzofuran	0.019	±	0.018[f]
1,2,3,4,6,7,8-Heptachlorodibenzofuran	1.0	±	0.1
1,2,3,4,7,8,9-Heptachlorodibenzofuran	0.040	±	0.006[e]
Octachlorodibenzofuran	1.0	±	0.1
Total Toxic Equivalents (TEQ)[g]	0.25	±	0.01
Total Tetrachlorodibenzo-*p*-dioxins	0.25	±	0.05[e]
Total Pentachlorodibenzo-*p*-dioxins	0.19	±	0.06
Total Hexachlorodibenzo-*p*-dioxins	0.63	±	0.09
Total Heptachlorodibenzo-*p*-dioxins	1.8	±	0.2
Total Tetrachlorodibenzofurans	0.7	±	0.2
Total Pentachlorodibenzofurans	0.74	±	0.07
Total Hexachlorodibenzofurans	1.0	±	0.1
Total Heptachlorodibenzofurans	1.5	±	0.1
Total Dibenzo-*p*-dioxins[h]	8.7	±	0.9
Total Dibenzofurans[h]	5.0	±	0.5

[a] Each reference value is the mean of the results from up to 14 laboratories participating in an interlaboratory exercise. The expanded uncertainty in the reference value is equal to $U = ku_c$, where u_c is the combined standard uncertainty calculated according to the ISO Guide [24] and k is the coverage factor. The value of u_c is intended to represent at the level of one standard deviation, the combined effect of all the uncertainties in the reference value. Here u_c is the uncertainty in the mean arising from the variation among the laboratory results. The degrees of freedom is equal to the number of available results minus one (13 unless noted otherwise). The coverage factor, k, is the value from a student's t-distribution for a 95 % confidence interval.

[b] Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

[c] Confirmation results using a 50 % cyanopropyl phenyl polysiloxane or 90 % *bis*-cyanopropyl 10 % cyanopropylphenyl polysiloxane phase columns.

[d] Degrees of freedom = 7 for this compound.

[e] Degrees of freedom = 12 for this compound.

[f] Degrees of freedom = 9 for this compound.

[g] TEQ is the sum of the products of each of the 2,3,7,8-substituted congeners multiplied by their individual toxic equivalency factors (TEFs) recommended by the North Atlantic Treaty Organization (NATO) [25]. With regard to 2,3,7,8-tetrachlorodibenzofuran, the results of the confirmation column were used when available to calculate the TEQ.

[h] Total of tetra- through octachlorinated congeners.

Table 10. Reference Values for Particle-Size Characteristics for SRM 1944

NOTE: These results are provided as reference values because the results are method specific as defined by the procedures described in the Preparation and Analysis section. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures.

Particle Measurement	Value[a]
Mean diameter (volume distribution, MV, μm)[b]	151.2 ± 0.4
Mean diameter (area distribution, μm)[c]	120.4 ± 0.1
Mean diameter (number distribution, μm)[d]	75.7 ± 0.3
Surface Area (m^2/cm^3)[e]	0.050 ± 0.013

[a] The reference value is the mean value of measurements from the analysis of subsamples from four bottles. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [24], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. The expanded uncertainty defines a range of values for the reference value within which the true value is believed to lie, at a level of confidence of 95 %.

[b] The mean diameter of the volume distribution represents the center of gravity of the distribution and compensates for scattering efficiency and refractive index. This parameter is strongly influenced by coarse particles.

[c] The mean diameter of the area distribution, calculated from the volume distribution with less weighting by the presence of coarse particles than MV.

[d] The mean diameter of the number distribution, calculated using the volume distribution weighted to small particles.

[e] Calculated specific surface area assuming solid, spherical particles. This is a computation and should not be interchanged with an adsorption method of surface area determination as this value does not reflect porosity or topographical characteristics.

The following data show the percent of the volume that is smaller than the indicated size:

Percentile	Particle Diameter (μm)[a]
95	296 ± 5
90	247 ± 2
80	201 ± 1
70	174 ± 1
60	152 ± 1
50[b]	135 ± 1
40	120 ± 1
30	106 ± 1
20	91 ± 1
10	74 ± 1

[a] The reference value for particle diameter is the mean value of measurements from the analysis of subsamples from four bottles. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [24], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. The expanded uncertainty defines a range of values for the reference value within which the true value is believed to lie, at a level of confidence of 95 %.

[b] Median diameter (50 % of the volume is less than 135 μm).

Table 11. Reference Values for Total Organic Carbon and Percent Extractable Mass in SRM 1944

NOTE: These results are provided as reference values because the results are method specific as defined by the procedures described in the Preparation and Analysis section. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures.

Total Organic Carbon (TOC)	4.4 % ± 0.3 % mass fraction[a,b]
Extractable Mass[c]	1.15 % ± 0.04 % mass fraction[a,d]

[a] Concentration is reported on a dry-mass basis; material as received contains approximately 1.3% moisture.
[b] The reference value for total organic carbon is an equally weighted mean value from routine measurements made by three laboratories. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [24], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. The expanded uncertainty defines a range of values for the reference value within which the true value is believed to lie, at a level of confidence of 95 %.
[c] Extractable mass as determined from Soxhlet extraction using DCM.
[d] The reference value for extractable mass is the mean value of six measurements. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [24], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. The expanded uncertainty defines a range of values for the reference value within which the true value is believed to lie, at a level of confidence of 95 %.

Table 12. Information Values for Concentrations for Selected Inorganic Constituents in SRM 1944 as Determined by INAA

NOTE: These results are provided as information values only because insufficient information is available to assess adequately the uncertainty associated with the value or only a limited number of analyses were performed.

Elements	Mass Fractions in percent (dry-mass basis)[a]
Magnesium[b]	1.0
	Mass Fractions in mg/kg (dry-mass basis)[a]
Antimony[b,c]	5
Cerium[c]	65
Europium[c]	1.3
Gold[c]	0.10
Lanthanum[c]	39
Thorium[c]	13
Uranium[c]	3.1

[a] Concentration is reported on a dry-mass basis; material as received contains approximately 1.3 % moisture.
[b] Measured at NIST using INAA.
[c] Measured at IAEA using INAA.

Table 13. Analytical Methods Used for the Analysis of SRM 1944 for Inorganic Constituents

Elements	Analytical Methods
Aluminum	FAAS, ICPAES, INAA, XRF
Antimony	GFAAS, HGAAS, ICP-MS, ID-ICPMS, INAA
Arsenic	GFAAS, HGAAS, ICPMS, INAA, XRF
Beryllium	GFAAS, ICP-AES, ICPMS
Bromine	INAA
Cadmium	FAAS, GFAAS, ICPMS, ID-ICPMS
Calcium	INAA
Cerium	INAA
Cesium	INAA
Chlorine	INAA
Chromium	FAAS, GFAAS, ICPMS, ID-ICPMS, INAA, XRF
Cobalt	INAA
Copper	FAAS, GFAAS, ICPAES, ICPMS, ID-ICPMS, XRF
Europium	INAA
Gold	INAA
Iron	FAAS, ICPAES, ICPMS, ID-ICPMS, INAA, XRF
Lanthanum	INAA
Lead	FAAS, GFAAS, ICPMS, ID-ICPMS, XRF
Magnesium	INAA
Manganese	FAAS, ICPAES, ICPMS, INAA, XRF
Mercury	CVAAS, ICPMS
Nickel	GFAAS, ICPAES, ICPMS, ID-ICPMS, INAA, XRF
Potassium	INAA
Rubidium	INAA
Scandium	INAA
Selenium	GFAAS, HGAAS, ICPMS, INAA
Silicon	FAAS, ICPAES, XRF
Silver	FAAS, GFAAS, ICPMS, INAA
Sodium	INAA
Thallium	GFAAS, ICPAES, ICPMS, ID-ICPMS
Thorium	INAA
Tin	GFAAS, ICPMS, ID-ICPMS
Titanium	INAA
Uranium	INAA
Vanadium	INAA
Zinc	FAAS, ICPAES, ICPMS, ID-ICPMS, XRF, INAA

Methods	
CVAAS	Cold vapor atomic absorption spectrometry
FAAS	Flame atomic absorption spectrometry
GFAAS	Graphite furnace atomic absorption spectrometry
HGAAS	Hydride generation atomic absorption spectrometry
ICPAES	Inductively coupled plasma atomic emission spectrometry
ICPMS	Inductively coupled plasma mass spectrometry
ID-ICPMS	Isotope dilution inductively coupled plasma mass spectrometry
INAA	Instrumental neutron activation analysis
XRF	X-ray fluorescence spectrometry

APPENDIX A

The analysts and laboratories listed below participated in the interlaboratory comparison exercise for the determination of polychlorinated dibenzo-*p*-dioxins and dibenzofurans in SRM 1944.

W.J. Luksemburg, Alta Analytical Laboratory, Inc., El Dorado Hills, CA
L. Phillips, Axys Analytical Services Ltd., Sidney, British Columbia, Canada
M.J. Armbruster, Battelle Columbus Laboratories, Columbus, OH
G. Reuel, Canviro Analytical Laboratories Ltd., Waterloo, Ontario, Canada
C. Brochu, Environment Québec, Laval, Québec, Canada
G. Poole, Environment Canada Environmental Technology Centre, Ottawa, Ontario, Canada
B. Henkelmann, GSF National Research Center for Environment and Health, Neuherberg, Germany
R. Anderson, Institute of Environmental Chemistry, Umeå University, Umeå, Sweden
C. Lastoria, Maxxam Analytics, Inc., Mississauga, Ontario, Canada
E. Reiner, Ontario Ministry of Environment and Energy, Etobicoke, Ontario, Canada
J. Macaulay, Research and Productivity Council, Fredericton, New Brunswick, Canada
T.L. Wade, GERG, Texas A&M University, College Station, TX
C. Tashiro, Wellington Laboratories, Guelph, Ontario, Canada
T.O. Tiernan, Wright State University, Dayton, OH

APPENDIX B

The analysts and laboratories listed below participated in the interlaboratory comparison exercise for the determination of trace elements in SRM 1944.

A. Abbgy, Applied Marine Research Laboratory, Old Dominion University, Norfolk, VA
A. Scott, Australian Government Analytical Laboratories, Pymble, Australia
H. Mawhinney, Animal Research Institute, Queensland Department of Primary Industries, Queensland, Australia
E. Crecelius, Battelle Pacific Northwest, Sequim, WA
M. Stephenson, California Department of Fish and Game, Moss Landing, CA
B. Presley, Department of Oceanography, Texas A&M University, College Station, TX
K. Elrick, U.S. Geological Survey, Atlanta, GA

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Appendix C

Results of Analysis of Waters Used for Toxicity Testing

10207 General Drive
Orlando, Florida 32824
Phone: (407) 826-5314
Fax: (407) 850-6945
www.encolabs.com

Environmental Conservation Laboratories

Fax

To: Joy Ryan **From:** Ronnie Wambles

Fax: **Pages:** (Not including coversheet) 12

Phone: **Date:**

Re: **CC:**

☐ **Urgent** ☐ **For Review** ☐ **Please Comment** ☐ **Please Reply** ☐ **Please Recycle**

• **Comments:**

This is the raw data from the Furnace Lead run that day. It includes the calibration curve and all QC associated with that sample (ICV, blanks, LCS, MS/MSD, etc.)

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RW

PerkinElmer AAWinLab: 04/10/2003, 06:24:54 PM

Pb
4/10/03
AR

Method Name: lead 4/21/98
Method Description: lead thga 4/21/98
Element: Pb

Date: 04/10/2003
Technique:Furnace
Calibration Type:
Pb, Zero Intercept: Nonlinear
Wavelength: 283.3 nm
Slit Width: 0.7
Lamp Current: 8 mA
Sample Info Name: PB041003.SIF Results Data Set Name: pb041003a

Element: Pb Seq. No.: 1 AS Loc.: 87 Date: 04/10/2003
Sample ID: Calib Blank
µL dispensed: 10 from 87, 5 from 86, 20 from 87

Repl #	SampleConc	StndConc	BlnkCorr Signal	Peak Area	Peak Height	Time
1			-0.0005	-0.0005	0.0107	06:27:12
2			-0.0017	-0.0017	0.0098	06:29:56
Mean:			-0.0011			
SD :			0.0008			
%RSD:			77.1			

Auto-zero performed.

Element: Pb Seq. No.: 2 AS Loc.: 85 Date: 04/10/2003
Sample ID: .005 ppm
µL dispensed: 20 from 87, 5 from 86, 10 from 85

Repl #	SampleConc	StndConc	BlnkCorr Signal	Peak Area	Peak Height	Time
1			0.0124	0.0114	0.0711	06:32:41
2			0.0147	0.0136	0.0699	06:35:26
Mean:			0.0135			
SD :			0.0016			
%RSD:			11.6			

[Pb] Standard number 1 applied. [0.0050]
Correlation Coefficient: 1.00000 Slope: 2.70901

Element: Pb Seq. No.: 3 AS Loc.: 85 Date: 04/10/2003
Sample ID: .010 ppm
µL dispensed: 10 from 87, 5 from 86, 20 from 85

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Peak Area	Peak Height	Time
1			0.0267	0.0256	0.1016	06:38:11
2			0.0244	0.0233	0.0922	06:40:55
Mean:			0.0256			
SD :			0.0017			
%RSD:			6.5			

[Pb] Standard number 2 applied. [0.0100]
Correlation Coefficient: 1.00000 Slope: 2.88250

Element: Pb Seq. No.: 4 AS Loc.: 88 Date: 04/10/2003
Sample ID: .025 ppm
µL dispensed: 20 from 87, 5 from 86, 10 from 88

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Peak Area	Peak Height	Time

PerkinElmer AAWinLab: 04/10/2003, 06:43:40 PM

1	0.0593	0.0582	0.2225	06:43:40
2	0.0616	0.0605	0.2398	06:46:25
Mean:	0.0605			
SD :	0.0016			
%RSD:	2.7			

[Pb] Standard number 3 applied. [0.0250]
Correlation Coefficient: 1.00000 Slope: 3.49766

==========

Element: Pb Seq. No.: 5 AS Loc.: 88 Date: 04/10/2003
Sample ID: .050 ppm
µL dispensed: 10 from 87, 5 from 86, 20 from 88

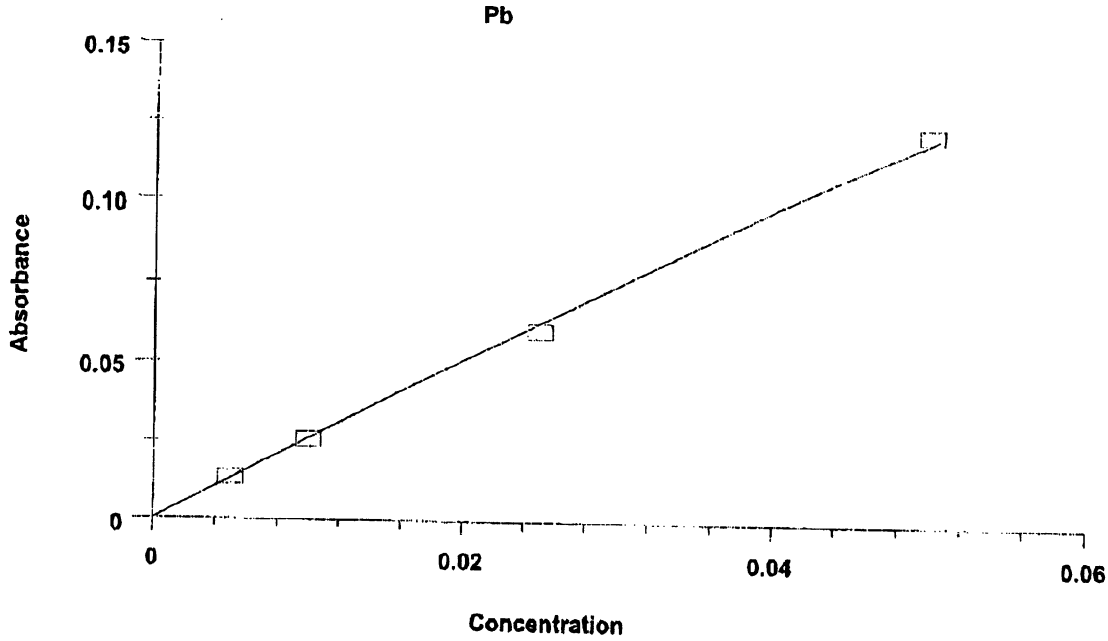
Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Peak Area	Peak Height	Time
1			0.1238	0.1227	0.3494	06:49:10
2			0.1211	0.1200	0.3270	06:51:55
Mean:			0.1224			
SD :			0.0019			
%RSD:			1.6			

S-shaped calibration curve detected. Two-coefficient equation used.
[Pb] Standard number 4 applied. [0.0500]
Correlation Coefficient: 0.99905 Slope: 2.64939

Calibration data for Pb

Standard ID	Mean Signal (Pk Area)	Entered Concentration (mg/L)	Calculated Concentration (mg/L)	Standard Deviation	%RSD
Calib Blank	-0.0011	----	----	----	----
.005 ppm	0.0135	0.0050	0.0052	0.00156	11.6
.010 ppm	0.0256	0.0100	0.0098	0.00167	6.5
.025 ppm	0.0605	0.0250	0.0239	0.00163	2.7
.050 ppm	0.1224	0.0500	0.0511	0.00193	1.6

Correlation Coefficient: 0.99905 Slope: 2.64939 ----



==========

PerkinElmer AAWinLab: 04/10/2003, 06:53:12 PM

Element: Pb Seq. No.: 6 AS Loc.: 1 Date: 04/10/2003
Sample ID: icv
µL dispensed: 10 from 87, 5 from 86, 20 from 1

8/8/03

97% Rec

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Peak Area	Peak Height	Time
1	0.0249	0.0249	0.0626	0.0616	0.2298	06:54:39
2	0.0238	0.0238	0.0602	0.0591	0.2266	06:57:23
Mean:	0.0243	0.0243	0.0614			
SD :	0.00073	0.00073	0.0017			
%RSD:	3.0	3.0	2.8			

QC value within specified limits. OK

Element: Pb Seq. No.: 7 AS Loc.: 87 Date: 04/10/2003
Sample ID: icb
µL dispensed: 10 from 87, 5 from 86, 20 from 87

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Peak Area	Peak Height	Time
1	-0.0012	-0.0012	-0.0033	-0.0044	0.0083	07:00:08
2	0.0003	0.0003	0.0008	-0.0003	0.0101	07:02:52
Mean:	-0.0005	-0.0005	-0.0012			
SD :	0.00110	0.00110	0.0029			
%RSD:	236.2	236.2	235.8			

QC value within specified limits. OK

Element: Pb Seq. No.: 8 AS Loc.: 2 Date: 04/10/2003
Sample ID: cvs
µL dispensed: 10 from 87, 5 from 86, 20 from 2

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Peak Area	Peak Height	Time
1	0.0240	0.0240	0.0606	0.0596	0.2226	07:05:33
2	0.0226	0.0226	0.0572	0.0561	0.2229	07:08:16
Mean:	0.0233	0.0233	0.0589			
SD :	0.00101	0.00101	0.0024			
%RSD:	4.3	4.3	4.1			

OK

Element: Pb Seq. No.: 9 AS Loc.: 3 Date: 04/10/2003
Sample ID: mp0304092-1 blk
µL dispensed: 10 from 87, 5 from 86, 20 from 3

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Peak Area	Peak Height	Time
1	0.0000	0.0000	0.0000	-0.0011	0.0083	07:11:02
2	-0.0003	-0.0003	-0.0008	-0.0018	0.0129	07:13:45
Mean:	-0.0002	-0.0002	-0.0004			
SD :	0.00019	0.00019	0.0005			
%RSD:	125.4	125.4	125.4			

OK

Element: Pb Seq. No.: 10 AS Loc.: 4 Date: 04/10/2003
Sample ID: mp0304092-2 lcs
µL dispensed: 10 from 87, 5 from 86, 20 from 4

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Peak Area	Peak Height	Time
1	0.0313	0.0313	0.0779	0.0768	0.2081	07:16:28
2	0.0326	0.0326	0.0810	0.0799	0.1787	07:19:12
Mean:	0.0320	0.0320	0.0794			
SD :	0.00092	0.00092	0.0021			
%RSD:	2.9	2.9	2.7			

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PerkinElmer AAWinLab: 04/10/2003, 07:20:29 PM

Element: Pb Seq. No.: 11 AS Loc.: 5 Date: 04/10/2003
Sample ID: .orl26361-1
µL dispensed: 10 from 87, 5 from 86, 20 from 5

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Peak Area	Peak Height	Time
1	0.0006	0.0006	0.0015	0.0004	0.0113	07:21:56
2	-0.0003	-0.0003	-0.0009	-0.0019	0.0135	07:24:40
Mean:	0.0001	0.0001	0.0003			
SD :	0.00062	0.00062	0.0016			
%RSD:	541.8	541.8	543.6			

Element: Pb Seq. No.: 12 AS Loc.: 5 Date: 04/10/2003
Sample ID: .orl26361-1
µL dispensed: 5 from 86, 20 from 5, 10 from 88

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Peak Area	Peak Height	Time
1	0.0278	0.0278	0.0696	0.0685	0.1628	07:27:28
2	0.0252	0.0252	0.0634	0.0623	0.1468	07:30:19
Mean:	0.0265	0.0265	0.0665			
SD :	0.00184	0.00184	0.0044			
%RSD:	7.0	7.0	6.6			

Recovery for Pb = 105.9 % within 85 % to 115 %

PDS OK spiked @ 0.025 µg/L ~ 8/8/03

Element: Pb Seq. No.: 13 AS Loc.: 6 Date: 04/10/2003
Sample ID: mp0304092-3 ms
µL dispensed: 10 from 87, 5 from 86, 20 from 6

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Peak Area	Peak Height	Time
1	0.0324	0.0324	0.0804	0.0793	0.2614	07:33:03
2	0.0320	0.0320	0.0796	0.0785	0.2529	07:35:45
Mean:	0.0322	0.0322	0.0800			
SD :	0.00026	0.00026	0.0006			
%RSD:	0.8	0.8	0.7			

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The measured recovery for locations 5 and 6 for [Pb] is 106.9 percent.

Element: Pb Seq. No.: 14 AS Loc.: 7 Date: 04/10/2003
Sample ID: mp0304092-4 msd
µL dispensed: 10 from 87, 5 from 86, 20 from 7

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Peak Area	Peak Height	Time
1	0.0321	0.0321	0.0799	0.0788	0.3407	07:38:29
2	0.0332	0.0332	0.0822	0.0811	0.2824	07:41:13
Mean:	0.0327	0.0327	0.0811			
SD :	0.00072	0.00072	0.0017			
%RSD:	2.2	2.2	2.1			

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The difference between locations 6 and 7 for [Pb] is 1.4 percent.

Element: Pb Seq. No.: 15 AS Loc.: 8 Date: 04/10/2003
Sample ID: .orl26378
µL dispensed: 10 from 87, 5 from 86, 20 from 8

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Peak Area	Peak Height	Time
1	0.0370	0.0370	0.0910	0.0899	0.4856	07:43:55
2	0.0166	0.0166	0.0424	0.0413	0.2761	07:46:38
Mean:	0.0268	0.0268	0.0667			
SD :	0.01443	0.01443	0.0344			

4/10/03

PerkinElmer AAWinLab: 04/10/2003, 07:46:38 PM

%RSD: 53.9 53.9 51.5
Precision (%RSD or SD) greater than specified limit. Repeating measurement.

Element: Pb Seq. No.: 16 AS Loc.: 8 Date: 04/10/2003
Sample ID: .or126378
µL dispensed: 10 from 87, 5 from 86, 20 from 8

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Peak Area	Peak Height	Time
1	0.0292	0.0292	0.0729	0.0719	0.5391	07:49:22
2	0.0341	0.0341	0.0844	0.0833	0.9939	07:52:05
Mean:	0.0316	0.0316	0.0786			
SD :	0.00346	0.00346	0.0081			
%RSD:	10.9	10.9	10.3			

Element: Pb Seq. No.: 17 AS Loc.: 2 Date: 04/10/2003
Sample ID: ccv
µL dispensed: 10 from 87, 5 from 86, 20 from 2

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Peak Area	Peak Height	Time
1	0.0230	0.0230	0.0582	0.0571	0.1799	07:54:47
2	0.0250	0.0250	0.0631	0.0620	0.2146	07:57:31
Mean:	0.0240	0.0240	0.0606			
SD :	0.00144	0.00144	0.0035			
%RSD:	6.0	6.0	5.7			

QC value within specified limits. OK

96% Rec 8/18/03

Element: Pb Seq. No.: 18 AS Loc.: 87 Date: 04/10/2003
Sample ID: ccb
µL dispensed: 10 from 87, 5 from 86, 20 from 87

Repl #	SampleConc mg/L	StndConc mg/L	BlnkCorr Signal	Peak Area	Peak Height	Time
1	0.0000	0.0000	-0.0001	-0.0011	0.0108	08:00:15
2	0.0004	0.0004	0.0010	0.0000	0.0088	08:03:00
Mean:	0.0002	0.0002	0.0005			
SD :	0.00029	0.00029	0.0008			
%RSD:	158.2	158.2	158.2			

QC value within specified limits. OK

PerkinElmer AAWinLab: 04/10/2003, 06:02:13 PM

Identification

Method Name: lead 4/21/98
Technique: Furnace
Method Desc: lead thga 4/21/98

Element: Pb
Instrument Model: AA600

Remarks:
Instrument serail number 6207

Instrument

Wavelength (nm): 283.3
Slit Width: 0.7

Read Time (sec): 5.0
Read Delay (sec): 0.0
BOC Time (sec): 2

Signal Type: AA-BG
Signal Measurement: Peak Area

Modified Spectrometer Settings: No

Lamp Current: Value in Align lamps

Calibration

Equations, Units, and Replicates:

Element	Calibration Equation	Calibration Units	Sample Units	Max Decimal Places	Max Significant Figures
Pb	Non-linear	mg/L	mg/L	4	5

Replicates: Fixed

Sample
Sample Volume (µL): 20 **Diluent Volume (µL):** 10 **Diluent Location:** 87

Matrix Modifiers:

	Modifier 1	Modifier 2
Volume (µL):	5	0
Location:	86	0
Add to blank and standards:	Yes	No
Add to reagent blank and samples:	Yes	No

	ID	Conc. (mg/L)	A/S Loc	Stock (µL)	Diluent (µL)
Calib. Blank	Calib Blank		87	20	10
Reslope Std.	Reslope	0.025	88	10	20
Calib.Std. 1	.005 ppm	0.005	85	10	20
Calib.Std. 2	.010 ppm	0.010	85	20	10
Calib.Std. 3	.025 ppm	0.025	88	10	20
Calib.Std. 4	.050 ppm	0.050	88	20	10
Calib.Std. 5				0	0
Calib.Std. 6				0	0
Calib.Std. 7				0	0
Calib.Std. 8				0	0
Calib.Std. 9				0	0
Calib.Std. 10				0	0
Calib.Std. 11				0	0
Calib.Std. 12				0	0
Calib.Std. 13				0	0
Calib.Std. 14				0	0
Calib.Std. 15				0	0

PerkinElmer AAWinLab: 04/10/2003, 06:02:13 PM

Furnace Conditions

Step #	Temp (°C)	Ramp Time	Hold Time	Internal Flow	Gas Type	Read Step
1	120	1	30	250	Normal	
2	150	5	20	250	Normal	
3	600	5	20	250	Normal	
4	1600	0	5	0	Normal	X
5	2400	1	3	250	Normal	

Extraction System: No **Injection Temp. (°C):** 80

Furnace Clean-out ? No

Sequence

Step	Action and Parameters
A	Pipet diluent + modifierl + sample/std + spike
B	Run furnace steps 1 to end

Pipet Speed (%): 85

Checks

Check correlation coefficient? .....Yes

Recalibrate instrument periodically? .....No

Analyze standards at end of run? .....No

Check Precision? .....Yes

Reanalyze samples beyond calibration range? ....Yes
Overcal Limit: 100 percent of Calibration Range.
Check Range On: Samples Recovery Samples
Dilute and reanalyze after: 1 Replicate
Alternate Sample Volumes (µL): 10 5 0 0 0

Perform matrix check calculations? .....Yes

Perform recovery measurements? .....Yes

Quality Control

QC Samples: Descriptions and Concentrations

	QC 1	QC 2	QC 3
QC Sample ID	icv	icb	ccv
Autosampler Loc	1	87	2
Count as a Sample	No	No	No
Use Reagent Blank	No	No	No
Concentration	0.025	0.000	0.025
Lower Conc Limit	0.022	-0.002	0.022
Upper Conc Limit	0.028	0.002	0.028

	QC 4	QC 5	QC 6
QC Sample ID	ccb	blk-1	lcs-1
Autosampler Loc	87	3	4
Count as a Sample	No	No	No
Use Reagent Blank	No	No	No
Concentration	0.000	0.000	0.030
Lower Conc Limit	-0.002	-0.002	0.025
Upper Conc Limit	0.002	0.002	0.035

Concentration Units: Sample (mg/L)

PerkinElmer AAWinLab: 04/10/2003, 06:02:14 PM

Schedule for Analyzing QC Samples

	QC 1	QC 2	QC 3
QC Sample ID	icv	icb	ccv
After Initial Calib.	Yes	Yes	No
After Recalibration	Yes	Yes	No
Periodically	No	No	Yes
Frequency	1	1	10
At End	No	No	Yes

	QC 4	QC 5	QC 6
QC Sample ID	ccb	blk-1	lcs-1
After Initial Calib.	No	No	No
After Recalibration	No	No	No
Periodically	Yes	No	No
Frequency	10	1	1
At End	Yes	No	No

Periodic Timing of Analyses

Count: Samples
Frequency: Variable

Failure Actions for After-Calibration QC's

	QC 1	QC 2	QC 3
QC Sample ID	icv	icb	ccv
Times to Retry QC	2	2	None
When All Tries Fail	Recalibrate	Recalibrate	Continue
Optional Message			

	QC 4	QC 5	QC 6
QC Sample ID	ccb	blk-1	lcs-1
Times to Retry QC	None	None	None
When All Tries Fail	Continue	Continue	Continue
Optional Message			

Failure Actions for Periodic QC's

	QC 1	QC 2	QC 3
QC Sample ID	icv	icb	ccv
Times to Retry QC	None	None	1
When All Tries Fail	Continue	Continue	Recal & Rerun
Optional Message			

	QC 4	QC 5	QC 6
QC Sample ID	ccb	blk-1	lcs-1
Times to Retry QC	1	None	None
When All Tries Fail	Recal & Rerun	Continue	Continue
Optional Message			

Failure Actions for At-End QC's

	QC 1	QC 2	QC 3
QC Sample ID	icv	icb	ccv
Times to Retry QC	None	None	1
When All Tries Fail	Continue	Continue	Recal & Rerun
Optional Message			

	QC 4	QC 5	QC 6
QC Sample ID	ccb	blk-1	lcs-1
Times to Retry QC	1	None	None
When All Tries Fail	Recal & Rerun	Continue	Continue
Optional Message			

Maximum Retries After QC Failure

Maximum allowed reanalyses: 1 Action when maximum exceeded: Continue

PerkinElmer AAWinLab: 04/10/2003, 06:02:14 PM

Options

Results Display - Modified Settings: No

Printed Log - Modified Settings: Yes

Peaks to Save: First Replicate for Each Sample

Environmental Conservation Laboratories, Inc.

WORK GROUP REPORT (wk02)

Apr 10 2003, 05:56 pm

Work Group: PB041003A for Department: 5 Metals Analysis

Created: 10-APR-03 Due: Operator:

Sample (Clientid)	Account Name	C	Product	Matrix	Stat	DA	Workdate	PR	Loc
MP0304092-1	BLK	S	PB239.2	GroundH2O	WIP	U	09-APR-03		
MP0304092-2	LCS	S	PBFUQC	GroundH2O	WIP	U	09-APR-03		
MP0304092-3	MS (ORL26361-1)	S	PBFUQC	GroundH2O	WIP	U	09-APR-03		
MP0304092-4	MSD (ORL26361-1)	S	PBFUQC	GroundH2O	WIP	U	09-APR-03		
ORL26378-1 (NAT, P)	MacTech Consulting &	S	PB239.2	GroundH2O	WIP	U	14-APR-03	S17	

Analysis Comments

ORL26378 -1 is filtered saltwater

PerkinElmer AAWinLab: 04/10/2003, 06:02:09 PM

Sample Information File C:\AAUSER\SAMPINFO\PB041003.SIF

Description : pb041003a
Batch ID :
Volume Units :
Weight Units :
Analyst : assefa
Sample Volume : 0.00

Pb
4/10/03
AA

AS Loc	Sample ID	Sample Weight	Sample Units	User Dilution	Remarks
1	icv				
2	cvs				
3	mp0304092-1 blk				
4	mp0304092-2 lcs				
5	.orl26361-1				
6	mp0304092-3 ms				
7	mp0304092-4 msd				
8	.orl26378				

PB041003A
R31453

CALB = RUP 22C //
ICV = RUP 23A //
CCV = RUP 22C //
PDS = RUP 22C //

WORKGROUP REPORT By Project
Workgroup: PB041003A (Run_ID: R31453)
Loginnum: ORL26378 Acctname: (EN012) MacTec Consulting & Eng.
Project: APRIL03

SAMPLE:	ORL26378-1	MP0304092-1
CLIENT ID:	NAT. FILT SALTWATER	Blank Sample
SAMPLE TAG:		
ENTER DATE:	10-APR-03	10-APR-03
PREP DATE:		
DATE ANALYZED:	04/10/03 00:00	04/10/03 00:00
COMMENTS:		BLK LINKS: 1

EPA METHOD: 239.2

	DIL	NUMVALUE	Q	RDL	C	DIL	NUMVALUE	Q	RDL	C	
Lead		0.032		0.0050			0.00030	U	0.0050		mg/L

QUALITY CONTROL DATA

Parameter	% RECOVERY MS/MSD/LCS	MS/MSD LIMITS	LCS LIMITS	% RPD MS/MSD	RPD LIMITS
Total Metals					
Lead, 239.2	107/109/107	66-140	-	2	17

Appendix D

Field Documentation

PONCE HARBOR SAMPLING: HARBOR STATIONS WATER COLLECTION FIELD DATA SHEET
(SAMPLING TO BE DONE AT FINAL VIBRACORE STATION PRIOR TO SEDIMENT COLLECTION)

Date: 03/07/2003

Station: E-PH03-1W

Coordinates: 17.96990° N
66.61948° W

Note: All coordinates are in Datum NAD 83

Differential Correction Method: Beacon WAAS Corrected

PPB Lab Needs:

Glass gallon jar	Time (AST)
1	0900
2	
3	
4	
5	
6	
7	
8	
9	
10	
11	
12	
13	
14	
15	
16	
17	
18	
19	
20	
21	
22	
23	
24	

Battelle Lab Needs:

Glass gallon jar	Time (AST)
1	0900
2	
3	
4	
5	
6	
7	
8	
9	
10	
11	
12	

	Time (AST)
1 Liter Teflon Bottle Preserve with nitric acid	0855

Form Completed by: [signature]

Date/Time: 03/07/2003 0930

This form should be signed and date and time indicated.

PONCE HARBOR SAMPLING: HARBOR STATIONS WATER QUALITY FIELD DATA SHEET (SAMPLING TO BE DONE AT MID-DEPTH)

Station	Date	Time (AST)	Latitude (NAD 83)	Longitude (NAD 83)	Total Depth (ft)	Actual Measurement Depth (ft)	Sea State	Precipitation	Wind Speed	Wind direction	Tidal Stage	Tidal Flow Direction	Water Temp (°C)	pH (su)	Dissolved Oxygen (mg/L)	Turbidity (NTUs)	Conductivity (mMhos/cm)	Salinity (ppt)
E-PH03-1	3-5-03	1130	17.9713499	66.6161149	38.1	19.0	C	NP	>15	E	E	/	22.96	7.84	7.31	7.1	59.6	35.4
E-PH03-2	3-6-03	1200	17.9724440	66.6185062	42.5	21.2	C	NP	>15	E	E	/	26.77	8.02	6.59	8.9	54.6	36.4
E-PH03-3	3-4-03	1425	17.9713366	66.6231983	30.5	15.1	S	NP	>15	SE	SL	/	27.12	7.98	7.56	2.5	53.9	35.5
E-PH03-4	3-4-03	1215	17.9702283	66.6208283	40.3	20.2	C	NP	>15	SE	SH	/	26.91	8.62	7.03	2.4	54.3	35.3
E-PH03-5	3-6-03	1042	17.9691600	66.6267300	34.4	17.2	S	NP	>15	SE	R	/	26.93	8.08	6.40	6.1	54.7	36.0
E-PH03-6	3-6-03	0815	17.9667966	66.6231800	39.5	19.5	C	NP	5-10	SE	R	/	22.40	8.25	7.33	7.7	60.7	40.1 *
E-PH03-7	3-6-03	0942	17.9668416	66.6278449	39.1	19.5	C	NP	5-10	SE	R	/	26.90	8.13	6.52	6.0	54.6	36.1
E-PH03-8	3-5-03	1001	17.9645850	66.6255383	40.5	20.2	S	NP	10-15	E	R	/	26.79	7.96	6.44	1.8	54.9	36.2
E-PH03-9	3-5-03	0830	17.9623099	66.6256166	48.5	24.3	S	NP	5-10	E	R	/	26.51	8.06	6.18	2.0	54.9	36.6

Various codes:

Sea State: C - Calm S - Slight R - Rough VR - Very Rough

Precipitation: NP - No Precipitation D - Drizzle SR - Slight Rain HR - Heavy Rain

Wind Speed: 0-5 5-10 10-15 >15 knots

Wind Direction: N NE E SE S SW W NW

Tidal Stage: E - Ebb SL - Slack Low R - Rising SH - Slack High

Differential Correction Method: Beacon

Comments: * Verified validity of high salinity value w/ refractometer model # RHS-10ATC

H20 Serial #: 34583

Surveyor 3/Scout 2 Serial #: 34684

Field Calibrated by: FJ

Date/Time Calibrated: Daily Pre & Post See Cal Sheets

Form Completed by: [signature]

Date/Time: 03/06/03 1300

This form should be signed with date and time indicated.

PONCE HARBOR
HYDROLAB CALIBRATION FORM

Client: PPB/AFI
Job No.: 1824-1
H20 Serial No.: 34583
Scout/Surveyor 3 Serial No.: 34684

Date: 03/04/2003
Site: Harbor
Cable Length: 30m

TIME (AST): 0902
Calibrated by: FJ

TIME (AST): 1655
Calibrated by: FJ

Temperature (°C)
Calibration Thermometer(°C) 30.0
Hydrolab Temperature(°C) 29.82

End-of-Day
Calibration Thermometer(°C) 30.5
Hydrolab Temperature(°C) 29.88

Dissolved Oxygen (DO)
Salinity Compensation: Yes No
Local Barometric Pressure (in Hg): 30.05
Multiplied by 25.4(mm Hg) = 763.27
% Saturation: 100.1
Dissolved Oxygen (mg/L): 7.80

End-of-Day
Salinity Compensation: Yes No
Local Barometric Pressure (in Hg): 30.00
Multiplied by 25.4(mm Hg) = 792.0
% Saturation: 100.3
Dissolved Oxygen (mg/L): 7.18

Depth (ft)
Hydrolab depth reading at 0': 0.0
Actual Depth of Sensor (ft): 10.00
Hydrolab depth reading (ft): 10.00

End-of-Day
Hydrolab depth reading at 0': 0
Actual Depth of Sensor (ft): 10.00
Hydrolab depth reading (ft): 10.1

pH (su)
Hydrolab Value at pH 7.00 Solution: 7.00
Hydrolab Value at pH 10.0 Solution: 10.00

End-of-Day
Hydrolab Value at pH 7.00 Solution: 6.99
Hydrolab Value at pH 10.0 Solution: 9.89

Conductivity (mmhos/cm)
Range: Auto ✓ Hi ____ Med ____ Low ____
Compensated Setting: (Yes) No

End-of-Day
Range: Auto ✓ Hi ____ Med ____ Low ____
Compensated Setting: (Yes) No

Salinity (ppt)
Actual salinity of solution (ppt): 34.9
Hydrolab salinity reading (ppt): 34.9

End-of-Day
Actual salinity of solution (ppt): 34.9
Hydrolab salinity reading (ppt): 34.4

Turbidity
Stabcal Sol'n #1 4.1 Hydrolab Reading 0.0
Stabcal Sol'n #2 10.0 Hydrolab Reading 10.0
Stabcal Sol'n #3 20.0 Hydrolab Reading 20.0

End-of-Day
Hydrolab Reading 4.1
Hydrolab Reading 10.8
Hydrolab Reading 22.0

PONCE HARBOR
HYDROLAB CALIBRATION FORM

Client: PPB/AFI
Job No.: 1824-1
H20 Serial No.: 34583
Scout/Surveyor 3 Serial No.: 34684

Date: 03/05/2003
Site: Harbor
Cable Length: 30m

TIME (AST): 0700
Calibrated by: FJ

TIME (AST): 1601
Calibrated by: FJ

Temperature (°C)
Calibration Thermometer(°C) 30.2
Hydrolab Temperature(°C) 29.86

End-of-Day
Calibration Thermometer(°C) ~~30.5~~ FJ 30.0
Hydrolab Temperature(°C) ~~29.88~~ FJ 29.46

Dissolved Oxygen (DO)
Salinity Compensation: (Yes) No
Local Barometric Pressure (in Hg): 30.00
Multiplied by 25.4(mm Hg) = 792
% Saturation: 100.0
Dissolved Oxygen (mg/L): 7.52

End-of-Day
Salinity Compensation: (Yes) No
Local Barometric Pressure (in Hg): na
Multiplied by 25.4(mm Hg) = 760
% Saturation: 94.5
Dissolved Oxygen (mg/L): 6.98

Depth (ft)
Hydrolab depth reading at 0': 0
Actual Depth of Sensor (ft): 10.00
Hydrolab depth reading (ft): 10.0

End-of-Day
Hydrolab depth reading at 0': 0.2
Actual Depth of Sensor (ft): 10.0
Hydrolab depth reading (ft): 10.3

pH (su)
Hydrolab Value at pH 7.00 Solution: 7.00
Hydrolab Value at pH 10.0 Solution: 10.00

End-of-Day
Hydrolab Value at pH 7.00 Solution: 6.76
Hydrolab Value at pH 10.0 Solution: 9.69

Conductivity (mmhos/cm)
Range: Auto ✓ Hi ___ Med ___ Low ___
Compensated Setting: (Yes) No

End-of-Day
Range: Auto ✓ Hi ___ Med ___ Low ___
Compensated Setting: (Yes) No

Salinity (ppt)
Actual salinity of solution (ppt): 34.99
Hydrolab salinity reading (ppt): 34.8

End-of-Day
Actual salinity of solution (ppt): 34.99
Hydrolab salinity reading (ppt): 34.6

Turbidity
Stabcal Sol'n #1 2.1 Hydrolab Reading 0.0
Stabcal Sol'n #2 20.0 Hydrolab Reading 20.0
Stabcal Sol'n #3 100.0 Hydrolab Reading 100.0

End-of-Day
Hydrolab Reading 0.0
Hydrolab Reading ~~18.7~~ 19.0
Hydrolab Reading 104

PONCE HARBOR
HYDROLAB CALIBRATION FORM

Client: PPB/AFI
Job No.: 1824-1
H20 Serial No.: 34583
Scout/Surveyor 3 Serial No.: 34684

Date: 03/05/2003
Site: Harbor
Cable Length: 30m

TIME (AST): 1615
Calibrated by: FJ

TIME (AST): 1240 3/6/03
Calibrated by: FJ

Temperature (°C)
Calibration Thermometer(°C) 30.0
Hydrolab Temperature(°C) 29.53

End-of-Day
Calibration Thermometer(°C) 28.0
Hydrolab Temperature(°C) 27.88

Dissolved Oxygen (DO)
Salinity Compensation: (Yes) No
Local Barometric Pressure (in Hg): 760
Multiplied by 25.4(mm Hg) = na
% Saturation: 100.1
Dissolved Oxygen (mg/L): 7.53

End-of-Day
Salinity Compensation: (Yes) No
Local Barometric Pressure (in Hg): FJ ~~n/a~~ 30.07
Multiplied by 25.4(mm Hg) = 760
% Saturation: 101.3
Dissolved Oxygen (mg/L): 7.89

Depth (ft)
Hydrolab depth reading at 0': 0.0
Actual Depth of Sensor (ft): 10.0
Hydrolab depth reading (ft): 10.0

End-of-Day
Hydrolab depth reading at 0': 0.1
Actual Depth of Sensor (ft): 10.0
Hydrolab depth reading (ft): 10.0

pH (su)
Hydrolab Value at pH 7.00 Solution: 7.00
Hydrolab Value at pH 10.0 Solution: 10.0

End-of-Day
Hydrolab Value at pH 7.00 Solution: 7.00
Hydrolab Value at pH 10.0 Solution: 9.99

Conductivity (mmhos/cm)
Range: Auto ✓ Hi___ Med___ Low___
Compensated Setting: (Yes) No

End-of-Day
Range: Auto ✓ Hi___ Med___ Low___
Compensated Setting: (Yes) No

Salinity (ppt)
Actual salinity of solution (ppt): 34.99
Hydrolab salinity reading (ppt): 34.9

End-of-Day
Actual salinity of solution (ppt): 34.99
Hydrolab salinity reading (ppt): 34.7

Turbidity
Stabcal Sol'n #1 <.1 Hydrolab Reading 0.1
Stabcal Sol'n #2 20.0 Hydrolab Reading 20.0
Stabcal Sol'n #3 100 Hydrolab Reading 100

End-of-Day
Hydrolab Reading .4
Hydrolab Reading 20.5
Hydrolab Reading 97.6

Evaluation of Sediment Samples
PONCE HARBOR SEDIMENT SAMPLING
Core Data Log

Date 03/05/2003

Station # E-PH03- 1 SC-A

Time (AST) 1145

Core number 1 of 2

Water depth 38.1

Field team MS, KH, SB, FJ

Project Depth 53' +2' MLW

Coordinates (NAD 83) N 17.9713499°

W 66.6161149°

Silty Clay Depth Ø → 17.5

Fine Sand Depth Ø

Differential Correction Method Beacon

Tidal Height at Time of Collection .46

Tidal Correction to MLW for Vibracore (e.g. at +1' high tide, drill to 56' versus 55' at MLW) ~~30~~ FJ 37.6

Sediment Profile	Description
Top (of sediment surface) Ø	Small trace of fine sand in Ø → 1'
	Dark Grey Silty Clay occas shell pieces sighted
Bottom 17.5'	

Penetration length 17.5'

Recovered core length 17.5'

Sample length retained 17.5'

Collected volume of **Fine Sand** Ø

Collected volume of **Silty Clay** 11 gal.

Live organisms present Y (N)

Oil present Y (N)

Odors observed Y (N)

Achieve project depth (55') (Y) N

Container type Gallon glass jars w/ Teflon –lined lids

Comments

Form Completed by: [signature] **Date/Time:** 03/05/03 1155

This form should be signed and date and time indicated.

Evaluation of Sediment Samples
PONCE HARBOR SEDIMENT SAMPLING
Core Data Log

Date 03/05/2003

Station # E-PH03- 15C-B

Time (AST) 1245

Core number 2 of 2

Water depth 38.1

Field team MS, FJ, KH, SB

Project Depth 53' +2' MLW

Coordinates (NAD 83) N 17.9713499°

W 66.6161149°

Silty Clay Depth 0 → 16'

Fine Sand Depth 0

Differential Correction Method Beacon

Tidal Height at Time of Collection ~~46~~ 29 FJ

Tidal Correction to MLW for Vibracore ~~37.8~~ FJ 8
(e.g. at +1' high tide, drill to 56' versus 55' at MLW)

Sediment Profile	Description
Top (of sediment surface) 0	
	Small amount of fine sand in the 0 → 1'
	depth.
	Dark Gray Silty Clay w/ some occas. shell
	fragments
Bottom 16'	

Penetration length 16.0'

Recovered core length 16.0'

Sample length retained 16.0'

Collected volume of **Fine Sand** 0

Collected volume of **Silty Clay** 8 gal.

Live organisms present Y (N)

Oil present Y (N)

Odors observed Y (N)

Achieve project depth (55') (Y) N

Container type Gallon glass jars w/ Teflon –lined lids

Comments

Form Completed by: [signature] **Date/Time:** 03/05/03 1300

This form should be signed and date and time indicated.

Evaluation of Sediment Samples
PONCE HARBOR SEDIMENT SAMPLING
Core Data Log

Date 03/06/2003

Station # E-PH03- 2 SC-A

Time (AST) 1206

Core number 1 of 1

Water depth 42.5

Field team MS, FJ, KH, SB, CF

Project Depth 53' +2' MLW

Coordinates (NAD 83) N 17.9724440°

W 66.6185062°

Silty Clay Depth 0 → 12'

Fine Sand Depth Ø

Differential Correction Method Beacon

Tidal Height at Time of Collection +.45

Tidal Correction to MLW for Vibracore 42.Ø
(e.g. at +1' high tide, drill to 56' versus 55' at MLW)

Sediment Profile	Description
Top (of sediment surface) Ø	0 → 1.5' Dark Gray Clay Silty
	very light trace of find sand & some shell some moisture content
	1.5' → 12.0' Dark Gray Silty Clay slight moisture content
Bottom 12.0'	

Penetration length 13.5'

Recovered core length 12.0'

Sample length retained 12.0'

Collected volume of **Fine Sand** Ø

Collected volume of **Silty Clay** 6.5 gal.

Live organisms present Y (N)

Oil present Y (N)

Odors observed Y (N)

Achieve project depth (55') (Y) N

Container type Gallon glass jars w/ Teflon –lined lids

Comments

Form Completed by: [signature] **Date/Time:** 03/06/03 1215

This form should be signed and date and time indicated.

Evaluation of Sediment Samples
PONCE HARBOR SEDIMENT SAMPLING
Core Data Log

Date 03/04/2003

Station # E-PH03- 35C-A

Time (AST) ~~14~~ FJ 3/4/03 1505

Core number 1 of 1

Water depth 30.5

Field team MS, FJ, KH, SB

Project Depth 53' +2' MLW

Coordinates (NAD 83) N 17.9713366°

W 66.6231983°

Silty Clay Depth 0 → 18'

Fine Sand Depth n/a

Differential Correction Method Beacon

Tidal Height at Time of Collection +.21

Tidal Correction to MLW for Vibracore 30.~~8~~ 3 FJ
(e.g. at +1' high tide, drill to 56' versus 55' at MLW)

Sediment Profile	Description
Top (of sediment surface)	
	Silty Clay, Moisture Diff near top
	of core Dark Gray Color
Bottom	

Penetration length 20'

Recovered core length 18'

Sample length retained 18'

Collected volume of **Fine Sand** na

Collected volume of **Silty Clay** 12 ~~18~~ FJ gal

Live organisms present Y (N)

Oil present Y (N)

Odors observed Y (N)

Achieve project depth (55') Y (N)

Container type Gallon glass jars w/ Teflon –lined lids

Comments

Form Completed by: FJ **Date/Time:** 03/04/03 ~~14~~ 1505 FJ

This form should be signed and date and time indicated.

Evaluation of Sediment Samples
PONCE HARBOR SEDIMENT SAMPLING
Core Data Log

Date 03/04/2003

Station # E-PH03- 4A SC

Time (AST) 1233

Core number 1 of 1

Water depth 40.3

Field team FJ, MS, SB, KH

Project Depth 53' +2' MLW

Coordinates (NAD 83) N 17.9702283°

W 66.6208283°

Silty Clay Depth 0 → 12'

Fine Sand Depth na

Differential Correction Method Beacon

Tidal Height at Time of Collection .38

Tidal Correction to MLW for Vibracore .38 → 40.0
(e.g. at +1' high tide, drill to 56' versus 55' at MLW)

Sediment Profile	Description
Top (of sediment surface) 0	
	Silty Clay Dark Gray
	Same Consistency Throughout
	Moisture varied toward surface
Bottom 12'	

Penetration length 12'

Recovered core length 12'

Sample length retained 12'

Collected volume of **Fine Sand** n/a

Collected volume of **Silty Clay** 8 gal

Live organisms present Y (N)

Oil present Y (N)

Odors observed Y (N)

Achieve project depth (55') Y (N)

Container type Gallon glass jars w/ Teflon –lined lids

Comments

Form Completed by: FJ **Date/Time:** 3/4/03 1235

This form should be signed and date and time indicated.

Evaluation of Sediment Samples
PONCE HARBOR SEDIMENT SAMPLING
Core Data Log

Date 03/06/2003

Station # E-PH03- 5 SC- A

Time (AST) 1100

Core number 1 of 1

Water depth 34.4

Field team MS, KH, CF, SB, FJ

Project Depth 53' +2' MLW

Coordinates (NAD 83) N 17. 9691600°
W 66.6267300°

Silty Clay Depth 0→19'

Fine Sand Depth Ø

Differential Correction Method Beacon

Tidal Height at Time of Collection .42

Tidal Correction to MLW for Vibracore 34.Ø
(e.g. at +1' high tide, drill to 56' versus 55' at MLW)

Sediment Profile	Description
Top (of sediment surface) Ø	0→2' Dark Gray Silty Clay presence
	of fine sand & shell particle slight
	moisture content
	2'→19' Dark Gray Silty Clay very slight moisture present
Bottom 19.Ø	

Penetration length 19.0

Recovered core length 19.0

Sample length retained 19.0

Collected volume of **Fine Sand** Ø

Collected volume of **Silty Clay** 10 gal.

Live organisms present Y (N)

Oil present Y (N)

Odors observed Y (N)

Achieve project depth (55') (Y) N

Container type Gallon glass jars w/ Teflon –lined lids

Comments Found 1 shell @ 18'

Form Completed by: [signature] **Date/Time:** 03/06/03 1120

This form should be signed and date and time indicated.

Evaluation of Sediment Samples
PONCE HARBOR SEDIMENT SAMPLING
Core Data Log

Date 03/06/2003

Station # E-PH03- 6 SC-A

Time (AST) 0840

Core number 2 of 3

Water depth 39.5

Field team MS, FJ, SB, KH

Project Depth 53' +2' MLW

Coordinates (NAD 83) N 17° 9667966°

W 66° 6231800°

Silty Clay Depth Ø → ~~14~~ 15 14'

Fine Sand Depth Ø

Differential Correction Method Beacon

Tidal Height at Time of Collection .37

Tidal Correction to MLW for Vibracore 39.1
(e.g. at +1' high tide, drill to 56' versus 55' at MLW)

Sediment Profile	Description
Top (of sediment surface) Ø	
	Top 0 → 1 Silty Clay, some shell fragments trace fine sand
	1' → 14' Silty Clay Dark Gray Color moderate to slight moisture content
Bottom 14'	

Penetration length 17.0'

Recovered core length 14.0'

Sample length retained 14.0'

Collected volume of **Fine Sand** Ø

Collected volume of **Silty Clay** 8 gal.

Live organisms present Y (N)

Oil present Y (N)

Odors observed Y (N)

Achieve project depth (55') (Y) N

Container type Gallon glass jars w/ Teflon –lined lids

Comments

Form Completed by: [signature] **Date/Time:** 03/06/03 0850

This form should be signed and date and time indicated.

Evaluation of Sediment Samples
PONCE HARBOR SEDIMENT SAMPLING
Core Data Log

Date 03/06/2003

Station # E-PH03- 6 SC-B

Time (AST) 0905

Core number 3 of 3

Water depth 39.5'

Field team MS, FJ, KH, SB

Project Depth 53' +2' MLW

Coordinates (NAD 83) N 17.9667966°

W 66.6231800°

Silty Clay Depth Ø-12'

Fine Sand Depth Ø

Differential Correction Method Beacon

Tidal Height at Time of Collection .41

Tidal Correction to MLW for Vibracore 39.1
(e.g. at +1' high tide, drill to 56' versus 55' at MLW)

Sediment Profile	Description
Top (of sediment surface) Ø	
	Top 0→1' Dark Gray Silty Clay
	high to moderate moisture content
	some trace of sand & shell fragments
	1'→12' Silty Clay Dark Gray Color
	moderate to slight moisture content
Bottom 12'	

Penetration length 17'

Recovered core length 12'

Sample length retained 12'

Collected volume of **Fine Sand** Ø

Collected volume of **Silty Clay** 7 gal.

Live organisms present Y (N)

Oil present Y (N)

Odors observed Y (N)

Achieve project depth (55') (Y) N

Container type Gallon glass jars w/ Teflon –lined lids

Comments

Form Completed by: [signature] **Date/Time:** 03/06/03 0930

This form should be signed and date and time indicated.

Evaluation of Sediment Samples
PONCE HARBOR SEDIMENT SAMPLING
Core Data Log

Date 03/06/2003

Station # E-PH03-~~6~~ F3 6 SC

Time (AST) 0820

Core number 1 of 3

Water depth 39.5

Field team MS, FJ, KH, SB

Project Depth 53' +2' MLW

Coordinates (NAD 83) N 17.9667966°

W 66.6231800°

Silty Clay Depth Ø → 4'

Fine Sand Depth Ø

Differential Correction Method Beacon

Tidal Height at Time of Collection .37

Tidal Correction to MLW for Vibracore 39.1
(e.g. at +1' high tide, drill to 56' versus 55' at MLW)

Sediment Profile	Description
Top (of sediment surface) Ø	
	Dark Gray Silty Clay High
	Moisture Content
	Did Not Process this core
	Not Enough Material
Bottom	

Penetration length 15'

Recovered core length 4'

Sample length retained Ø

Collected volume of **Fine Sand** Ø

Collected volume of **Silty Clay** Ø

Live organisms present Y (N)

Oil present Y (N)

Odors observed Y (N)

Achieve project depth (55') (Y) N

Container type Gallon glass jars w/ Teflon –lined lids

Comments Did not process this sample

Form Completed by: [signature] **Date/Time:** 03/06/03 0825

This form should be signed and date and time indicated.

Evaluation of Sediment Samples
PONCE HARBOR SEDIMENT SAMPLING
Core Data Log

Date 03/06/2003

Station # E-PH03- 7 SC-A

Time (AST) 0950

Core number 1 of 1

Water depth 39.5

Field team MS, KH, SB, FJ, CF

Project Depth 53' +2' MLW

Coordinates (NAD 83) N 17.9668416°
W 66.6278449°

Silty Clay Depth 0 → 15'

Fine Sand Depth Ø

Differential Correction Method Beacon

Tidal Height at Time of Collection + .43

Tidal Correction to MLW for Vibracore 39.0
(e.g. at +1' high tide, drill to 56' versus 55' at MLW)

Sediment Profile	Description
Top (of sediment surface) Ø	
	0-2' Dark Gray Silty Clay presence of
	fine sand & shell particles moderate
	moisture content
	2' → 15' Dark Gray Silty Clay slight moisture
	content
Bottom 15'	

Penetration length 17.0'

Recovered core length 15'

Sample length retained 15'

Collected volume of **Fine Sand** Ø

Collected volume of **Silty Clay** 8 gal.

Live organisms present Y (N)

Oil present Y (N)

Odors observed Y (N)

Achieve project depth (55') (Y) N

Container type Gallon glass jars w/ Teflon –lined lids

Comments

Form Completed by: [signature] **Date/Time:** 03/06/03 1005

This form should be signed and date and time indicated.

Evaluation of Sediment Samples
PONCE HARBOR SEDIMENT SAMPLING
Core Data Log

Date 03/05/2003

Station # E-PH03- 8 SC-A

Time (AST) 1005

Core number 1 of 2

Water depth 40.5'

Field team MS, FJ, KH, FJ

Project Depth 53' +2' MLW

Coordinates (NAD 83) N 17.9645850° W 66.6255333°

Silty Clay Depth 0 → 13.5'

Fine Sand Depth Ø

Differential Correction Method Beacon

Tidal Height at Time of Collection .45'

Tidal Correction to MLW for Vibracore 40.0'
(e.g. at +1' high tide, drill to 56' versus 55' at MLW)

Sediment Profile	Description
Top (of sediment surface) Ø	
	Dark Gray Silty Clay
	Very small trace of fine sand @ top 1.5'
Bottom 13.5'	

Penetration length 16'

Recovered core length 13.5'

Sample length retained 13.5'

Collected volume of **Fine Sand** Ø

Collected volume of **Silty Clay** 8 gal

Live organisms present Y (N)

Oil present Y (N)

Odors observed Y (N)

Achieve project depth (55') (Y) N

Container type Gallon glass jars w/ Teflon –lined lids

Comments

Form Completed by: [signature] **Date/Time:** 03/05/03 1015

This form should be signed and date and time indicated.

Evaluation of Sediment Samples
PONCE HARBOR SEDIMENT SAMPLING
Core Data Log

Date 03/05/2003

Station # E-PH03- 8 SC-B

Time (AST) 1040

Core number 2 of 2

Water depth 40.5'

Field team MS, KH, FJ, SB

Project Depth 53' +2' MLW

Coordinates (NAD 83) N 17.9645850°

W 66.6255333°

Silty Clay Depth Ø → 12.5'

Fine Sand Depth —

Differential Correction Method Beacon

Tidal Height at Time of Collection .45'

Tidal Correction to MLW for Vibracore 40.0'
(e.g. at +1' high tide, drill to 56' versus 55' at MLW)

Sediment Profile	Description
Top (of sediment surface) Ø	
	Dark Gray Silty Clay
	Very light trace of fine sand
	in top 1.5'
Bottom 12.5'	

Penetration length 16.0'

Recovered core length 12.5

Sample length retained 12.5

Collected volume of **Fine Sand** Ø

Collected volume of **Silty Clay** 7 gal

Live organisms present Y (N)

Oil present Y (N)

Odors observed Y (N)

Achieve project depth (55') (Y) N

Container type Gallon glass jars w/ Teflon –lined lids

Comments WOOD WAS OBSERVED IN THE CORE AT A DEPTH OF 7.5'

Form Completed by: [signature] **Date/Time:** 03/05/03 1100

This form should be signed and date and time indicated.

Evaluation of Sediment Samples
PONCE HARBOR SEDIMENT SAMPLING
Core Data Log

Date 03/05/2003

Station # E-PH03- 9SC-A

Time (AST) 0851

Core number 1 of 2

Water depth 48.5'

Field team MS, KH, FS, SB, CF

Project Depth 53' +2' MLW

Coordinates (NAD 83) N 17.9623099° W 66.6256166°

Silty Clay Depth 0 → 11.5'

Fine Sand Depth Ø

Differential Correction Method Beacon

Tidal Height at Time of Collection .39'

Tidal Correction to MLW for Vibracore 48.1'
(e.g. at +1' high tide, drill to 56' versus 55' at MLW)

Sediment Profile	Description
Top (of sediment surface) 0'	↑
	Silty Clay throughout
	Dark Gray Color
	Moist Texture
	↓
Bottom 11.5'	

Penetration length 18'

Recovered core length 11.5'

Sample length retained 7.0'

Collected volume of **Fine Sand** Ø

Collected volume of **Silty Clay** 4 gal

Live organisms present Y (N)

Oil present Y (N)

Odors observed Y (N)

Achieve project depth (55') (Y) N

Container type Gallon glass jars w/ Teflon –lined lids

Comments

Form Completed by: [signature] **Date/Time:** 03/05/03

This form should be signed and date and time indicated.

Evaluation of Sediment Samples
PONCE HARBOR SEDIMENT SAMPLING
Core Data Log

Date 03/05/2003

Station # E-PH03- 9 SC-B

Time (AST) 0918

Core number 2of2

Water depth 48.5'

Field team MS, SB, FJ, KH

Project Depth 53' +2' MLW

Coordinates (NAD 83) N 17.9623099°

W 66.6256166°

Silty Clay Depth 0 → 12'

Fine Sand Depth 0

Differential Correction Method Beacon

Tidal Height at Time of Collection .39'

Tidal Correction to MLW for Vibracore 48.1'
(e.g. at +1' high tide, drill to 56' versus 55' at MLW)

Sediment Profile	Description
Top (of sediment surface) 0	
	Same as Core A
	Silty Clay Dark Gray Color
7.0'	
Bottom	

Penetration length 17.5

Recovered core length 12.0

Sample length retained 7.0

Collected volume of **Fine Sand** 0

Collected volume of **Silty Clay** 4 gal

Live organisms present Y (N)

Oil present Y (N)

Odors observed Y (N)

Achieve project depth (55') (Y) N

Container type Gallon glass jars w/ Teflon –lined lids

Comments Roosfelder

Form Completed by: [signature] **Date/Time:** 03/05/03 0930

This form should be signed and date and time indicated.

FJ Taxi Amex
Tip 10 — cash

Sunday 03/02/03

0630 TSh, MS, AS, EH lv Jupiter → Ft Laud

0815 FJ lv Orlando → Orlando Int

1630 3 vehicles (1 w/ FA, 1 w/ AS, EH box truck, 1 w/ SB, TSh van) leave SJ after picking up equip in warehouse begin transit to Ponce

1830 Ar Ponce

FJ Bfast — Room Charge
EH Dinner 4 - Cash

Monday 03/03/03

0700 Lv hotel transit to Port of Ponce

1930 CSA crew leaves port mob 75% complete

Bfast FJ Hotel Charge
EH - Dinner - SCC

Tuesday 03/04/03

0730 Ar port begin crew badging for security, equip cleaning, equip cal, & final preps

1155 Lv dock for E-PH03-4

1210 Anchored @ stat 4

1215 Begin H lab cast

1230 Begin V.C. @ E-PH03-4SC-A

1356 Complete VC sampling, processing & cleaning @ E-PH03-4SC-A begin ~~processing @~~ transit to E-PH03-3

1420 Anchored / Spuds set @ E-PH03-3

Tuesday 03/04/03 (cont'd)

1425 H lab cast to mid depth 15' @ this station
preping V.C. for deployment

1500 Begin V.C sampling @ E-PH03-3

1510 V.C #1 @ E-PH03-3 on deck penetrate
to 20'

Note Prior to sampling @ #3 MS called MR
to inform him of depth in relation to
position, vessel moored @ station but
depth 30' instead of expected 40 MR
ok us to sample the position not to
worry about depth. MR also indicated
20' penetration w/ 18' retainage was acceptable.

1634 Complete sampling, processing, & cleaning
@ E-PH03-3SC-A

1640 Begin transit to dock

1705 Ar dock begin sample re ice & storage
equip post & pre cal for a.m sampling
& equip prep for a.m sampling
CF/MS spoke w/ M Riese again to
tell him that reference sta B was very
difficult to sample and told CF to go
back and collect B sample @ A site

Tuesday 03/04/03

1830 Ops Cease, lv dock transit to hotel

FJ Breakfast Change Hotel

Wednesday 03/05/03

0715 Ar dock begin re-icing samples for yesterday
equip prep nav set up, etc

0745 Begin transit from dock to E-PH03-9

0805 Ar & spuds set @ site E-PH03-9 have some
problem w/ block rigging on crane

0830 H lab cast complete

0845 Crane rigging complete begin V.C. prep

0850 Begin V.C sampling @ E-PH03-9

0858 Core 1 @ E-PH03-9 on deck

0905 Begin processing E-PH03-9 SC-A & rigging
for next core

0918 Core 2 @ E-PH03-9 SC-B

0925 Core on deck begin processing, pulling spuds
& setting buoy @ E-PH03-8

1000 Spuds set & begin H lab cast @ E-PH03-8

1005 Begin V.C sampling @ E-PH03-8

1000 Core #1 @ E-PH03-8 SC-A

1015 Core #1 on deck begin processing

1040 Core #2 sampled @ E-PH03-8 SC-B

Wednesday 03/05/03 (cont'd)

1040 Begin processing, pulling spuds, cleaning @ E-PH03-8SCB
MS called Mr. Lopez to inform him of operational status. MS called Ferdinand Q. to inform him that stations 9 & 8 were complete.

1125 Spuds set @ E-PH03-1

1130 H lab cast @ E-PH03-1

1144 Core #1 @ E-PH03-1SC-A begin processing

1245 Core #2 @ E-PH03-1SC-B

1300 Core #2 on deck begin processing & transit to dock to offload samples & process for shipment via Fed Ex

1305 At dock begin sample prep

1530 Samples iced, inventoried, & p/u by Fed Ex
20 coolers sent to PPB Lab

1535 Begin H lab post & pre cal

1650 Cal complete

1700 Begin off loading samples from Kruger B into Box Truck for storage

1730 Kruger off loaded, reloaded Kruger B freezer w/ ice begin SeaBird prep for a.m sampling

1830 Ops Cease transit → hotel

Thursday 03/06/03

0700 Av dock begin cooler & ice mob, nav setup, equip. prep, sample prep, etc

0725 Kruger B leaves dock transit to ODMDS

0755 Barge Mobro lv dock transit to E-PH03-6

0810 Barge spuds set @ E-PH03-6

0815 H lab cast

0820 Core # 1 @ E-PH03-6

0830 Not enough retention of core # 1 to keep

0840 Core # 2 @ E-PH03-6 SC-A

0905 Core # 3 @ E-PH03-6 SC-B

0915 Transit to site E-PH03-7

0942 H lab cast @ E-PH03-7

0950 Core # 1 @ E-PH03-7 SC-A

0955 # 1 on deck begin processing

1020 Begin transit to E-PH03-5

1042 H lab cast @ E-PH03-5

1100 Core # 1 @ E-PH03-5 SC-A

1115 Core on deck begin transit to E-PH03-2 & processing of E-PH03-5 SC-A

1200 H. lab cast @ E-PH03-2

1206 Core # 1 @ E-PH03-2 begin processing, h lab post cal, sample shipment prep, icing, labeling, etc

Thursday 3/6/03 (cont'd)

1515 Samples preped waiting for Fed Ex to arrive
begin breaking down equip on barge

1545 Fed Ex samples picked up cont. barge de-mob

1930 Samples sent off

Friday

0700 Lv hotel transit → dock

0800 Begin reicing coolers, demob etc, H_2O Sample Prep

0855 Begin water sample collection @ E-PH03-1W

0905 H_2O sampling complete begin sample prep
for shipment & vessel demob, equip

1630 Fed Ex p/u

1800 Ar freight ready forms filled out

1800 Lv port transit to hotel

Saturday

0800 Lv hotel transit to San Juan

1000 Ar S.J.U d/o S.B. flight → Newark

1200 Remaining crew ar. SJU

1330 Flight → Ft. Laud

1800 Ar CSA Jupiter

1 of 4

ppb
ENVIRONMENTAL LABORATORIES, INC.
6821 SW Archer Road, Gainesville, FL 32608
(352) 377-2349 · FAX (352) 395-6639

CHAIN-OF-CUSTODY RECORD

LABTROL CLIENT No.	BATCH NO.	FOR LAB USE ONLY
		Subcontractor
		Shipping Method

CLIENT NAME: AFI

CLIENT PROJECT: PONCE HARBOR

SITE NAME & ADDRESS: PONCE, PUERTO RICO OFFSHORE REF.

SAMPLERS: (Signature) Bryan Cotter BC

LAB REPORT GOES TO (Client contact person):

IDENTIFY PARAMETERS DESIRED AND NO. OF CONTAINERS

PRESERVATION
CF Chilled-Filtered
SF Sulfuric-Filtered
NF Nitric-Filtered
C Chilled
S Sulfuric
N Nitric
B Basic/NaOH
Z Zinc
T Thiosulfate
H HCL
Ot Other (see Remarks)

NUMBER	DATE	TIME	COMP.	GRAB	STATION LOCATION / NUMBER	SAMPLE MATRIX	NUMBER OF CONTAINERS	AS PER CONTRACT	APPROX TTL (GALLONS)						Lab I.D. Number
1	3/4/03	12:57		✓	RS-PH03-A	S	1	✓	1						
2				✓	RS-PH03-A	S	1	✓	1						
3				✓	RS-PH03-A	S	1	✓	1						
4				✓	RS-PH03-A	S	1	✓	1						
5				✓	RS-PH03-A	S	1	✓	1						
6				✓	RS-PH03-A	S	1	✓	1						
7				✓	RS-PH03-A	S	1	✓	1						
8				✓	RS-PH03-A	S	1	✓	1						
9				✓	RS-PH03-A	S	1	✓	1						
10				✓	RS-PH03-A	S	1	✓	1						
11				✓	RS-PH03-A	S	1	✓	1						
12				✓	RS-PH03-A	S	1	✓	1						
13				✓	RS-PH03-A	S	1	✓	1						
14				✓	RS-PH03-A	S	1	✓	1						
15				✓	RS-PH03-A	S	1	✓	1						

Precleaned Containers Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Date / Time
Relinquished by: (Signature) Kurt W. Konrad KWH	Date / Time 3/5/03 15:30	Received by: (Signature) FED EX	Date / Time 3/5/03 15:30
Relinquished by: (Signature) Fed Ex	Date / Time 3/6/03 1020	Received by: (Signature) [signature]	Date / Time 3/6/03 1025
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Date / Time

Remarks and Observations

Matrix Types S Soil or Sediment WW Wastewater T Animal Tissue SW Surface Water SL Sludge or Solid Waste MW Marine Water GW Ground Water DW Drinking Water P Plant Tissue F Filter A Air Ot Other (See Remarks)

2 of 4 TAB

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(352) 377-2349 · FAX (352) 395-6639

CHAIN-OF-CUSTODY RECORD

LABTROL CLIENT No.	BATCH NO.	FOR LAB USE ONLY
		Subcontractor
		Shipping Method

CLIENT NAME: AFI

CLIENT PROJECT: PONCE HARBOR

SITE NAME & ADDRESS: PONCE, PUERTO RICO OFFSHORE REF.

SAMPLERS: (Signature) Bryan Cotter

LAB REPORT GOES TO (Client contact person):

IDENTIFY PARAMETERS DESIRED AND NO. OF CONTAINERS: AS PER CONTRACT; APPROX. TTL (GALLONS)

PRESERVATION: CF Chilled-Filtered; SF Sulfuric-Filtered; NF Nitric-Filtered; C Chilled; S Sulfuric; N Nitric; B Basic/NaOH; Z Zinc; T Thiosulfate; H HCL; Ot Other (see Remarks)

NUMBER	DATE	TIME	COMP.	GRAB	STATION LOCATION / NUMBER	SAMPLE MATRIX	NUMBER OF CONTAINERS	AS PER CONTRACT	APPROX. TTL (GALLONS)	Lab I.D. Number
16	3/4/03	12:54		✓	RS-PH03-A	S	1	✓	1	
17				✓	RS-PH03-A	S	1	✓	1	
18				✓	RS-PH03-A	S	1	✓	1	
19				✓	RS-PH03-A	S	1	✓	1	
20				✓	RS-PH03-A	S	1	✓	1	
21				✓	RS-PH03-A	S	1	✓	1	
				✓	RS-PH03-A	S	1	✓	1	
				✓	RS-PH03-A	S	1	✓	1	
				✓	RS-PH03-A	S	1	✓	1	
				✓	RS-PH03-A	S	1	✓	1	

Precleaned Containers Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Date / Time
Relinquished by: (Signature) Kurt W. Edwards KWH	Date / Time 3/5/03 15:30	Received by: (Signature) FED EX	Date / Time 3/5/03 15:30
Relinquished by: (Signature) Fed Ex	Date / Time 3/6/03 1020	Received by: (Signature) [illegible]	Date / Time 3/6/03 1025
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Date / Time

Remarks and Observations

Matrix Types S Soil or Sediment; WW Wastewater; T Animal Tissue; SW Surface Water; SL Sludge or Solid Waste; MW Marine Water; GW Ground Water; DW Drinking Water; P Plant Tissue; F Filter; A Air; Ot Other (See Remarks)

3 of 84 THAS

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(352) 377-2349 · FAX (352) 395-6639

CHAIN-OF-CUSTODY RECORD

LABTROL CLIENT No.	BATCH NO.	FOR LAB USE ONLY
		Subcontractor
		Shipping Method

CLIENT NAME: AFI

CLIENT PROJECT: PONCE HABOR

SAMPLERS: (Signature): Bryan Cotten BC

SITE NAME & ADDRESS: PONCE, PUERTO RICO OFFSHORE REF.

LAB REPORT GOES TO (Client contact person):

IDENTIFY PARAMETERS DESIRED AND NO. OF CONTAINERS: AC PER CONTRACT; APPROX. TOTAL (GALL.)

PRESERVATION: CF Chilled-Filtered; SF Sulfuric-Filtered; NF Nitric-Filtered; C Chilled; S Sulfuric; N Nitric; B Basic/NaOH; Z Zinc; T Thiosulfate; H HCL; Ot Other (see Remarks)

NUMBER	DATE	TIME	COMP.	GRAB	STATION LOCATION / NUMBER	SAMPLE MATRIX	NUMBER OF CONTAINERS	AC PER CONTRACT	APPROX. TOTAL (GALL.)	Lab I.D. Number
	3/4/03	12:57		✓	RS-PH03-1A	S	1	✓	1	

Precleaned Containers Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Date / Time
Relinquished by: (Signature) Kurt W. Hamel Kurt	Date / Time 3/5/03 15:30	Received by: (Signature) FED EX	Date / Time 3/5/03 15:30
Relinquished by: (Signature) Fed Ex	Date / Time 3/6/03 1020	Received by: (Signature) TW [illegible]	Date / Time 3/6/03 1025
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Date / Time

Remarks and Observations

Matrix Types S Soil or Sediment; WW Wastewater; T Animal Tissue; SW Surface Water; SL Sludge or Solid Waste; MW Marine Water; GW Ground Water; DW Drinking Water; P Plant Tissue; F Filter; A Air; Ot Other (See Remarks)

4 of 6 9+ms

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CHAIN-OF-CUSTODY RECORD

LABTROL CLIENT No.	BATCH NO.	FOR LAB USE ONLY
		Subcontractor _____
		Shipping Method _____

CLIENT NAME: AFI

CLIENT PROJECT: PONCE HARBOR

SITE NAME & ADDRESS: PONCE, PUERTO RICO HARBOR

SAMPLERS: (Signature) Kurt Hamel KWH

LAB REPORT GOES TO (Client contact person):

IDENTIFY PARAMETERS DESIRED AND NO. OF CONTAINERS

PRESERVATION
CF Chilled-Filtered
SF Sulfuric-Filtered
NF Nitric-Filtered
C Chilled
S Sulfuric
N Nitric
B Basic/NaOH
Z Zinc
T Thiosulfate
H HCL
Ot Other (see Remarks)

NUMBER	DATE	TIME	COMP.	GRAB	STATION LOCATION / NUMBER	SAMPLE MATRIX	NUMBER OF CONTAINERS	As Per Contract		Approx. Total Gal.						Lab I.D. Number
	03/04/03	~~13:15~~ 12:33 KH 3/4/03		✓	E-PH 03-4SC-A	S	1	✓		1						
				✓	E-PH 03-4SC-A	S	1	✓		1						
				✓	E-PH 03-4SC-A	S	1	✓		1						
				✓	E-PH03-4SC-A	S	1	✓		1						
				✓	E-PH03-4SC-A	S	1	✓		1						
				✓	E-PH03-4SC-A	S	1	✓		1						
				✓	E-PH03-4SC-A	S	1	✓		1						
				✓	E-PH03-4SC-A	S	1	✓		1						
		15:05		✓	E-PH03-3SC-A	S	1	✓		1						
				✓	E-PH03-3SC-A	S	1	✓		1						
				✓	E-PH03-3SC-A	S	1	✓		1						
				✓	E-PH03-3SC-A	S	1	✓		1						
				✓	E-PH03-3SC-A	S	1	✓		1						
				✓	E-PH03-3SC-A	S	1	✓		1						

Precleaned Containers Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Date / Time
Relinquished by: (Signature) Kurt W. Hamel	Date / Time 3/5/03 15:30	Received by: (Signature) FED EX	Date / Time 3/5/03 15:30
Relinquished by: (Signature) FedEx	Date / Time 3/6/03 1023	Received by: (Signature) [signature]	Date / Time 3/6/03 1025
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Date / Time

Remarks and Observations

Matrix Types S Soil or Sediment WW Wastewater T Animal Tissue SW Surface Water SL Sludge or Solid Waste MW Marine Water GW Ground Water DW Drinking Water P Plant Tissue F Filter A Air Ot Other (See Remarks)

5 of 8 & 9 TMB

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(352) 377-2349 · FAX (352) 395-6639

CHAIN-OF-CUSTODY RECORD

LABTROL CLIENT No.	BATCH NO.	FOR LAB USE ONLY
		Subcontractor ____
		Shipping Method ____

CLIENT NAME: AFI

CLIENT PROJECT: PONCE HARBOR

SITE NAME & ADDRESS: PONCE, PUERTO RICO HARBOR

SAMPLERS: (Signature) Kurt Hamel KWH

LAB REPORT GOES TO (Client contact person):

IDENTIFY PARAMETERS DESIRED AND NO. OF CONTAINERS

PRESERVATION
CF Chilled-Filtered
SF Sulfuric-Filtered
NF Nitric-Filtered
C Chilled
S Sulfuric
N Nitric
B Basic/NaOH
Z Zinc
T Thiosulfate
H HCL
Ot Other (see Remarks)

NUMBER	DATE	TIME	COMP.	GRAB	STATION LOCATION / NUMBER	SAMPLE MATRIX	NUMBER OF CONTAINERS	As Rex Con Trace		Approx Total Gall						Lab I.D. Number
	03/4/03	15:05		✓	E-PH03-3SC-A	S	1	✓		1						
				✓	E-PH03-3SC-A	S	1	✓		1						
				✓	E-PH03-3SC-A	S	1	✓		1						
				✓	E-PH03-3SC-A	S	1	✓		1						
				✓	E-PH03-3SC-A	S	1	✓		1						
				✓	E-PH03-3SC-A	S	1	✓		1						

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Date / Time
Precleaned Containers Relinquished by: (Signature)			
Kurt W. Hamel	3/5/03 15:30	FED EX	3/5/03 15:30
Fed Ex	3/6/03 1020	TMBell	3/6/03 1025

Remarks and Observations

Matrix Types S Soil or Sediment WW Wastewater T Animal Tissue SW Surface Water SL Sludge or Solid Waste MW Marine Water GW Ground Water DW Drinking Water P Plant Tissue F Filter A Air Ot Other (See Remarks)

YMB

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CHAIN-OF-CUSTODY RECORD

LABTROL CLIENT No.	BATCH NO.	FOR LAB USE ONLY
		Subcontractor
		Shipping Method

CLIENT NAME: AFI

CLIENT PROJECT: PONCE HARBOR

SITE NAME & ADDRESS: PONCE, PUERTO RICO HARBOR

SAMPLERS: (Signature) Kurt Howell KWH

LAB REPORT GOES TO (Client contact person):

IDENTIFY PARAMETERS DESIRED AND NO. OF CONTAINERS: As Per Contract; Approx. Total (Gallons)

PRESERVATION
CF Chilled-Filtered
SF Sulfuric-Filtered
NF Nitric-Filtered
C Chilled
S Sulfuric
N Nitric
B Basic/NaOH
Z Zinc
T Thiosulfate
H HCL
Ot Other (see Remarks)

NUMBER	DATE	TIME	COMP.	GRAB	STATION LOCATION / NUMBER	SAMPLE MATRIX	NUMBER OF CONTAINERS	As Per Contract		Approx. Total (Gallons)						Lab I.D. Number
	3/5/03	08:51		✓	E-PH03-9SC-A	S	1	✓		1						
				✓	E-PH03-9SC-A	S	1	✓		1						
				✓	E-PH03-9SC-A	S	1	✓		1						
				✓	E-PH03-9SC-A	S	1	✓		1						
	3/5/03	09:15		✓	E-PH03-9SC-B	S	1	✓		1						
				✓	E-PH03-9SC-B	S	1	✓		1						
				✓	E-PH03-9SC-B	S	1	✓		1						
				✓	E-PH03-9SC-B	S	1	✓		1						
	3/5/03	10:05		✓	E-PH03-8SC-~~B~~A KWH 3/5/03	S	1	✓		1						
				✓	E-PH03-8SC-A	S	1	✓		1						
				✓	E-PH03-8SC-A	S	1	✓		1						
				✓	E-PH03-8SC-A	S	1	✓		1						
				✓	E-PH03-8SC-A	S	1	✓		1						

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Date / Time
Precleaned Containers Relinquished by: (Signature)			
Kurt W. Howell	3/5/03 15:30	FED EX	3/5/03 15:30
Fed Ex	3/6/03 1020	TM Zul	3/6/03 1025

Remarks and Observations

Matrix Types S Soil or Sediment WW Wastewater T Animal Tissue SW Surface Water SL Sludge or Solid Waste MW Marine Water GW Ground Water DW Drinking Water P Plant Tissue F Filter A Air Ot Other (See Remarks)

7 of 9 TWB

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6821 SW Archer Road, Gainesville, FL 32608
(352) 377-2349 · FAX (352) 395-6639

CHAIN-OF-CUSTODY RECORD

LABTROL CLIENT No.	BATCH NO.	FOR LAB USE ONLY
		Subcontractor
		Shipping Method

CLIENT NAME: AFI

CLIENT PROJECT: PONCE HARBOR

SAMPLERS: (Signature) Kurt W. Hansel KWH

SITE NAME & ADDRESS: PONCE, PUERTO RICO HARBOR

LAB REPORT GOES TO (Client contact person):

IDENTIFY PARAMETERS DESIRED AND NO. OF CONTAINERS

PRESERVATION

CF	Chilled-Filtered
SF	Sulfuric-Filtered
NF	Nitric-Filtered
C	Chilled
S	Sulfuric
N	Nitric
B	Basic/NaOH
Z	Zinc
T	Thiosulfate
H	HCL
Ot	Other (see Remarks)

NUMBER	DATE	TIME	COMP.	GRAB	STATION LOCATION / NUMBER	SAMPLE MATRIX	NUMBER OF CONTAINERS	AS PER CONTRACT		APPROX TOTAL (GALLON)						Lab I.D. Number
	3/5/03	10:05		✓	E-PH03-8SC-A	S	1	✓		1						
				✓	E-PH03-8SC-A	S	1	✓		1						
				✓	E-PH03-8SC-A	S	1	✓		1						
	3/5/03	10:40		✓	E-PH03-8SC-B	S	1	✓		1						
				✓	E-PH03-8SC-B	S	1	✓		1						
				✓	E-PH03-8SC-B	S	1	✓		1						
				✓	E-PH03-8SC-B	S	1	✓		1						
				✓	E-PH03-8SC-B	S	1	✓		1						
				✓	E-PH03-8SC-B	S	1	✓		1						
				✓	E-PH03-8SC-B	S	1	✓		1						
	3/5/03	11:45		✓	E-PH03-1SC-A	S	1	✓		1						
				✓	E-PH03-1SC-A	S	1	✓		1						
				✓	E-PH03-1SC-A	S	1	✓		1						

Precleaned Containers Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Date / Time
Relinquished by: (Signature) Kurt W. Hansel	Date / Time 3/5/03 15:30	Received by: (Signature) FED EX	Date / Time 3/5/03 15:30
Relinquished by: (Signature) Fed Ex	Date / Time 3/6/03 1020	Received by: (Signature) [signature]	Date / Time 3/6/03 1025
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Date / Time

Remarks and Observations

Matrix Types S Soil or Sediment WW Wastewater T Animal Tissue SW Surface Water SL Sludge or Solid Waste MW Marine Water GW Ground Water DW Drinking Water P Plant Tissue F Filter A Air Ot Other (See Remarks)

8 of 9 tms

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ENVIRONMENTAL LABORATORIES, INC.
6821 SW Archer Road, Gainesville, FL 32608
(352) 377-2349 · FAX (352) 395-6639

CHAIN-OF-CUSTODY RECORD

LABTROL CLIENT No.	BATCH NO.	FOR LAB USE ONLY
		Subcontractor ____
		Shipping Method ____

CLIENT NAME: AFI

CLIENT PROJECT: PONCE HARBOR

SITE NAME & ADDRESS: PONCE, PUERTO RICO HARBOR

SAMPLERS: (Signature) Kent W. Hamel

LAB REPORT GOES TO (Client contact person):

IDENTIFY PARAMETERS DESIRED AND NO. OF CONTAINERS: As per contract; Approx. Total (Grab)

PRESERVATION: CF Chilled-Filtered; SF Sulfuric-Filtered; NF Nitric-Filtered; C Chilled; S Sulfuric; N Nitric; B Basic/NaOH; Z Zinc; T Thiosulfate; H HCL; Ot Other (see Remarks)

NUMBER	DATE	TIME	COMP.	GRAB	STATION LOCATION / NUMBER	SAMPLE MATRIX	NUMBER OF CONTAINERS	As per contract	Approx. Total (Grab)						Lab I.D. Number
	3/5/03	11:45		✓	E-PH03-1SC-A	S	1	✓	1						
				✓	E-PH03-1SC-A	S	1	✓	1						
				✓	E-PH03-1SC-A	S	1	✓	1						
				✓	E-PH03-1SC-A	S	1	✓	1						
				✓	E-PH03-1SC-A	S	1	✓	1						
				✓	E-PH03-1SC-A	S	1	✓	1						
				✓	E-PH03-1SC-A	S	1	✓	1						
				✓	E-PH03-1SC-A	S	1	✓	1						
	3/5/03	12:45		✓	E-PH03-1SC-B	S	1	✓	1						
				✓	E-PH03-1SC-B	S	1	✓	1						
				✓	E-PH03-1SC-B	S	1	✓	1						
				✓	E-PH03-1SC-B	S	1	✓	1						
				✓	E-PH03-1SC-B	S	1	✓	1						
				✓	E-PH03-1SC-B	S	1	✓	1						

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Date / Time
Precleaned Containers Relinquished by: (Signature)			
Kent W. Hamel	3/5/03 15:30	FED EX	3/5/03 15:30
Fed Ex	3/6/03 1020	[illegible]	3/6/03 1025

Remarks and Observations

Matrix Types S Soil or Sediment; WW Wastewater; T Animal Tissue; SW Surface Water; SL Sludge or Solid Waste; MW Marine Water; GW Ground Water; DW Drinking Water; P Plant Tissue; F Filter; A Air; Ot Other (See Remarks)

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(352) 377-2349 · FAX (352) 395-6639

CHAIN-OF-CUSTODY RECORD

LABTROL CLIENT No.	BATCH NO.	FOR LAB USE ONLY
		Subcontractor ____
		Shipping Method ____

CLIENT NAME: AFI

CLIENT PROJECT: PONCE HARBOR

SITE NAME & ADDRESS: PONCE, PUERTO RICO HARBOR

SAMPLERS: (Signature) Kurt W. Hamel KWH

LAB REPORT GOES TO (Client contact person):

IDENTIFY PARAMETERS DESIRED AND NO. OF CONTAINERS: AS PER CONTRACT; APPROX. TOTAL (GAL.)

PRESERVATION

CF	Chilled-Filtered
SF	Sulfuric-Filtered
NF	Nitric-Filtered
C	Chilled
S	Sulfuric
N	Nitric
B	Basic/NaOH
Z	Zinc
T	Thiosulfate
H	HCL
Ot	Other (see Remarks)

NUMBER	DATE	TIME	COMP.	GRAB	STATION LOCATION / NUMBER	SAMPLE MATRIX	NUMBER OF CONTAINERS	AS PER CONTRACT		APPROX. TOTAL (GAL.)						Lab I.D. Number
	3/5/03	12:45		✓	E-PH03-1SC-B	S	1	✓		1						
	1	1		✓	E-PH03-1SC-B	S	1	✓		1						

Precleaned Containers Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Date / Time
Relinquished by: (Signature) Kurt W Hamel	Date / Time 3/5/03 15:30	Received by: (Signature) FED EX	Date / Time 3/5/03 15:30
Relinquished by: (Signature) Fed Ex	Date / Time 3/6/03 1020	Received by: (Signature) TM [illegible]	Date / Time 3/6/03 1025
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Date / Time

Remarks and Observations

Matrix Types S Soil or Sediment WW Wastewater T Animal Tissue SW Surface Water SL Sludge or Solid Waste MW Marine Water GW Ground Water DW Drinking Water P Plant Tissue F Filter A Air Ot Other (See Remarks)

Sediment samples to Mactec / ESE

ppb
ENVIRONMENTAL LABORATORIES, INC.
6821 SW Archer Road, Gainesville, FL 32608
(352) 377-2349 · FAX (352) 395-6639

CHAIN-OF-CUSTODY RECORD

LABTROL CLIENT No.	BATCH NO.	FOR LAB USE ONLY / Subcontractor MACTEC/ESE / Shipping Method

CLIENT NAME: AFI - Ponce Harbor

CLIENT PROJECT: Ponce Harbor

SITE NAME & ADDRESS: Ponce, Puerto Rico

SAMPLERS: (Signature) CSA/PPB

LAB REPORT GOES TO (Client contact person):

IDENTIFY PARAMETERS DESIRED AND NO. OF CONTAINERS: As per contract

PRESERVATION

CF	Chilled-Filtered
SF	Sulfuric-Filtered
NF	Nitric-Filtered
C	Chilled
S	Sulfuric
N	Nitric
B	Basic/NaOH
Z	Zinc
T	Thiosulfate
H	HCL
Ot	Other (see Remarks)

NUMBER	DATE	TIME	COMP.	GRAB	STATION LOCATION / NUMBER	SAMPLE MATRIX	NUMBER OF ~~CONTAINERS~~ Coolers	Lab I.D. Number
	3/4/03	1257	✓		RS-PH03	S	8	
	3/4/03	1233	✓		E-PH03-SC	S	5	

Precleaned Containers Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Date / Time
	3/10/03 1200		
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Date / Time
	3/11/03 1145 ~~1000~~		3/11/03 1145
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Date / Time
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Date / Time

Remarks and Observations

Matrix Types S Soil or Sediment WW Wastewater T Animal Tissue SW Surface Water SL Sludge or Solid Waste MW Marine Water GW Ground Water DW Drinking Water P Plant Tissue F Filter A Air Ot Other (See Remarks)

MACTEC Engineering and Consulting, Inc. (formerly Harding ESE)
Toxicology Laboratory
Gainesville, Florida

QA Form Number: 063
Revised: Jan2003

SAMPLE CHAIN OF CUSTODY

Facility: MacTec E engineering and Consulting Inc. Tox lab

Location: 404 SW 140 terrace Newberry, Fl 32669

Permit Number: Outfall Number:

SAMPLE IDENTIFICATION

Sample Identification	Sample Type Grab	Sample Type Comp	Date	Time	Observations
PH03 - Ctrl	✓	~~✓~~	3/10/03	12:00	
Total Number of Samples:					

SAMPLE INFORMATION

Sampler(s): Tox lab

Sampling Point (Description): Marine land Fl

Water Quality Measurements (optional):

Temp: pH: Diss. Oxygen: Salinity/Cond.: Chlorine (TRC):

Water Quality Measured by: Date: Time:

Observations/Remarks:

CHAIN OF CUSTODY

Relinquished By:	Received By:	Date	Time
R. Dillman	[illegible]	3-10-03	1145

Appendix E

Chemical Laboratory Reports

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Final Data Report

Ponce Harbor Sediment and Water/Elutriate Data

Prepared for:

ppb Environmental Laboratories, Inc.

Prepared by:

Battelle
397 Washington Street
Duxbury, Massachusetts 02332

May 22, 2003

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Custody Information

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Ponce, Puerto Rico Station ID's

Harbor				
RS-PH03		Ponce Harbor Reference Composite of Reference Station A & Reference Station B		
Samples				
E-PH03-SC		Ponce Harbor Composite Sample (Silty Clay composite of Harbor Stations 1-9)		
E-PH03-1		Discrete Ponce Harbor sample from Station 1		
E-PH03-2		Discrete Ponce Harbor sample from Station 2		
E-PH03-3		Discrete Ponce Harbor sample from Station 3		
E-PH03-4		Discrete Ponce Harbor sample from Station 4		
E-PH03-5		Discrete Ponce Harbor sample from Station 5		
E-PH03-6		Discrete Ponce Harbor sample from Station 6		
E-PH03-7		Discrete Ponce Harbor sample from Station 7		
E-PH03-8		Discrete Ponce Harbor sample from Station 8		
E-PH03-9		Discrete Ponce Harbor sample from Station 9		

ODMDS				
RS-PH03-1A		Ponce ODMDS Reference "Native" Sample from Station A		
RS-PH03-1B		Ponce ODMDS Reference "Duplicate" Sample from Station A		
RS-PH03-1C		Ponce ODMDS Reference "Triplicate" Sample from Station A		
RS-PH03-2A		Ponce ODMDS Reference "Native" Sample from Station B		
RS-PH03-2B		Ponce ODMDS Reference "Duplicate" Sample from Station B		
RS-PH03-2C		Ponce ODMDS Reference "Triplicate" Sample from Station B		
E-PODS03-1A		Ponce ODMDS Sample from Station 1 "Native"		
E-PODS03-1B		Ponce ODMDS Sample from Station 1 "Duplicate"		
E-PODS03-1C		Ponce ODMDS Sample from Station 1 "Triplicate"		
E-PODS03-2		Ponce ODMDS Sample from Station 2		
E-PODS03-3A		Ponce ODMDS Sample from Station 3 "Native"		
E-PODS03-3B		Ponce ODMDS Sample from Station 3 "Duplicate"		
E-PODS03-3C		Ponce ODMDS Sample from Station 3 "Triplicate"		
E-PODS03-4A		Ponce ODMDS Sample from Station 4 "Native"		
E-PODS03-4B		Ponce ODMDS Sample from Station 4 "Duplicate"		
E-PODS03-4C		Ponce ODMDS Sample from Station 4 "Triplicate"		
E-PODS03-5		Ponce ODMDS Sample from Station 5		
E-PODS03-6		Ponce ODMDS Sample from Station 6		
E-PODS03-7A		Ponce ODMDS Sample from Station 7 "Native"		
E-PODS03-7B		Ponce ODMDS Sample from Station 7 "Duplicate"		
E-PODS03-7C		Ponce ODMDS Sample from Station 7 "Triplicate"		
E-PODS03-8A		Ponce ODMDS Sample from Station 8 "Native"		
E-PODS03-8B		Ponce ODMDS Sample from Station 8 "Duplicate"		
E-PODS03-8C		Ponce ODMDS Sample from Station 8 "Triplicate"		
E-PODS03-9		Ponce ODMDS Sample from Station 9		
E-PODS03-10		Ponce ODMDS Sample from Station 10		
E-PODS03-11		Ponce ODMDS Sample from Station 11		
E-PODS03-12		Ponce ODMDS Sample from Station 12		
E-PODS03-13		Ponce ODMDS Sample from Station 13		
E-PODS03-14		Ponce ODMDS Sample from Station 14		
E-PODS03-15		Ponce ODMDS Sample from Station 15		
E-PODS03-16		Ponce ODMDS Sample from Station 16		
E-PODS03-17		Ponce ODMDS Sample from Station 17		
E-PODS03-18		Ponce ODMDS Sample from Station 18		

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ShpNo SHP-030308-02

Battelle Project No:

Sample Receipt Form

Approved: ☑

Project Number:

Client: Ponce ODMDS

Received by: Fahey, Jessica

Date/Time Received: Saturday, March 08, 2003 11:00 AM

No. of Shipping Containers: 5

SHIPMENT

Method of Delivery: Commercial Carrier

Tracking Number: 8347 4798 5825

COC Forms: ☑ Shipped with samples ☐ No Forms

Cooler(s)/Box(es)

Container	Type	Sealed With	Seal Condition	Container Condition	Temp C	Total Samples
1 of 5	Cooler	Tape	Intact	Intact	1.8	1
2 of 5	Cooler	Tape	Intact	Intact	0.4	0
3 of 5	Cooler	Tape	Intact	Intact	1.4	17
4 of 5	Cooler	Tape	Intact	Intact	1.4	17
5 of 5	Cooler	Tape	Intact	Intact	3.3	0

Samples

Sample Labels:
☑ Sample labels agree with COC forms
☐ Discrepancies (see Sample Custody Corrective Action Form)

Container Seals:
☐ Tape ☐ Custody Seals ☐ Other Seals (See sample Log)
☑ Seals intact for each shipping container
☐ Seals broken (See sample log for impacted samples)

Condition of Samples:
☐ Sample containers intact
☑ Sample containers broken/leaking (See Custody Corrective Action Form)

Temperature upon receipt (°C): 1.8 Temperature Blank used ☐ Yes ☑ No
(Note: If temperature upon receipt differs from required conditions, see sample log comment field)

Samples Acidified: ☐ Yes ☐ No ☑ Unknown

Initial pH 5-9?: ☐ Yes ☐ No ☑ NA
If no, individual sample adjustments on the Auxiliary Sample Receipt Form

Total Residual Chlorine Present?: ☐ Yes ☐ No ☑ NA
If yes, individual sample adjustments on the Auxiliary Sample Receipt Form

Head Space <1% in samples for water VOC analysis: ☐ Yes ☐ No ☑ NA
Individual sample deviations noted on sample log

Samples Containers:
Samples returned in PC-grade jars: ☐ Yes ☑ No ☐ Unknown /Lot No.: UnKnown

002

Approved: ☑

Storage Location:	Upper Cold Room Refrigerator	**BDO IDs Assigned:**	U8093 - U8127
Samples logged in by:	Fahey, Jessica	**Date/Time:**	03/08/2003 12:16 PM
Approved By:	Smith, Joe	**Approved On:**	3/10/2003 2:13:
Authorized By:		**Authorized On:**	

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ShpNo: SHP-030308-02

Battelle Project No:

Report Corrective Actions

1 1

Approved:

COC Client: Ponce ODMDS
COC Project: PPB/AFI Ponce Harbor
COC Date: 3/8/2003 12:16:0

	Description of Problem:	Explanation:
Sample Container Integrity	Sample broken	1 of the 8 1-gallon water samples was broken upon receipt. The sample was lost

Documentation of project manager notification

Sample Custodian Fahey, Jessica **Date:** 3/8/2003 1:11:00 PM
Laboratory Manager: [signature] **Date:** 3·10·03
Project Manager: [signature] **Date:** 3/11/2003

Documentation of client notification (should be completed by project manager within 24 hrs):

On 3/9 **I contacted** Bryan Cotter **at** PPB

Results of communication with client (Describe any corrective action directed by the client):

Proceed - sample lost. All waters are the same sample. We should be able to make do - we sent extra bottles.

Date this form was received back to the custodian: 3/10/03
Reference Number:

SAMPLE CUSTODY RECORD

(SOP# MSL-A-001 & MSL-A-002)

Date: 3/7/03

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Pacific Northwest Division
Marine Sciences Laboratory
1529 West Sequim Bay Road
Sequim, Washington 98382

Project Name: PPB/AFI - Ponce
Project Manager: Bryan Cotter
Phone Number: 352/377-2349

Shipment Method: FedEx
Preservation: Ice + for 1L Teflon HNO3

Line	Field Sample ID	Collection Date/Time	Matrix	No. of Containers	Test Parameters: As Per Contract					Laboratory ID	Observations/Comments
1	E-PH03-1W	3/7/03 0900	Seawater	9	X					U 8093	8 1-Gal Glass Amber 2
2											1 1-L for Metals 1
3											
4											
5											
6											
7											
8											
9											
10											
11											
12											
13											
14											
15											

Relinquished By:	Company:	Received By:	Company:
Charles R. Fellows	PPB /WAR	FedEx	
Signature/Printed Name	3/7/03 ~1530 Date/Time	Signature/Printed Name	3/7/03 ~1530 Date/Time

Relinquished By:	Company:	Received By:	Company:
FEDEX		Jessica M Fahy	Battelle
Signature/Printed Name	Date/Time	Signature/Printed Name	3/8/03 11:00 Date/Time

005

Rev: 000102COC

Distribution: White - Laboratory Project Files Yellow - Laboratory Client Pink - Sampler

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Chain of Custody

Proj. No

Proj. Name PPB-AFI Ponce ODMDS

SAMPLERS: Signature BFC-PPB + CSA

ANALYSIS REQUESTED → "NUMBER OF CONTAINERS"

DATE	TIME	BATTELLE ID	CLIENT ID	SAMPLE DESCRIPTION	As per contract ~~TEST~~	PCB	TPH FINGERPRINT	PAH	VOA	TBT	METALS	OTHER	ACIDIFIED	PRESERVED	Total Number of Containers
3/04/03	1414	U8094	RS-PH03-1A	~~RS-PH03-1A~~ 3/5/03 ODMDS reference	✓										2
	1437	U8095	RS-PH03-1B		✓										2
	1508	U8096	RS-PH03-1C		✓										2
3/05/03	0916	U8097	RS-PH03-2A	ODMDS reference	✓										2
	0936	U8098	RS-PH03-2B		✓										2
	0950	U8099	RS-PH03-2C		✓										2
3/05/03	1159	U81000	E-PODS03-1A	ODMDS sample	✓										2
	1159	U8101	-1B		✓										2
	1159	U8102	-1C		✓										2
3/05/03	1539	U8103	E-PODS03-2	"	✓										2
3/05/03	1314	U8104	E-PODS03-3A	"	✓										2
	1314	U8105	-3B	"	✓										2
	1314	U8106	-3C	"	✓										2

Relinquished by:	Date/Time		Received by:	Date/Time	
Kits to Ponce	02/27/03	1500	Recd in Ponce	03/2/03	1200
Ponce → Battelle, Dux. B. Cotter	03/07/03	1530	Battelle	3/8/03	10:00AM

Comments: All kits recd by BFC w/ custody seals intact. (to Ponce)
Spaces between samples are intentional.

ORIGINAL

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Chain of Custody

Proj. No

Proj. Name PPB-AFI Ponce ODMDS

SAMPLERS: Signature BFC-PPB ∀ CSA

ANALYSIS REQUESTED → "NUMBER OF CONTAINERS"

DATE	TIME	BATTELLE ID	CLIENT ID	SAMPLE DESCRIPTION	As per contract	PCB	TPH FINGERPRINT	PAH	VOA	TBT	METALS	OTHER	ACIDIFIED	PRESERVED	Total Number of Containers
03/05/03	1408	U8107	E-PODS03-4A (BC 3/6/03)	ODMDS sample	✓										2
↓	1408	U8108	-4B		✓										2
	1408	U8109	-4C		✓										2
03/05/03	1502	U8110	E-PODS03-5	"	✓										
BFC 3/6/03															
03/06/03	0823	U8111	E-PODS03-6	"	✓										2
03/06/03	0930	U8112	E-PODS03-7A	"	✓										2
	~~0930~~ BC														
03/06/03	1034 3/6/03	U8113	E-PODS03-8A	"	✓										2
	1034	U8114	-8B	"	✓										2
↓	1034	U8115	-8C	"	✓										2
03/06/03	0930	U8116	E-PODS03-7B	"	✓										
↓	0930	U8117	-7C	"	✓										
BC 3/6/03															

Relinquished by:	Date/Time		Received by:	Date/Time	
Kits → Ponce BFC-PPB	02/27/03	1500	Recd in Ponce	03/02/03	1200
Relinquished by: Ponce → Battelle Dux B. Cotter	03/07/03	1530	Received by: Recd in Duxbury	3/8/03	11:00 AM

Comments: All kits rec'd by BFC w/ the custody seals intact (to Ponce)
ODMDS stations 7A, 7B, 7C should have been recorded on the COC sequentially. BFC
Spaces between samples are intentional.
Kits

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Chain of Custody

Proj. No	Proj. Name

SAMPLERS: Signature

ANALYSIS REQUESTED → "NUMBER OF CONTAINERS"

DATE	TIME	BATTELLE ID	CLIENT ID	SAMPLE DESCRIPTION	~~PEST~~ As per contract	PCB	TPH FINGERPRINT	PAH	VOA	TBT	METALS	OTHER	ACIDIFIED	PRESERVED	Total Number of Containers
3/06/03	1140	U8118	E-PODS03-9	ODMDS Sample	✓										2
03/06/03	1251	U8119	E-PODS03-10	"	✓										2
3/06/03	1758	U8120	E-PODS03-11	"	✓										
03/06/03	1721	U8121	~~E-PH03~~ POPS03 -12	"	✓										
03/06/03	1411	U8122	E-PODS03-13	"	✓										
3/06/03	1025	U8123	E-PODS03-14	"	✓										2
03/07/03	0951	U8124	E-PODS03-15	"	✓										2
03/07/03	0912	U8125	E-PODS03-16	"	✓										2
	1159	U8126	E-PODS03-17	"	✓										2
03/07/03	0831	U8127	E-PODS03-18	"	✓										2

Relinquished by:	Date/Time		Received by:	Date/Time	
Kits to Ponce [signature]	02/27/03	1500	Rec'd in Ponce [signature]	03/2/03	1200
Relinquished by: Ponce → Duxbury [signature]	03/07/03	1530	Received by: Battelle [signature]	3/8/03	11:00am

Comments: All Kits rec'd by BFC w/Custody seals intact. (to Ponce) Spaces between samples are intentional.

008

ORIGINAL

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ShpNo SHP-030308-02

Battelle Project No:

Sample Receipt Form Details

Approved: Authorized

Project Number:

Client: Ponce ODMDS

Received by: Fahey, Jessica

Date/Time Received: Saturday, March 08, 2003 11:00 AM

No. of Shipping Containers: 5

BDO Id:	Client Sample ID:	Collection Date:	Login Date:	Ctrs:	Matrix:	Temp:	pH:	TRC:	VOC:	Stored In:	Loc:	No:	Comments:
U8093	E-PH03-1W	03/07/03 9:00	03/08/03 12:30	9	Water	1.8	NA	NA	NA	Cold Room Refrig			Received 1 1-L samples
U8094	RS-PH03-1A	03/04/03 14:14	03/08/03 12:34	2	Sediment	1.4	NA	NA	NA	Cold Room Refrig			large container in cooler
U8095	RS-PH03-1B	03/04/03 14:37	03/08/03 12:35	2	Sediment	1.4	NA	NA	NA	Cold Room Refrig			large container in cooler
U8096	RS-PH03-1C	03/04/03 15:08	03/08/03 12:36	2	Sediment	1.4	NA	NA	NA	Cold Room Refrig			large container in cooler
U8097	RS-PH03-2A	03/05/03 9:16	03/08/03 12:43	2	Sediment	1.4	NA	NA	NA	Cold Room Refrig			
U8098	RS-PH03-2B	03/05/03 9:36	03/08/03 12:43	2	Sediment	1.4	NA	NA	NA	Cold Room Refrig			
U8099	RS-PH03-2C	03/05/03 9:50	03/08/03 12:43	2	Sediment	1.4	NA	NA	NA	Cold Room Refrig			
U8100	E-PODS03-1A	03/05/03 11:59	03/08/03 12:44	2	Sediment	1.4	NA	NA	NA	Cold Room Refrig			
U8101	E-PODS03-1B	03/05/03 11:59	03/08/03 12:44	2	Sediment	1.4	NA	NA	NA	Cold Room Refrig			
U8102	E-PODS03-1C	03/05/03 11:59	03/08/03 12:45	2	Sediment	1.4	NA	NA	NA	Cold Room Refrig			
U8103	E-PODS03-2	03/05/03 15:39	03/08/03 12:47	2	Sediment	1.4	NA	NA	NA	Cold Room Refrig			LARGE CONTAINER IN
U8104	E-PODS03-3A	03/05/03 13:14	03/08/03 12:51	2	Sediment	1.4	NA	NA	NA	Cold Room Refrig			
U8105	E-PODS03-3B	03/05/03 13:14	03/08/03 12:51	2	Sediment	1.4	NA	NA	NA	Cold Room Refrig			
U8106	E-PODS03-3C	03/05/03 13:14	03/08/03 12:52	2	Sediment	1.4	NA	NA	NA	Cold Room Refrig			
U8107	E-PODS03-4A	03/05/03 14:08	03/08/03 12:53	2	Sediment	1.4	NA	NA	NA	Cold Room Refrig			
U8108	E-PODS03-4B	03/05/03 14:08	03/08/03 12:53	2	Sediment	1.4	NA	NA	NA	Cold Room Refrig			
U8109	E-PODS03-4C	03/05/03 14:08	03/08/03 12:54	2	Sediment	1.4	NA	NA	NA	Cold Room Refrig			
U8110	E-PODS03-5	03/05/03 15:02	03/08/03 12:55	2	Sediment	1.4	NA	NA	NA	Cold Room Refrig			LARGE CONTAINER IN
U8111	E-PODS03-6	03/06/03 8:23	03/08/03 12:55	2	Sediment	-1.4	NA	NA	NA	Cold Room Refrig			
U8112	E-PODS03-7A	03/06/03 9:30	03/08/03 12:56	2	Sediment	-1.4	NA	NA	NA	Cold Room Refrig			
U8113	E-PODS03-8A	03/06/03 10:34	03/08/03 12:57	2	Sediment	-1.4	NA	NA	NA	Cold Room Refrig			
U8114	E-PODS03-8B	03/06/03 10:34	03/08/03 12:57	2	Sediment	-1.4	NA	NA	NA	Cold Room Refrig			
U8115	E-PODS03-8C	03/06/03 10:34	03/08/03 12:58	2	Sediment	-1.4	NA	NA	NA	Cold Room Refrig			LARGE CONTAINER I
U8116	E-PODS03-7B	03/06/03 9:30	03/08/03 12:59	2	Sediment	-1.4	NA	NA	NA	Cold Room Refrig			LARGE CONTAINER I
U8117	E-PODS03-7C	03/06/03 9:30	03/08/03 12:59	2	Sediment	-1.4	NA	NA	NA	Cold Room Refrig			LARGE CONTAINER I
U8118	E-PODS03-9	03/06/03 11:40	03/08/03 13:03	2	SEDIMENT	-1.4	NA	NA	NA	Cold Room Refrig			
U8119	E-PODS03-10	03/06/03 12:51	03/08/03 13:04	2	SEDIMENT	-1.4	NA	NA	NA	Cold Room Refrig			
U8120	E-PODS03-11	03/06/03 17:58	03/08/03 13:04	2	SEDIMENT	-1.4	NA	NA	NA	Cold Room Refrig			LARGE CONTAINER I

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ShpNo SHP-030308-02

Battelle Project No:

Sample Receipt Form Details

Approved: ■ Authorized ■

Project Number:

Client: Ponce ODMDS

Received by: Fahey, Jessica

Date/Time Received: Saturday, March 08, 2003 11:00 AM

No. of Shipping Containers: 5

BDO Id:	Client Sample ID:	Collection Date:	Login Date:	Ctrs:	Matrix:	Temp:	pH:	TRC:	VOC:	Stored In:	Loc:	No:	Comments:
U8121	E-PODS03-12	03/06/03 17:21	03/08/03 13:05	2	SEDIMENT	-1.4	NA	NA	NA	Cold Room Refrig			LARGE CONTAINER I
U8122	E-PODS03-13	03/06/03 14:11	03/08/03 13:05	2	SEDIMENT	-1.4	NA	NA	NA	Cold Room Refrig			LARGE CONTAINER I
U8123	E-PODS03-14	03/06/03 10:25	03/08/03 13:06	2	SEDIMENT	-1.4	NA	NA	NA	Cold Room Refrig			
U8124	E-PODS03-15	03/07/03 9:51	03/08/03 13:06	2	SEDIMENT	-1.4	NA	NA	NA	Cold Room Refrig			
U8125	E-PODS03-16	03/07/03 9:12	03/08/03 13:08	2	SEDIMENT	-1.4	NA	NA	NA	Cold Room Refrig			
U8126	E-PODS03-17	03/07/03 11:59	03/08/03 13:08	2	SEDIMENT	-1.4	NA	NA	NA	Cold Room Refrig			
U8127	E-PODS03-18	03/07/03 8:31	03/08/03 13:09	2	SEDIMENT	-1.4	NA	NA	NA	Cold Room Refrig			

Total Samples: 35

010

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ShpNo SHP-030311-07

Battelle Project No: N005561

Sample Receipt Form

Approved: ☑

Project Number:

Client: Ponce Harbor

Received by: Smith, Joe

Date/Time Received: Tuesday, March 11, 2003 12:00 AM

No. of Shipping Containers: 2

SHIPMENT

Method of Delivery: Commercial Carrier

Tracking Number: 790227010673

COC Forms: ☑ **Shipped with samples** ☐ **No Forms**

Cooler(s)/Box(es)

Container	Type	Sealed With	Seal Condition	Container Condition	Temp C	Total Samples
1 of 2	Cooler	Tape	Intact	Intact	0.9	2
2 of 2	Cooler	Tape	Intact	Intact	0.9	10

Samples

Sample Labels:
☑ Sample labels agree with COC forms
☐ Discrepancies (see Sample Custody Corrective Action Form)

Container Seals:
☐ Tape ☐ Custody Seals ☐ Other Seals (See sample Log)
☑ Seals intact for each shipping container
☐ Seals broken (See sample log for impacted samples)

Condition of Samples:
☑ Sample containers intact
☐ Sample containers broken/leaking (See Custody Corrective Action Form)

Temperature upon receipt (°C): -0.9 Temperature Blank used ☐ Yes ☑ No
(Note: If temperature upon receipt differs from required conditions, see sample log comment field)

Samples Acidified: ☐ Yes ☐ No ☑ Unknown

Initial pH 5-9?: ☐ Yes ☐ No ☑ NA
If no, individual sample adjustments on the Auxiliary Sample Receipt Form

Total Residual Chlorine Present?: ☐ Yes ☐ No ☑ NA
If yes, individual sample adjustments on the Auxiliary Sample Receipt Form

Head Space <1% in samples for water VOC analysis: ☐ Yes ☐ No ☑ NA
Individual sample deviations noted on sample log

Samples Containers:
Samples returned in PC-grade jars: ☐ Yes ☑ No ☐ Unknown /Lot No.: Unknown

Storage Location: Upper Cold Room Refrigerator

BDO IDs Assigned: U8240 - U8251

Samples logged in by: Smith, Joe

Date/Time: 03/11/2003 11:09 AM

Approved By: Fahey, Jessica

Approved On: 3/13/2003 9:01:

Authorized By:

Authorized On:

011

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ShpNo: SHP-030311-07

Battelle Project No:

Report Corrective Actions

1 1

Approved:

COC Client: Ponce Harbor

COC Project: PPB/ AFI Ponce

COC Date: 3/11/2003 11:09:00 AM

	Description of Problem:	Explanation:
Client Id	Other	E-PH03-SC ONE OF THE 8 JARS IS MISSING A LABLE, BUT HAS ID ON LID. LABLE WAS CREATED BY CUSTODIAN.

Documentation of project manager notification

Sample Custodian Smith, Joe **Date:** 3/11/2003 11:59:00 A

Laboratory Manager: [illegible] **Date:** 3·11·03

Project Manager: [illegible] **Date:** 3/11/2003

Documentation of client notification (should be completed by project manager within 24 hrs):

On _____ **I contacted** _____ **at**

Results of communication with client (Describe any corrective action directed by the client):

corrected by custodian

Date this form was received back to the custodian: _____

Reference Number: _____

012

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Chain of Custody

Proj. No	Proj. Name
	PPB/AFI-Ponce

SAMPLERS: Signature

PPB composited F.J.C BFC + techs

ANALYSIS REQUESTED → "NUMBER OF CONTAINERS"

As per contract

DATE	TIME	BATTELLE ID	CLIENT ID	SAMPLE DESCRIPTION	~~PEST~~ 3/10/03	PCB	TPH FINGERPRINT	PAH	VOA	TBT	METALS	OTHER	ACIDIFIED	PRESERVED	Total Number of Containers
		U8240	RS-PH03	Reference sample composite (Harbor ref)	✓										2
		U8241	E-PH03-SC	E-PH03- silty clay composite (1-9)	✓										8
		U8242	E-PH03-control	control sediment	✓										6
		U8243	E-PH03-1SC	individual core samples	✓										1
		U8244	-2SC		✓										1
		U8245	-3SC		✓										1
		U8246	-4SC		✓										1
		U8247	-5SC		✓										1
		U8248	-6SC		✓										1
		U8249	-7SC		✓										1
		U8250	-8SC		✓										1
		U8251	-9SC		✓										1

Relinquished by:	Date/Time		Received by:	Date/Time	
F.J.C BFC-PPB	03/10/03	1800	Fed Ex	03/10/03	1800
Relinquished by: Fed Ex to Duxbury	Date/Time		Received by: Battelle	Date/Time	
				March 11, 2003	

Comments: cooler Custody seal applied by BFC on 3/10/03 1700

013

ORIGINAL

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ShpNo SHP-030311-07

Battelle Project No:

Sample Receipt Form Details

Approved: ■ Authorized ■

Project Number:

Client: Ponce Harbor

Received by: Smith, Joe

Date/Time Received: Tuesday, March 11, 2003 12:00 AM

No. of Shipping Containers: 2

BDO Id:	Client Sample ID:	Collection Date:	Login Date:	Ctrs:	Matrix:	Temp:	pH:	TRC:	VOC:	Stored In:	Loc:	No:	Comments:
U8240	RS-PH03		03/11/03 11:24	2	SEDIMENT	-0.9	NA	NA	NA	Cold Room Refrig			
U8241	E-PH03-SC		03/11/03 11:25	8	SEDIMENT	-0.9	NA	NA	NA	Cold Room Refrig			SOME OF JARS IN CO
U8242	E-PHO3-CONTROL		03/11/03 11:28	6	SEDIMENT	-0.9	NA	NA	NA	Cold Room Refrig			SOME OF JARS IN CO
U8243	E-PHO3-1SC		03/11/03 11:29	1	SEDIMENT	-0.9	NA	NA	NA	Cold Room Refrig			
U8244	E-PHO3-2SC		03/11/03 11:29	1	SEDIMENT	-0.9	NA	NA	NA	Cold Room Refrig			
U8245	E-PHO3-3SC		03/11/03 11:29	1	SEDIMENT	-0.9	NA	NA	NA	Cold Room Refrig			
U8246	E-PHO3-4SC		03/11/03 11:29	1	SEDIMENT	-0.9	NA	NA	NA	Cold Room Refrig			
U8247	E-PHO3-5SC		03/11/03 11:29	1	SEDIMENT	-0.9	NA	NA	NA	Cold Room Refrig			
U8248	E-PHO3-6SC		03/11/03 11:30	1	SEDIMENT	-0.9	NA	NA	NA	Cold Room Refrig			
U8249	E-PHO3-7SC		03/11/03 11:30	1	SEDIMENT	-0.9	NA	NA	NA	Cold Room Refrig			
U8250	E-PHO3-8SC		03/11/03 11:30	1	SEDIMENT	-0.9	NA	NA	NA	Cold Room Refrig			
U8251	E-PHO3-9SC		03/11/03 11:30	1	SEDIMENT	-0.9	NA	NA	NA	Cold Room Refrig			

Total Samples: 12

C14

Water/Elutriate Metals Data

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QA/QC NARRATIVE

PROJECT: Chemical Evaluation of Dredged Material from Ponce Harbor
PARAMETER: Metals
LABORATORY: Battelle Marine Sciences Laboratory, Sequim, Washington
MATRIX: Site Water and Elutriates

SAMPLE CUSTODY AND PROCESSING: Battelle received one site water and one elutriate seawater for metals analyses on 03/14/2003 and 03/13/2003, respectively. All samples were received in good condition (i.e., no sample containers were broken). Samples were assigned a Battelle Central File (CF) identification number (1982) and were entered into Battelle's sample tracking system.

The following lists information on sample receipt and processing activities:

Lab Sample IDs:	1982* 38 and 39
Description:	Site water and Elutriate
Sample collection dates	03/07/2003, 03/12/2003
Laboratory arrival date	03/13/2003, 03/14/2003
Cooler temp. on arrival	2.5°C and 2.7°C
Preconcentration	03/14/2003, 03/24/2003 (Zn)
CVAF analysis (Hg, MSL-I-013)	03/18/2003
FIAS analysis (Se, MSL-I-030)	03/17/2003
GFAA analysis (Ag, MSL-I-029)	03/20/2003
ICP-MS analysis (MSL-I-022) (As, Cd, Cr, Cu, Ni, Pb and Zn)	03/18/2003, 03/25/2003 (Zn)

DATA QUALITY OBJECTIVES:

Analyte	Analytical Method	Range of Recovery	SRM Accuracy [1]	Relative Precision [1]	Achieved Detection Limits (µg/L) [2]
Ag	GFAA	75-125%	≤ 20%	≤ 20%	0.0128
As	ICP-MS	75-125%	≤ 20%	≤ 20%	0.05
Cd	ICP-MS	75-125%	≤ 20%	≤ 20%	0.01
Cr	ICP-MS	75-125%	≤ 20%	≤ 20%	0.04
Cu	ICP-MS	75-125%	≤ 20%	≤ 20%	0.02
Hg	CVAA	75-125%	≤ 20%	≤ 20%	0.0002
Ni	ICP-MS	75-125%	≤ 20%	≤ 20%	0.04
Pb	ICP-MS	75-125%	≤ 20%	≤ 20%	0.01
Se	FIAS	75-125%	≤ 20%	≤ 20%	0.5
Zn	ICP-MS	75-125%	≤ 20%	≤ 20%	0.1

[1] Evaluated for analytes > 10 times the MDL.
[2] Water MDLs (as listed in QAPP) were determined from analysis of 7-replicates of spiked water, except Ag is from the batch specific MDL.

QA/QC NARRATIVE

METHODS:

Seawater samples were analyzed for ten metals: silver (Ag), arsenic (As), cadmium (Cd), chromium (Cr), copper (Cu), mercury (Hg), nickel (Ni), lead (Pb), selenium (Se), and zinc (Zn).

Seawater samples were preconcentrated using iron (Fe) and palladium (Pd) in accordance with the Battelle SOP MSL-I-025, *Methods of Sample Preconcentration*, derived from EPA Method 1640. The sample preconcentration was submitted for analysis by ICP-MS and GFAA. The original preconcentration batch (ICP-MS Batch ID: 031803-5000a) yielded low recoveries for the Zn matrix spike. The low recoveries indicated matrix interferences in the chelating process for Zn. The pH of the sample was adjusted and a second preconcentration batch was prepared and analyzed. The ICP-MS batch ID is 032503-5000b. The Zn values for seawater samples are reported by method of additions and freshwater samples (DI blanks and SRM 1640) are reported using the standard external calibration standards.

Samples were analyzed by inductively coupled plasma-mass spectrometry (ICP-MS) for As, Cd, Cr, Cu, Ni, Pb and Zn following Battelle SOP MSL-I-022, *Determination of Elements in Aqueous and Digestate Samples by ICP-MS.* The SOP procedures for this method are based on two EPA Methods: 200.8 and 1638.

Samples were analyzed for Se using hydride generation flow injection atomic spectroscopy (FIAS) according to Battelle SOP MSL-I-030 *Determination of Metals in Aqueous and Digestate Samples by HGAA-FIAS.*

Ag was analyzed in the original Fe-Pd preconcentrate by graphite furnace atomic absorption (GFAA) following Battelle SOP MSL-I-029, *Determination of Metals in Aqueous and Digestate Samples by GFAA*, based on EPA Method 200.9.

Hg was analyzed directly (with no preconcentration step) using cold-vapor atomic fluorescence (CVAF) spectroscopy according to Battelle SOP MSL-I-013, *Total Mercury in Aqueous Samples by CVAF*, which is derived from EPA Method 1631.

All results were reported in units of µg/L.

HOLDING TIMES:

The recommended holding times for metals analyses in water are 28 days from sample collection for Hg analysis and 6 months for analysis of all other metals. The holding times for all metals were achieved.

DETECTION LIMITS:

Analytical results were reported to laboratory achieved detection limits listed in the QAPP from the 2002 MDL study, with the exception of Ag which is reported from the batch specific MDL. Target detection limits are listed in the USACE – NYD EPA Region II Regional Testing Manual. Laboratory achieved detection limits were below the targets for all analytes. The data were evaluated and flagged to the following criteria:

- B Analyte detected in the blank is greater than 3 times the MDL
- U Undetected at or above MDL; MDL reported
- & QC value outside the accuracy or precision DQOs

METHOD BLANKS:

One method blank was analyzed with the set of water samples. The blank did not contain analytes above the criteria of 3 times the MDL, with the exception of Cu. The data are not blank corrected.

QA/QC NARRATIVE

MATRIX SPIKE ACCURACY:

The site water was selected for a matrix spike sample and fortified with approximately 5 µg/L Ag, As, Cd, Cr, Cu, Ni, Pb, Se and Zn; and 0.02 µg/L Hg. The matrix spike recoveries were within the QC criteria of 75-130% with a range from 77% to 104% recovery.

STANDARD REFERENCE MATERIAL ACCURACY:

The SRMs 1641 for Hg, 1640 and CASS-4 were analyzed with the set of samples for certified analytes. SRM accuracy is expressed as the percent difference (PD) between the certified and measured concentrations of each metal of interest.

The percent difference for SRM 1641 for Hg and 1640 for all other analytes ranged from 1% to 18% PD and were within the QC criteria of ±20% PD.

All analytes of interest in SRM CASS-4 are certified <10x the achieved detection limits with the exception of Cu. Therefore, this SRM is not an appropriate QC indicator of data set accuracy. Currently, no seawater SRM is certified at levels appropriate for all analytes. The above listed freshwater SRMs validate the data set accuracy. The PD for CASS-4 is reported in the table, as well as the reagent corrected values for CASS-4. The low level certification of CASS-4 requires a reagent correction, as reagents with zero metal contribution, necessary for the preconcentration of the samples, are not available. The PD values for the reagent corrected CASS-4 is within the QC criteria with a range of 1% to 15%. The recommendation is to reagent correct the sample data. The data are reported without any corrections.

Clean seawater was spiked with a certified standard to demonstrate data set accuracy in seawater. The percent recoveries ranged from 78% to 110% and were within the QC criteria.

REPLICATE PRECISION:

All samples were analyzed in triplicate to assess the laboratory precision. Precision is expressed as the relative standard deviation (RSD) of replicate results. The RSD values for laboratory triplicates ranged from 0% to 14% and were within the QC criteria of <20% for concentrations greater than 10 times the MDL.

BATTELLE MARINE SCIENCES LABORATORIES
1529 West Sequim Bay Road
Sequim, Washington 98382
(360) 681-4564

CHEMICAL EVALUATION OF DREDGE MATERIAL FROM PONCE HARBOR
Metals in Water
(Samples Received 03/13/03, 3/14/03)

(concentrations in µg/L, data are not blank corrected)

MSL Code	Duxbury ID	Client ID	Ag	As	Cd	Cr	Cu	Hg	Ni	Pb	Se	Zn
		Instrument:	GFAA	ICP-MS	ICP-MS	ICP-MS	ICP-MS	*CVAF*	ICP-MS	ICP-MS	FIAS	ICP-MS
		Analytical Batch ID:	*T032003A*	*031803-5000a*	*031803-5000a*	*031803-5000a*	*031803-5000a*	*031803HGA1*	*031803-5000a*	*031803-5000a*	*F031703A*	*032503-5000b*
1982-38 R1	W8253	Ponce Harbor Elut	0.0316	9.71	0.0111	0.568	1.24 B	0.000793	0.963	0.0795	0.955	1.55
1982-38 R2	W8253	Ponce Harbor Elut	0.0278	10.6	0.0116	0.568	1.29 B	0.000981	1.00	0.0917	1.26	1.50
1982-38 R3	W8253	Ponce Harbor Elut	0.0290	10.3	0.0114	0.583	1.28 B	0.000763	1.03	0.0846	1.19	1.85
	Mean		0.0295	10.2	0.0114	0.573	1.27	0.000846	0.998	0.0853	1.14	1.63
	RSD		**7%**	**4%**	**2%**	**2%**	**2%**	**14%**	**3%**	**7%**	**14%**	**12%**
1982-39 R1	U8093	E-PH03-1W	0.0275	0.893	0.01 U	0.308	2.71 B	0.000579	0.358	0.0518	0.5 U	4.35
1982-39 R2	U8093	E-PH03-1W	0.0293	0.892	0.01 U	0.298	2.70 B	0.000557	0.357	0.0497	0.5 U	4.44
1982-39 R3	U8093	E-PH03-1W	0.0299	0.939	0.01 U	0.293	2.68 B	0.000558	0.355	0.0519	0.5 U	4.12
	Mean		0.0289	0.908	NA	0.300	2.70	0.000565	0.357	0.0511	NA	4.30
	RSD		**4%**	**3%**		**3%**	**1%**	**2%**	**0%**	**2%**		**4%**
Laboratory Achieved Detection Limits [1]			0.0128	0.05	0.01	0.04	0.02	0.0002	0.04	0.01	0.5	0.1
		*B flag criteria (3 * MDL)*	*0.0384*	*0.15*	*0.03*	*0.1*	*0.06*	*0.0006*	*0.12*	*0.03*	*1.5*	*0.3*
METHOD BLANK												
Blank			0.0185	0.05 U	0.01 U	0.065	0.188 B	0.0002 U	0.0984	0.0147	0.5 U	0.250
MATRIX SPIKE RESULTS												
Spike Concentration			5.0	5.0	5.0	5.0	5.0	0.0159	5.0	5.0	5.0	5.0
1982-39 mean			0.0289	0.908	0.01 U	0.300	2.70	0.000565	0.357	0.0511	0.0307	4.30
1982-39 MS			4.37	4.78	4.47	4.81	6.68	0.0169	4.51	4.23	5.25	8.96
Percent Recovery			**87%**	**77%**	**89%**	**90%**	**80%**	**103%**	**83%**	**84%**	**104%**	**93%**
SEAWATER SPIKED WITH A CERTIFIED STANDARD												
Spike Concentration			5.0	5.0	5.0	5.0	5.0	0.00487	5.0	5.0	5.0	5.0
1982-SB Blank			0.0272	1.15	0.0800	0.212	0.535	NA	0.516	0.0222	0.5 U	0.534
1982-SB LCS			4.19	5.03	4.36	4.58	4.51	0.00538	4.46	4.07	5.01	6.02
Percent Recovery			**84%**	**78%**	**86%**	**87%**	**80%**	**110%**	**79%**	**81%**	**100%**	**110%**

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CHEMICAL EVALUATION OF DREDGE MATERIAL FROM PONCE HARBOR
Metals in Water
(Samples Received 03/13/03, 3/14/03)

(concentrations in µg/L, data are not blank corrected)

MSL Code	Duxbury ID	Client ID	Ag	As	Cd	Cr	Cu	Hg	Ni	Pb	Se	Zn
		Instrument:	GFAA	ICP-MS	ICP-MS	ICP-MS	ICP-MS	*CVAF*	ICP-MS	ICP-MS	FIAS	ICP-MS
		Analytical Batch ID:	*T032003A*	*031803-5000a*	*031803-5000a*	*031803-5000a*	*031803-5000a*	*031803HGA1*	*031803-5000a*	*031803-5000a*	*F031703A*	*032503-5000b*
STANDARD REFERENCE MATERIAL												
1640 Direct or 1641d for Hg			6.96	30.8	24.7	39.4	87.2	1570	28.2	27.9	21.5	62.8
1640 Direct			NA	30.1	24.5	37.1	83.0	NA	26.7	27.4	NA	62.8
		certified value	7.62	26.67	22.79	38.6	85.2	1590	27.4	27.89	21.96	53.2
		range	±0.25	±0.41	±0.96	±1.6	±1.2	±40	±0.8	±0.14	±0.51	±1.1
		percent difference	**9%**	**15%**	**8%**	**2%**	**2%**	**1%**	**3%**	**0%**	**2%**	**18%**
			NA	**13%**	**8%**	**4%**	**3%**	NA	**3%**	**2%**	NA	**18%**
CASS-4			0.0292	1.06	0.0299	0.216	0.740	NA	0.426	0.0192	NA	0.577
CASS-4, Reagent Corrected			0.01 U	1.06	0.0299	0.151	0.552		0.328	0.0045		0.327
		certified value		1.11	0.026	0.144	0.592		0.314	0.0098		0.381
		range		±0.16	±0.003	±0.029	±0.055		±0.03	±0.0036		±0.057
		PD, CASS-4	NA	**5%**	**15%**	**50% &**	**25% &**	NA	**36% &**	**NC**	NA	**51% &**
		PD, CASS-4 Reagent Corrected	NA	**5%**	**15%**	**5%**	**7%**	NA	**4%**	**NC**	NA	**14%**

[1] Water MDLs (as listed in QAPP) determined from analysis of 7-replicate, spiked water, except Ag is from the batch specific MDL
B Analyte detected in blank is greater than 3x MDL
U Undetected at or above MDL; MDL reported
& QC value outside the accuracy or precision DQO (SRM and RSD <20%, Spike Recoveries ±25%)
NA Not applicable/available
NC Not calculated, SRM certified <10 times the MDL

Water/Elutriate Chlorinated Hydrocarbon Data

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PCB/Pesticide – WATER and ELUTRIATE QA/QC SUMMARY
Batch 03-0240

PROJECT: Ponce Harbor
PARAMETER: PCB/Pesticide
LABORATORY: Battelle, Duxbury, MA
MATRIX: Water
SAMPLE CUSTODY: The water sample was collected on March 7, 2003 and received at Battelle Duxbury on March 8, 2003. The sample was received in good condition. One 4-L bottle broke in transit, however the field team collected more water than necessary for the required analyses, so this was of little consequence. No other samples were impacted. The cooler temperature was 1.8 °C on arrival. Samples were stored at 4 °C until processing.

QA/QC DATA QUALITY OBJECTIVES:

	Reference Method	Blank Criteria	Surrogate Recovery	LCS/MS Recovery	SRM % Diff.	Sample Replicate Relative Precision	Achieved Detection Limit (PDL) (ng/L)
Pest/ PCB	General NS&T	B<3xMDL	30-150% Recovery	50-120% Recovery (for at least 80% of analytes; analyte conc. in MS must be >10x background)	≤25% PD (on average; not to exceed 35% for more than 30% of analytes; measured from range of certified values, for analytes >10x MDL)	≤30% RSD	~0.5 - 3 (one exception, Cl2(08) 16 ng/L due to chromatographic interference; MDL study being repeated) **Target DL:** Pest: 2 to 100 PCB: 0.5

METHOD: Water samples were extracted for PCB and pesticides following general NS&T methods. Approximately 1 liter of water was spiked with surrogates and extracted three times with dichloromethane using separatory funnel techniques. The combined extract was dried over anhydrous sodium sulfate, concentrated, processed through alumina cleanup column, concentrated, and further purified by GPC/HPLC. The post-HPLC extract was concentrated, fortified with RIS and solvent exchanged into hexane. Extracts were analyzed using gas chromatography/electron capture detection (GC/ECD), following general NS&T methods. Sample data were quantified by the method of internal standards, using the Recovery Internal Standard (RIS) compounds. The HPLC cleanup provides a superior extract for anlaysis – little to no interferences. Because of this cleanup, an additional dilution factor of ~1.7 is applied to the final data, and therefore the MDLs are elevated. The chromatography was excellent, and because of the signal-to-noise relationship which determines which chromatographic peaks should be integrated (3:1), no additional PCB or pesticides would be reported if the detection limits were reduced by that factor.

Elutriates were prepared following procedures described in EPA's Evaluation of Dredged Material Proposed for Ocean Disposal ("The Green Book"). Two liters of test sediment was combined with 8 L of site water to prepare the elutriate. The final elutriate was centrifuged in Teflon centrifuge containers after settling, and a portion was acidified for metals analysis. The remainder was immediately extracted for PCB/pesticide analysis.

PCB/Pesticide – WATER and ELUTRIATE QA/QC SUMMARY
Batch 03-0240

HOLDING TIMES: Samples were prepared for analysis in one analytical batch and were extracted within 7 days of sample collection and analyzed within 40 days of extraction.

Batch	Extraction Date	Analysis Date
03-0240	03/13/2003	03/22/2003 – 03/24/2003

BLANKS: A procedural blank (PB) was prepared with each analytical batch. Blanks were analyzed to ensure the sample extraction and analysis methods were free of contamination.

03-0240 – No target analytes were detected in the blank

Comments – None.

LABORATORY CONTROL SAMPLE: A laboratory control sample (LCS) was prepared with each analytical batch. The percent recoveries of target PCB and pesticides were calculated to measure data quality in terms of accuracy.

03-0240 – All target analytes were recovered within the laboratory control limits.

Comments – None.

MATRIX SPIKE: A matrix spike (MS) sample was prepared with each analytical batch. The percent recoveries of target PCB/pesticides were calculated to measure data quality in terms of accuracy.

03-0240 – All target analytes were recovered within the laboratory control limits.

Comments – None.

SURROGATES: Two surrogate compounds were added prior to extraction, including Cl3(34) and Cl5(112). The recovery of each surrogate compound was calculated to measure data quality in terms of accuracy (extraction efficiency).

03-0240 – Percent recoveries for all surrogate compounds were within the laboratory control limits specified by the method (30 – 150% recovery).

Comments – None.

REPLICATES: Replicate (triplicate) samples for waters were prepared with each analytical batch. The RSD between triplicate analyses for PCB/pesticides is calculated to measure data quality in terms of precision.

03-0240 – The RSD between triplicate analyses of all target PCBs were within the DQO of <30% RSD.

Comments – None; no target analytes were detected in any of the samples.

PCB/Pesticide – WATER and ELUTRIATE QA/QC SUMMARY
Batch 03-0240

SRM: A standard reference material (SRM, a certified second source standard was spiked as an SRM) was prepared with each analytical batch. The percent difference (PD) between the measured value and the certified range was calculated to measure data quality in terms of accuracy.

03-0240 – SRM PDs were within the control limits for all target analytes.

Comments – None.

Procedural Blank Data

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Project Name: Ponce Harbor
Project Number: N005561

Client Sample ID:	NA
Battelle Sample ID:	BB716PB
Battelle Batch ID:	03-0240
Sample Volume (L):	1.00
Units:	ng/L

Aldrin	2.826 U
a Chlordane	1.078 U
trans Nonachlor	1.013 U
Dieldrin	0.978 U
4,4 DDT	0.557 U
2,4 DDT	1.985 U
4,4 DDD	0.595 U
2,4 DDD	0.747 U
4,4 DDE	0.838 U
2,4 DDE	1.706 U
Endosulfan I	1.110 U
Endosulfan II	0.510 U
Endosulfan sulfate	0.565 U
Heptachlor	1.173 U
Heptachlor epoxide	0.948 U
Cl2 08	16.000 U
Cl3 18	1.385 U
Cl3 28	1.730 U
Cl4 44	1.453 U
Cl4 49	1.487 U
Cl4 52	1.435 U
Cl4 66	1.490 U
Cl5 87	1.128 U
Cl5 101	1.145 U
Cl5 105	0.575 U
Cl5 118	0.868 U
Cl6 128	1.400 U
Cl6 138	1.325 U
Cl6 153	1.065 U
Cl7 170	1.015 U
Cl7 180	0.960 U
Cl7 183	0.930 U
Cl7 184	0.918 U
Cl7 187	0.862 U
Cl8 195	1.085 U
Cl9 206	1.217 U
Cl10 209	1.265 U

Surrogate Recoveries (%):

Cl3(34)	82
Cl5(112)	92

U - Analyte not detected, ssMDL value inserted.
J - Analyte detected below the ssMDL value.
N - QC value outside the accuracy or precision DQO.

Field Sample Data

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Project Name: Ponce Harbor
Project Number: N005561

Client Sample ID:	E-PH03-1W	Ponce Harbor Elut
Battelle Sample ID:	U8093	U8253
Battelle Batch ID:	03-0240	03-0240
Sample Volume (L):	1.00	1.00
Units:	ng/L	ng/L
Aldrin	2.826 U	2.826 U
a Chlordane	1.078 U	1.078 U
trans Nonachlor	1.013 U	1.013 U
Dieldrin	0.978 U	0.978 U
4,4 DDT	0.557 U	0.557 U
2,4 DDT	1.985 U	1.985 U
4,4 DDD	0.595 U	0.595 U
2,4 DDD	0.747 U	0.747 U
4,4 DDE	0.838 U	0.838 U
2,4 DDE	1.706 U	1.706 U
Endosulfan I	1.110 U	1.110 U
Endosulfan II	0.510 U	0.510 U
Endosulfan sulfate	0.565 U	0.565 U
Heptachlor	1.173 U	1.173 U
Heptachlor epoxide	0.948 U	0.948 U
Cl2 08	16.000 U	16.000 U
Cl3 18	1.385 U	1.385 U
Cl3 28	1.730 U	1.730 U
Cl4 44	1.453 U	1.453 U
Cl4 49	1.487 U	1.487 U
Cl4 52	1.435 U	1.435 U
Cl4 66	1.490 U	1.490 U
Cl5 87	1.128 U	1.128 U
Cl5 101	1.145 U	1.145 U
Cl5 105	0.575 U	0.575 U
Cl5 118	0.868 U	0.868 U
Cl6 128	1.400 U	1.400 U
Cl6 138	1.325 U	1.325 U
Cl6 153	1.065 U	1.065 U
Cl7 170	1.015 U	1.015 U
Cl7 180	0.960 U	0.960 U
Cl7 183	0.930 U	0.930 U
Cl7 184	0.918 U	0.918 U
Cl7 187	0.862 U	0.862 U
Cl8 195	1.085 U	1.085 U
Cl9 206	1.217 U	1.217 U
Cl10 209	1.265 U	1.265 U
Surrogate Recoveries:		
Cl3(34)	82	81
Cl5(112)	90	86

U - Analyte not detected, ssMDL value inserted.
J - Analyte detected below the ssMDL value.
N - QC value outside the accuracy or precision DQO.

Triplicate Data

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Project Name: Ponce Harbor
Project Number: N005561
Site Water Data

Client Sample ID:	E-PH03-1W	E-PH03-1W	E-PH03-1W	
Battelle Sample ID:	U8093	U8093DUP	U8093TRP	
Battelle Batch ID:	03-0240	03-0240	03-0240	
Sample Volume (L):	1.00	1.00	1.00	**RSD**
Units:	ng/L	ng/L	ng/L	%
Aldrin	2.826 U	2.826 U	2.826 U	NA
a Chlordane	1.078 U	1.078 U	1.078 U	NA
trans Nonachlor	1.013 U	1.013 U	1.013 U	NA
Dieldrin	0.978 U	0.978 U	0.978 U	NA
4,4 DDT	0.557 U	0.557 U	0.557 U	NA
2,4 DDT	1.985 U	1.985 U	1.985 U	NA
4,4 DDD	0.595 U	0.595 U	0.595 U	NA
2,4 DDD	0.747 U	0.747 U	0.747 U	NA
4,4 DDE	0.838 U	0.838 U	0.838 U	NA
2,4 DDE	1.706 U	1.706 U	1.706 U	NA
Endosulfan I	1.110 U	1.110 U	1.110 U	NA
Endosulfan II	0.510 U	0.510 U	0.510 U	NA
Endosulfan sulfate	0.565 U	0.565 U	0.565 U	NA
Heptachlor	1.173 U	1.173 U	1.173 U	NA
Heptachlor epoxide	0.948 U	0.948 U	0.948 U	NA
Cl2 08	16.000 U	16.000 U	16.000 U	NA
Cl3 18	1.385 U	1.385 U	1.385 U	NA
Cl3 28	1.730 U	1.730 U	1.730 U	NA
Cl4 44	1.453 U	1.453 U	1.453 U	NA
Cl4 49	1.487 U	1.487 U	1.487 U	NA
Cl4 52	1.435 U	1.435 U	1.435 U	NA
Cl4 66	1.490 U	1.490 U	1.490 U	NA
Cl5 87	1.128 U	1.128 U	1.128 U	NA
Cl5 101	1.145 U	1.145 U	1.145 U	NA
Cl5 105	0.575 U	0.575 U	0.575 U	NA
Cl5 118	0.868 U	0.868 U	0.868 U	NA
Cl6 128	1.400 U	1.400 U	1.400 U	NA
Cl6 138	1.325 U	1.325 U	1.325 U	NA
Cl6 153	1.065 U	1.065 U	1.065 U	NA
Cl7 170	1.015 U	1.015 U	1.015 U	NA
Cl7 180	0.960 U	0.960 U	0.960 U	NA
Cl7 183	0.930 U	0.930 U	0.930 U	NA
Cl7 184	0.918 U	0.918 U	0.918 U	NA
Cl7 187	0.862 U	0.862 U	0.862 U	NA
Cl8 195	1.085 U	1.085 U	1.085 U	NA
Cl9 206	1.217 U	1.217 U	1.217 U	NA
Cl10 209	1.265 U	1.265 U	1.265 U	NA
Surrogate Recoveries:				
Cl3(34)	82	82	75	
Cl5(112)	90	90	81	

U - Analyte not detected, ssMDL value inserted.
J - Analyte detected below the ssMDL value.
N - QC value outside the accuracy or precision DQO.

Triplicate Data

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Project Name: Ponce Harbor
Project Number: N005561
Elutriate Data

Client Sample ID:	Ponce Harbor Elut	Ponce Harbor Elut	Ponce Harbor Elut	
Battelle Sample ID:	U8253	U8253DUP	U8253TRP	
Battelle Batch ID:	03-0240	03-0240	03-0240	
Sample Volume (L):	1.00	1.00	1.00	RSD
Units:	ng/L	ng/L	ng/L	%
Aldrin	2.826 U	2.826 U	2.826 U	NA
a Chlordane	1.078 U	1.078 U	1.078 U	NA
trans Nonachlor	1.013 U	1.013 U	1.013 U	NA
Dieldrin	0.978 U	0.978 U	0.978 U	NA
4,4 DDT	0.557 U	0.557 U	0.557 U	NA
2,4 DDT	1.985 U	1.985 U	1.985 U	NA
4,4 DDD	0.595 U	0.595 U	0.595 U	NA
2,4 DDD	0.747 U	0.747 U	0.747 U	NA
4,4 DDE	0.838 U	0.838 U	0.838 U	NA
2,4 DDE	1.706 U	1.706 U	1.706 U	NA
Endosulfan I	1.110 U	1.110 U	1.110 U	NA
Endosulfan II	0.510 U	0.510 U	0.510 U	NA
Endosulfan sulfate	0.565 U	0.565 U	0.565 U	NA
Heptachlor	1.173 U	1.173 U	1.173 U	NA
Heptachlor epoxide	0.948 U	0.948 U	0.948 U	NA
Cl2 08	16.000 U	16.000 U	16.000 U	NA
Cl3 18	1.385 U	1.385 U	1.385 U	NA
Cl3 28	1.730 U	1.730 U	1.730 U	NA
Cl4 44	1.453 U	1.453 U	1.453 U	NA
Cl4 49	1.487 U	1.487 U	1.487 U	NA
Cl4 52	1.435 U	1.435 U	1.435 U	NA
Cl4 66	1.490 U	1.490 U	1.490 U	NA
Cl5 87	1.128 U	1.128 U	1.128 U	NA
Cl5 101	1.145 U	1.145 U	1.145 U	NA
Cl5 105	0.575 U	0.575 U	0.575 U	NA
Cl5 118	0.868 U	0.868 U	0.868 U	NA
Cl6 128	1.400 U	1.400 U	1.400 U	NA
Cl6 138	1.325 U	1.325 U	1.325 U	NA
Cl6 153	1.065 U	1.065 U	1.065 U	NA
Cl7 170	1.015 U	1.015 U	1.015 U	NA
Cl7 180	0.960 U	0.960 U	0.960 U	NA
Cl7 183	0.930 U	0.930 U	0.930 U	NA
Cl7 184	0.918 U	0.918 U	0.918 U	NA
Cl7 187	0.862 U	0.862 U	0.862 U	NA
Cl8 195	1.085 U	1.085 U	1.085 U	NA
Cl9 206	1.217 U	1.217 U	1.217 U	NA
Cl10 209	1.265 U	1.265 U	1.265 U	NA
Surrogate Recoveries:				
Cl3(34)	81	86	85	
Cl5(112)	86	94	92	

U - Analyte not detected, ssMDL value inserted.
J - Analyte detected below the ssMDL value.
N - QC value outside the accuracy or precision DQO.

Laboratory Control Sample Data

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Project Name: Ponce Harbor
Project Number: N005561

Client Sample ID:		NA	
Battelle Sample ID:		BB717LCS	
Battelle Batch ID:		03-0240	
Sample Volume (L):		NA	
Units:	ng	ng	%
	FW67		Recovery
Aldrin	75.100	70.993	95
a Chlordane	75.375	73.839	98
trans Nonachlor	75.025	68.516	91
Dieldrin	75.075	71.683	95
4,4 DDT	75.100	70.533	94
2,4 DDT	75.250	69.126	92
4,4 DDD	75.075	79.475	106
2,4 DDD	75.025	69.396	92
4,4 DDE	75.050	68.443	91
2,4 DDE	75.250	89.398	119
Endosulfan I	75.075	57.974	77
Endosulfan II	75.075	60.301	80
Endosulfan sulfate	75.075	69.260	92
Heptachlor	75.075	66.550	89
Heptachlor epoxide	75.100	67.073	89
Cl2 08	75.100	69.896	93
Cl3 18	75.100	64.124	85
Cl3 28	75.100	69.876	93
Cl4 44	75.100	71.316	95
Cl4 49	75.100	72.803	97
Cl4 52	75.100	70.570	94
Cl4 66	75.100	70.806	94
Cl5 87	75.100	73.446	98
Cl5 101	75.100	74.343	99
Cl5 105	75.100	71.506	95
Cl5 118	75.100	56.474	75
Cl6 128	75.100	69.163	92
Cl6 138	75.100	72.056	96
Cl6 153	75.100	63.567	85
Cl7 170	75.100	72.166	96
Cl7 180	75.100	73.439	98
Cl7 183	75.100	73.376	98
Cl7 184	75.100	71.120	95
Cl7 187	75.100	68.976	92
Cl8 195	75.100	73.776	98
Cl9 206	75.100	69.653	93
Cl10 209	75.100	74.616	99

Surrogate Recoveries:

Cl3(34)	93
Cl5(112)	91

U - Analyte not detected, ssMDL value inserted.
J - Analyte detected below the ssMDL value.
N - QC value outside the accuracy or precision DQO.

Matrix Spike Data

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Project Name: Ponce Harbor
Project Number: N005561

Client Sample ID:		E-PH03-1W	E-PH03-1W	
Battelle Sample ID:		U8093	U8093MS	
Battelle Batch ID:		03-0240	03-0240	
Sample Volume (L):		1.00	1.00	
Units:	ng	ng/L	ng/L	%
	FW67			Recovery
Aldrin	75.100	2.826 U	66.697	89
a Chlordane	75.375	1.078 U	73.483	97
trans Nonachlor	75.025	1.013 U	70.613	94
Dieldrin	75.075	0.978 U	68.563	91
4,4 DDT	75.100	0.557 U	73.739	98
2,4 DDT	75.250	1.985 U	69.180	92
4,4 DDD	75.075	0.595 U	76.976	103
2,4 DDD	75.025	0.747 U	68.803	92
4,4 DDE	75.050	0.838 U	66.287	88
2,4 DDE	75.250	1.706 U	65.487	87
Endosulfan I	75.075	1.110 U	76.332	102
Endosulfan II	75.075	0.510 U	66.100	88
Endosulfan sulfate	75.075	0.565 U	71.273	95
Heptachlor	75.075	1.173 U	66.673	89
Heptachlor epoxide	75.100	0.948 U	67.333	90
Cl2 08	75.100	16.000 U	67.970	91
Cl3 18	75.100	1.385 U	60.254	80
Cl3 28	75.100	1.730 U	70.863	94
Cl4 44	75.100	1.453 U	71.456	95
Cl4 49	75.100	1.487 U	72.806	97
Cl4 52	75.100	1.435 U	70.133	93
Cl4 66	75.100	1.490 U	74.723	99
Cl5 87	75.100	1.128 U	71.653	95
Cl5 101	75.100	1.145 U	75.689	101
Cl5 105	75.100	0.575 U	76.799	102
Cl5 118	75.100	0.868 U	70.300	94
Cl6 128	75.100	1.400 U	73.016	97
Cl6 138	75.100	1.325 U	72.633	97
Cl6 153	75.100	1.065 U	61.900	82
Cl7 170	75.100	1.015 U	72.309	96
Cl7 180	75.100	0.960 U	74.059	99
Cl7 183	75.100	0.930 U	73.719	98
Cl7 184	75.100	0.918 U	71.016	95
Cl7 187	75.100	0.862 U	69.176	92
Cl8 195	75.100	1.085 U	74.919	100
Cl9 206	75.100	1.217 U	70.743	94
Cl10 209	75.100	1.265 U	74.199	99

Surrogate Recoveries:

Cl3(34)	82	93
Cl5(112)	90	89

U - Analyte not detected, ssMDL value inserted.
J - Analyte detected below the ssMDL value.
N - QC value outside the accuracy or precision DQO.

Standard Reference Material Data
Surrogate Corrected

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Project Name: Ponce Harbor
Project Number: N005561

Client Sample ID:	SRM FW21		
Battelle Sample ID:	BB718SRM		
Battelle Batch ID:	03-0240		
Sample Volume (L):	1.00	FW21	
Units:	ng/L	ng	PD
a Chlordane	91.301	78.875	16
trans Nonachlor	89.815	79.450	13
Dieldrin	80.060	78.875	2
4,4 DDT	85.385	78.675	9
2,4 DDT	78.782	78.350	1
4,4 DDD	84.292	79.675	6
4,4 DDE	80.299	79.050	2
2,4 DDE	82.258	79.050	4
Heptachlor	87.154	79.075	10
Heptachlor epoxide	81.136	79.075	3
Cl2 08	85.457	85.600	0
Cl3 18	80.341	82.325	2
Cl4 44	93.937	82.150	14
Cl4 52	90.260	82.675	9
Cl4 66	95.871	82.050	17
Cl5 87	80.479	82.750	3
Cl5 101	94.470	81.400	16
Cl5 105	87.380	81.675	7
Cl5 118	63.346	82.550	23
Cl6 128	84.112	82.350	2
Cl6 138	82.348	81.075	2
Cl6 153	78.269	81.600	4
Cl7 170	86.334	81.800	6
Cl7 180	87.223	82.400	6
Cl7 187	83.222	81.875	2
Cl8 195	88.313	82.075	8
Cl9 206	79.413	80.050	1
Cl10 209	86.847	82.475	5

Surrogate Recoveries:

Cl3(34)	79
Cl5(112)	85

U - Analyte not detected, ssMDL value inserted.
J - Analyte detected below the ssMDL value.
N - QC value outside the accuracy or precision DQO.
Percent difference calculated vs closest range lower or higher.

Sediment Metals Data

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QA/QC NARRATIVE

PROJECT: Chemical Evaluation of Dredged Material from Ponce Harbor
PARAMETER: Metals
LABORATORY: Battelle Marine Sciences Laboratory, Sequim, Washington
MATRIX: Sediment

SAMPLE CUSTODY AND PROCESSING: Two sediment samples (one harbor reference and one site composite) for metals analyses were received on 03/12/2003. All samples were received in good condition (i.e., no sample containers were broken). Samples were assigned a Battelle Central File (CF) identification number (1982) and were entered into Battelle's central tracking system.

The following lists information on sample receipt and processing activities:

Lab Sample IDs:	1982*34, 35
Description:	Marine Sediment
Sample collection dates	Provided by client
Laboratory arrival date	03/12/2003
Cooler temp. on arrival	5.8°C
Deep Freeze (-58 °C ± 20 °C)	03/12/2003
Freeze Dryer (MSL-C-003)	03/12/2003
Digestion 1 (MSL-I-006 Boric Acid)	03/19/2003
Digestion 2 (MSL-I-006 HNO_3/HCl)	03/17/2003
CVAA analysis (Hg, MSL-I-016)	03/20/2003
FIAS analysis (Se, MSL-I-030)	03/19/2003
GFAA analysis (Ag, MSL-I-029)	03/21/03, 03/24/03, 03/25/03
ICP-MS analysis (MSL-I-022)	Digestion 1 03/25/03
(Al, As, Cd, Cr, Cu, Fe, Pb, Ni and Zn)	Digestion 2 03/18/2003, 03/19/03 (reruns)

DATA QUALITY OBJECTIVES:

Analyte	Analytical Method	Range of Recovery	SRM Accuracy [1]	Relative Precision [1]	Achieved Detection Limits [2] (µg/g dry wt.)
Al	ICP-MS	75-125%	≤20%	20%	0.5
As	ICP-MS	75-125%	≤20%	20%	0.05
Cd	ICP-MS	75-125%	≤20%	20%	0.01
Cr	ICP-MS	75-125%	≤20%	20%	0.04
Cu	ICP-MS	75-125%	≤20%	20%	0.02
Fe	ICP-MS	75-125%	≤20%	20%	5
Pb	ICP-MS	75-125%	≤20%	20%	0.01
Hg	CVAA	75-125%	≤20%	20%	0.002
Ni	ICP-MS	75-125%	≤20%	20%	0.03
Ag	GFAA	75-125%	≤20%	20%	0.03
Se	FIAS	75-125%	≤20%	20%	0.08
Zn	ICP-MS	75-125%	≤20%	20%	0.1

[1] Evaluated for analytes > 10 times the MDL.
[2] Sediment MDL from the QAPP

QA/QC NARRATIVE

METHODS: Sediment samples were analyzed for 12 metals: aluminum (Al), arsenic (As), cadmium (Cd), chromium (Cr), copper (Cu), iron (Fe), lead (Pb), mercury (Hg), nickel (Ni), silver (Ag), selenium (Se), and zinc (Zn). Samples were freeze-dried and homogenized using a ball-mill prior to digestion according to Battelle SOP MSL-C-003, *Percent Dry Weight and Homogenizing Dry Sediment, Soil and Tissue.* Sediment samples were digested using two methods: 1) Boric acid (BA) in accordance with Battelle SOP MSL-I-006, *Mixed Acid Sediment Digestion.* An approximately 200-mg (dry weight) aliquot of each sample was combined with nitric, hydrochloric and hydrofluoric acids in a Teflon vessel and heated in an oven at 130ºC (±10ºC) for a minimum of 8 hours. The addition of boric acid neutralized the hydrofluoric acid and deionized water was added to the sediment digestate to achieve analysis volume. 2) HNO_3/HCl (Aqua regia) in accordance with Battelle SOP MSL-I-006, *Mixed Acid Sediment Digestion.* An approximately 200-mg (dry weight) aliquot of each sample was combined with nitric and hydrochloric acids (aqua regia) in a Teflon bomb and heated in an oven at 130ºC (±10ºC) for a minimum of eight hours. After heating and cooling, deionized water was added to the sediment digestate to achieve analysis volume. Digestates were submitted for analysis by four methods: ICP-MS, CVAA, FIAS and GFAA.

Sample digestates were analyzed for Al, As, Cd, Cr, Cu, Fe, Ni, Pb, and Zn using inductively coupled plasma-mass spectrometry (ICP-MS) according to Battelle SOP MSL-I-022, *Determination of Elements in Aqueous and Digestate Samples by ICP/MS.* Analytes reported from the boric acid digestion include: Al, Cr and Fe. Analytes reported from the aqua regia digestion include: As, Cd, Cu, Ni, Pb and Zn.

Samples digested using aqua regia were analyzed for Hg using cold-vapor atomic absorption spectroscopy (CVAA) according to Battelle SOP MSL-I-016, *Total Mercury in Tissues and Sediments by Cold Vapor Atomic Absorption.*

Samples digested using aqua regia were analyzed for Se using hydride generation flow injection atomic sprectroscopy (FIAS) according to Battelle SOP MSL-I-030 *Determination of Metals in Aqueous and Digestate Samples by HGAA-FIAS.*

Samples digested using boric acid were analyzed for Ag by graphite furnace atomic absorption (GFAA) according to Battelle SOP MSL-I-029, *Determination of Metals in Aqueous and Disgestate samples by GFAA.*

All results were reported in units of µg/g on a dry-weight basis.

HOLDING TIMES: The recommended holding times for metals analyses in sediment are 28 days from sample collection for Hg analysis and 6 months for analysis of all other metals. Recommended holding times were achieved for all metals.

DETECTION LIMITS: Analytical results were reported to laboratory achieved detection limits listed in the QAPP from the 2002 sediment MDL study. Target detection limits are listed in the USACE – NYD EPA Region II Regional Testing Manual. Laboratory achieved detection limits were below the targets for all analytes. The data were evaluated and flagged to the following criteria:

B Analyte detected in the blank is greater than 3 times the MDL
U Undetected at or above MDL; MDL reported
& QC value outside the accuracy or precision DQOs

QA/QC NARRATIVE

METHOD BLANKS:

One method blank was analyzed with the set of sediment samples. The concentrations of As, Cd, Cu, Ni, Pb and Zn in the method blank are determined from the aqua regia digestion and reported from the reanalysis of the method blank R1 in data set ID ICP-MS 031903-6100a. The original analysis of the digestion batch is reported from data set ID ICP-MS 031803-6100a. The blank, along with R1 of Mess-3 and 1944 were reanalyzed due to instrumental problems early in the analysis run ID ICP-MS 031803-6100a. The concentration of the method blank did not exceed the QC criteria of three times the MDL for all analytes, with the following exceptions. The detected level of Al in the method blank was above the acceptance criterion, but sample concentrations were not impacted, as the value of the blank is less than 0.5% of the sample concentration. The method blank contained detectable quantities of As, Cr and Zn. The values detected in the blank were less than 1% of the sample concentrations, therefore no corrective action was required. The data are not blank corrected.

MATRIX SPIKE ACCURACY:

One sediment sample (reference sediment) was selected and spiked at approximately four concentrations: 1 µg/g for Ag, Cd, Hg and Se; 50 µg/g for As, Cu, Ni and Pb; 100 µg/g for Cr and Zn; 5000 µg/g for Al and Fe µg/g. Matrix spike recoveries ranged from 84% to 113% and were within the QC criteria of 75-125% recovery.

STANDARD REFERENCE MATERIAL ACCURACY:

Two sediment SRMs were analyzed with the set of samples: MESS-3 and 1944. SRM accuracy is expressed as the percent difference (PD) between the certified and measured concentrations for each metal of interest. The concentrations of As, Cd, Cu, Ni, Pb and Zn in R1 of Mess-3 and 1944 are reported from the reanalysis data set ID ICP-MS 031903-6100a. The original analyses of both SRMs were not reported due to instrumental problems early in the analysis run ID ICP-MS 031803-6100a. Secondary replicates of these standards (reported with the Ponce ODMDS sediment) validate the accuracy of the original data set.

One replicate of MESS-3 was analyzed with the samples. The percent differences for all metals, except Hg, certified at an appropriate concentration were within the QC criteria and ranged from 0% to 14% PD. Mercury in SRM MESS-3 (31%) was outside the QC criteria. An evaluation of MESS-3 recoveries from January through March identified a consistently high recovery of this SRM for Hg. Twenty-one replicates digested by various methods and analyzed for Hg have an average PD of 21% with a standard deviation of 8%. This may indicate that the lot of MESS-3 used for digestions contains mercury elevated above the certified value, but the sample concentrations were not altered during the digestion process, as the alternate SRM 1944 was within the QC criterion. Metals certified <10x the achieved detection limits for this SRM (Ag) were not used to evaluate data quality.

One replicate of SRM 1944 was analyzed with the samples. The percent differences for all metals certified at an appropriate concentration were within the QC criteria and ranged from 0% to 18% PD.

REPLICATE PRECISION:

Precision for this set of samples was assessed by the analysis of laboratory triplicates and expressed as the relative standard deviation (RSD) of replicate results. The harbor composite sample was analyzed in triplicate. The RSD values were within the QC criteria and ranged from 1% to 7% RSD. Replicate precision was evaluated for sample concentrations greater than 10 times the MDL. Data set precision for Ag was evaluated using the three replicates of SRM 1944, which were digested and analyzed with the samples. The three replicates were 6.74µg/g, 6.00µg/g, and 6.01µg/g with an RSD of 7%. The additional replicates for 1944 are reported with the Ponce ODMDS sediments.

BATTELLE MARINE SCIENCES LABORATORIES
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CHEMICAL EVALUATION OF DREDGE MATERIAL FROM PONCE HARBOR
Metals in Sediment
(Samples Received 3/12/03)

CHEMICAL EVALUATION OF DREDGE MATERIAL FROM PONCE HARBOR
Metals in Sediment
(Samples Received 3/12/03)

(concentrations in µg/g dry wt, data are not blank corrected)

MSL Code	Duxbury ID	Client ID	Percent Moisture	Al	Ag	As	Cd	Cr	Cu	Fe	Hg	Ni	Pb	Se	Zn
		Analytical Batch ID:		*032503-6100a*	*T032503A*	*031803-6100a*	*031803-6100a*	*032503-6100a*	*031803-6100a*	*032503-6100a*	*032203HGB2*	*031803-6100a*	*031803-6100a*	*F031903A*	*031803-6100a*
		Digestion:		*Boric Acid*	*Boric Acid*	*Aqua Regia*	*Aqua Regia*	*Boric Acid*	*Aqua Regia*	*Boric Acid*	*Aqua Regia*	*Aqua Regia*	*Aqua Regia*	*Aqua Regia*	*Aqua Regia*
1982-34	U8240	RS-PH03	54.7	59900 B	0.03 U	7.65 B	0.0369	39.7 B	42.5	44200	0.0447	20.1	9.33	0.273	64.9 B
1982-35	U8241	E-PH03-SC	32.7	70000 B	0.740	7.70 B	0.0620	65.0 B	47.0	53000	0.0266	23.8	5.05	0.224	71.6 B
Laboratory Achieved Detection Limits [1]				0.5	0.03	0.05	0.01	0.04	0.02	5	0.002	0.03	0.01	0.08	0.1
		*B flag criteria (3 * MDL)*		*1.5*	*0.09*	*0.2*	*0.03*	*0.1*	*0.06*	*15*	*0.006*	*0.09*	*0.03*	*0.2*	*0.3*
METHOD BLANK															
Blank 031703 r1 [2]				141 B	0.03 U	0.244 B	0.0165	0.245 B	0.0508	5 U	0.002 U	0.03 U	0.0248	0.08 U	0.660 B
MATRIX SPIKE RESULTS															
Spike Concentration				4762	0.976	49.0	1.0	95.2	49.0	4762	1.01	49.0	49.0	1.01	98.0
1982-34	U8240	RS-PH03	54.7	59900	0.03 U	7.65	0.0369	39.7	42.5	44200	0.0447	20.1	9.33	0.273	64.9
1982-34 MS				63900	1.01	55.4	1.00	142	85.9	49600	1.08	63.4	62.2	1.20	148
Percent Recovery				**84%**	**103%**	**97%**	**95%**	**107%**	**89%**	**113%**	**103%**	**88%**	**108%**	**92%**	**85%**
REPLICATE ANALYSIS RESULTS															
1982-35 r1	U8241	E-PH03-SC	32.7	70000	0.740	7.70	0.0620	65.0	47.0	53000	0.0266	23.8	5.05	0.224	71.6
1982-35 r2	U8241	E-PH03-SC	32.7	71700	0.0704	7.90	0.0588	65.7	48.8	54600	0.0272	24.6	5.11	0.247	73.4
1982-35 r3	U8241	E-PH03-SC	32.7	71000	0.111	7.95	0.0652	63.9	48.9	54100	0.0271	24.6	5.07	0.214	73.0
	Mean			70900	0.307	7.85	0.0620	64.9	48.2	53900	0.0270	24.3	5.08	0.228	72.7
	RSD			**1%**	**NC**	**2%**	**5%**	**1%**	**2%**	**2%**	**1%**	**2%**	**1%**	**7%**	**1%**
STANDARD REFERENCE MATERIAL															
MESS-3 R1 [2]				73700	0.151	20.4	0.225	110	31.1	43200	0.119	44.6	21.2	0.725	168
	certified value			85900	0.18	21.2	0.24	105	33.9	43400	0.091	46.9	21.1	0.72	159
	range			±2300	±0.02	±1.1	±0.01	±4	±1.6	±1100	±0.009	±2.2	±0.7	±0.05	±8
	percent difference			**14%**	**NC**	**4%**	**6%**	**5%**	**8%**	**0%**	**31% &**	**5%**	**0%**	**1%**	**6%**
1944 R1 [2]				43600	6.74	18.4	8.53	253	344	35300	3.66	68.8	316	NA	590
	certified value			53300	6.34	18.9	8.8	266	380	35300	3.4	76.1	330		656
	range			±4900	±1.7	±2.8	±1.4	±24	±40	±1600	±0.05	±5.6	±48		±75
	percent difference			**18%**	**6%**	**3%**	**3%**	**5%**	**9%**	**0%**	**8%**	**10%**	**4%**		**10%**

B Analyte detected in blank is greater than 3x MDL
U Undetected at or above MDL; MDL reported
& QC value outside the accuracy or precision DQO (SRM and RSD <20%, Spike Recoveries ±25%)
NA Not applicable/available
NC Not calculated. QC sample not appropriate for evaluation of DQOs. See narrative.
[1] Sediment MDLs (as listed in QAPP) determined from analysis of 7-replicates, spiked water, MDL study, and calculated for sediments using representative dry sample mass and instrument dilution factor
[2] As, Cd, Cu, Ni, Pb and Zn for Blank R1, Mess-3 R1 and 1944 R1 are reported from 031903-6100a

Sediment Chlorinated Hydrocarbon Data

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PCB/Pesticide – SEDIMENT QA/QC SUMMARY
Batch 03-0231

PROJECT: Ponce Harbor
PARAMETER: PCB/Pesticides
LABORATORY: Battelle, Duxbury, MA
MATRIX: Sediment
SAMPLE CUSTODY: The sediment sample composite was received at Battelle Duxbury on March 11, 2003. Sediment was received in good condition. The cooler temperature on arrival was –0.9 °C. Sediment was stored at 4 °C until processing began.

QA/QC DATA QUALITY OBJECTIVES:

	Reference Method	Blank Criteria	Surrogate Recovery	LCS/MS Recovery	SRM % Diff.	Sample Replicate Relative Precision	Achieved Detection Limit (ng/g DW)
PCB/ Pest	General NS&T	B<3xMDL	30-120% Recovery	50-120% Recovery (for at least 80% of analytes; analyte conc. in MS must be >10x background)	≤25% PD (on average; not to exceed 35% for more than 30% of analytes; measured from range of certified values, for analytes >10x MDL)	≤30% RSD	~ 0.03 – 0.1 **Target DL:** 0.4

METHOD: Sediment samples were extracted for PCB and pesticides following general NS&T methods. Approximately 30 g of sediment was spiked with surrogates and extracted three times with dichloromethane using shaker table techniques. The combined extract was dried over anhydrous sodium sulfate, concentrated, processed through alumina cleanup column, concentrated, and further purified by GPC/HPLC. The post-HPLC extract was concentrated, fortified with recovery internal standards and split for the required analyses. Extracts intended for PCB and pesticide analysis were solvent exchanged into hexane and analyzed using gas chromatography/electron capture detection (GC/ECD), following general NS&T methods. Sample data were quantified by the method of internal standards, using the Recovery Internal Standard (RIS) compounds.

HOLDING TIMES: Samples were prepared for analysis in one analytical batch and were extracted within 14 days of sample collection and analyzed within 40 days of extraction.

Batch	Extraction Date	Analysis Date
03-0231	03/12/2003	03/23/2003 – 03/24/2003

BLANKS: A procedural blank (PB) was prepared with each analytical batch. Blanks were analyzed to ensure the sample extraction and analysis methods were free of contamination.

03-0231 – No target analytes were detected in the blank

Comments – None.

PCB/Pesticide – SEDIMENT QA/QC SUMMARY
Batch 03-0231

LABORATORY CONTROL SAMPLE (Blank Spike)

A laboratory control sample (LCS) was prepared with each analytical batch. The percent recoveries of target PCB and pesticides were calculated to measure data quality in terms of accuracy.

03-0231 – All target analytes were recovered within the laboratory control limits.

Comments – None.

MATRIX SPIKE:

A matrix spike (MS) sample was prepared with each analytical batch. The percent recoveries of target analytes were calculated to measure data quality in terms of accuracy.

03-0231 – Percent recoveries for all target analytes were within data quality criteria.

Comments – None.

SURROGATES:

Three surrogate compounds were added prior to extraction, including Cl3(34) and Cl5(112). The recovery of each surrogate compound was calculated to measure data quality in terms of accuracy (extraction efficiency).

03-0231 – Percent recoveries for all surrogate compounds were within the laboratory control limits.

Comments – None.

REPLICATES:

Replicate (triplicate) samples for sediments were prepared with each analytical batch. The RSD between triplicate analyses for PCB and pesticides is calculated to measure data quality in terms of precision.

03-0231 – The RSD between triplicate analyses of all PCB and pesticides were within the DQO of <30% RSD.

Comments – None.

SRM:

A standard reference material (SRM, NIST 1944) was prepared with each analytical batch. The percent difference (PD) between the measured value and the certified range was calculated to measure data quality in terms of accuracy.

03-0231 – SRM PDs were within the control limits for all certified target analytes except trans nonachlor and 4,4'-DDT. Average PD = 17%

Comments – The percent difference for trans nonachlor and 4,4'-DDT were 66 and 53%, respectively (over-recovery in the SRM). The concentration of trans nonachlor in this SRM is relatively low (certified value ~8 ng/g). The concentration of 4,4'-DDT is relatively high in the SRM – there may have been a chromatographic interference. All other data quality criteria required for SRMs are met. The recoveries of these two compounds were acceptable in the LCS, MS, and IC samples. Chromatography was checked, as were the calibrations and calculations; no errors found. Neither compound was detected in the test sediment.

Procedural Blank Data

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Project Name: Ponce Harbor
Project Number: N005561

Client Sample ID:	NA
Battelle Sample ID:	BB671PB
Percent Moisture (%)	NA
Battelle Batch ID:	03-0231
Sample Dry Weight (g):	23.00
Sample Wet Weight (g):	41.00
Units:	ng/g Dry Wt.

Aldrin	0.041 U
a Chlordane	0.047 U
trans Nonachlor	0.053 U
Dieldrin	0.062 U
4,4 DDT	0.060 U
2,4 DDT	0.081 U
4,4 DDD	0.063 U
2,4 DDD	0.069 U
4,4 DDE	0.058 U
2,4 DDE	0.086 U
Endosulfan I	0.066 U
Endosulfan II	0.064 U
Endosulfan sulfate	0.067 U
Heptachlor	0.053 U
Heptachlor epoxide	0.049 U
Cl2 08	0.106 U
Cl3 18	0.050 U
Cl3 28	0.073 U
Cl4 44	0.065 U
Cl4 49	0.076 U
Cl4 52	0.069 U
Cl4 66	0.077 U
Cl5 87	0.057 U
Cl5 101	0.066 U
Cl5 105	0.058 U
Cl5 118	0.076 U
Cl6 128	0.123 U
Cl6 138	0.063 U
Cl6 153	0.094 U
Cl7 170	0.062 U
Cl7 180	0.066 U
Cl7 183	0.059 U
Cl7 184	0.046 U
Cl7 187	0.061 U
Cl8 195	0.057 U
Cl9 206	0.056 U
Cl10 209	0.061 U

Surrogate Recoveries (%):

Cl3(34)	72
Cl5(112)	76

U - Analyte not detected, ssMDL value inserted.
J - Analyte detected below the ssMDL value.
N - QC value outside the accuracy or precision DQO.

Field Sample Data

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Project Name: Ponce Harbor
Project Number: N005561

Client Sample ID:	E-PH03-SC	RS-PH03
Battelle Sample ID:	U8241	U8240
Percent Moisture (%)	32.57	54.91
Battelle Batch ID:	03-0231	03-0231
Sample Dry Weight (g):	27.46	18.35
Sample Wet Weight (g):	40.72	40.7
Units:	ng/g Dry Wt.	ng/g Dry Wt.
Aldrin	0.034 U	0.027 U
a Chlordane	0.039 U	0.031 U
trans Nonachlor	0.044 U	0.035 U
Dieldrin	0.052 U	0.041 U
4,4 DDT	0.050 U	0.063
2,4 DDT	0.068 U	0.053 U
4,4 DDD	0.044 J	0.070
2,4 DDD	0.058 U	0.045 U
4,4 DDE	0.022 J	0.320
2,4 DDE	0.072 U	0.057 U
Endosulfan I	0.055 U	0.043 U
Endosulfan II	0.054 U	0.042 U
Endosulfan sulfate	0.056 U	0.044 U
Heptachlor	0.044 U	0.035 U
Heptachlor epoxide	0.041 U	0.032 U
Cl2 08	0.089 U	0.070 U
Cl3 18	0.042 U	0.033 U
Cl3 28	0.061 U	0.048 U
Cl4 44	0.055 U	0.043 U
Cl4 49	0.064 U	0.050 U
Cl4 52	0.058 U	0.045 U
Cl4 66	0.064 U	0.051 U
Cl5 87	0.048 U	0.037 U
Cl5 101	0.056 U	0.044 U
Cl5 105	0.049 U	0.038 U
Cl5 118	0.063 U	0.050 U
Cl6 128	0.103 U	0.081 U
Cl6 138	0.053 U	0.041 U
Cl6 153	0.018 J	0.062 U
Cl7 170	0.052 U	0.041 U
Cl7 180	0.055 U	0.044 U
Cl7 183	0.049 U	0.039 U
Cl7 184	0.039 U	0.031 U
Cl7 187	0.051 U	0.040 U
Cl8 195	0.048 U	0.038 U
Cl9 206	0.047 U	0.037 U
Cl10 209	0.051 U	0.038 U
Surrogate Recoveries:		
Cl3(34)	68	65
Cl5(112)	72	69

U - Analyte not detected, ssMDL value inserted.
J - Analyte detected below the ssMDL value.
N - QC value outside the accuracy or precision DQO.

Triplicate Data

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Project Name: Ponce Harbor
Project Number: N005561

Client Sample ID:	E-PH03-SC	E-PH03-SC	E-PH03-SC	
Battelle Sample ID:	U8241	U8241DUP	U8241TRP	
Percent Moisture (%)	32.57	32.57	32.57	
Battelle Batch ID:	03-0231	03-0231	03-0231	
Sample Dry Weight (g):	27.46	27.46	27.02	
Sample Wet Weight (g):	40.72	40.72	40.07	
Units:	ng/g Dry Wt.	ng/g Dry Wt.	ng/g Dry Wt.	% RSD
Aldrin	0.034 U	0.034 U	0.035 U	NA
a Chlordane	0.039 U	0.039 U	0.040 U	NA
trans Nonachlor	0.044 U	0.044 U	0.045 U	NA
Dieldrin	0.052 U	0.052 U	0.053 U	NA
4,4 DDT	0.050 U	0.050 U	0.051 U	NA
2,4 DDT	0.068 U	0.068 U	0.069 U	NA
4,4 DDD	0.044 J	0.045 J	0.046 J	2
2,4 DDD	0.058 U	0.058 U	0.059 U	NA
4,4 DDE	0.022 J	0.029 J	0.029 J	15
2,4 DDE	0.072 U	0.072 U	0.073 U	NA
Endosulfan I	0.055 U	0.055 U	0.056 U	NA
Endosulfan II	0.054 U	0.054 U	0.054 U	NA
Endosulfan sulfate	0.056 U	0.056 U	0.057 U	NA
Heptachlor	0.044 U	0.044 U	0.045 U	NA
Heptachlor epoxide	0.041 U	0.041 U	0.042 U	NA
Cl2 08	0.089 U	0.089 U	0.090 U	NA
Cl3 18	0.042 U	0.042 U	0.043 U	NA
Cl3 28	0.061 U	0.061 U	0.062 U	NA
Cl4 44	0.055 U	0.055 U	0.056 U	NA
Cl4 49	0.064 U	0.064 U	0.065 U	NA
Cl4 52	0.058 U	0.058 U	0.059 U	NA
Cl4 66	0.064 U	0.064 U	0.065 U	NA
Cl5 87	0.048 U	0.048 U	0.048 U	NA
Cl5 101	0.056 U	0.056 U	0.056 U	NA
Cl5 105	0.049 U	0.049 U	0.049 U	NA
Cl5 118	0.063 U	0.063 U	0.064 U	NA
Cl6 128	0.103 U	0.103 U	0.105 U	NA
Cl6 138	0.053 U	0.053 U	0.054 U	NA
Cl6 153	0.018 J	0.018 J	0.022 J	12
Cl7 170	0.052 U	0.052 U	0.053 U	NA
Cl7 180	0.055 U	0.055 U	0.056 U	NA
Cl7 183	0.049 U	0.049 U	0.050 U	NA
Cl7 184	0.039 U	0.039 U	0.039 U	NA
Cl7 187	0.051 U	0.051 U	0.052 U	NA
Cl8 195	0.048 U	0.048 U	0.049 U	NA
Cl9 206	0.047 U	0.047 U	0.048 U	NA
Cl10 209	0.051 U	0.051 U	0.052 U	NA
Surrogate Recoveries:				
Cl3(34)	68	65	80	
Cl5(112)	72	68	84	

U - Analyte not detected, ssMDL value inserted.
J - Analyte detected below the ssMDL value.
N - QC value outside the accuracy or precision DQO.

Laboratory Control Sample Data

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Project Name: Ponce Harbor
Project Number: N005561

Client Sample ID:		NA	
Battelle Sample ID:		BB672LCS	
Percent Moisture (%)		NA	
Battelle Batch ID:		03-0231	
Sample Dry Weight (g):		NA	
Sample Wet Weight (g):		NA	
Units:	ng	ng	%
	FW67		Recovery
Aldrin	75.100	55.231	74
a Chlordane	75.375	60.696	81
trans Nonachlor	75.025	60.213	80
Dieldrin	75.075	56.117	75
4,4 DDT	75.100	63.100	84
2,4 DDT	75.250	57.839	77
4,4 DDD	75.075	65.541	87
2,4 DDD	75.025	56.843	76
4,4 DDE	75.050	55.305	74
2,4 DDE	75.250	60.919	81
Endosulfan I	75.075	55.857	74
Endosulfan II	75.075	60.246	80
Endosulfan sulfate	75.075	58.438	78
Heptachlor	75.075	57.829	77
Heptachlor epoxide	75.100	54.542	73
Cl2 08	75.100	56.420	75
Cl3 18	75.100	50.443	67
Cl3 28	75.100	58.668	78
Cl4 44	75.100	60.240	80
Cl4 49	75.100	61.815	82
Cl4 52	75.100	60.789	81
Cl4 66	75.100	63.397	84
Cl5 87	75.100	58.954	79
Cl5 101	75.100	62.467	83
Cl5 105	75.100	60.143	80
Cl5 118	75.100	56.424	75
Cl6 128	75.100	58.049	77
Cl6 138	75.100	61.119	81
Cl6 153	75.100	60.107	80
Cl7 170	75.100	63.064	84
Cl7 180	75.100	63.150	84
Cl7 183	75.100	63.190	84
Cl7 184	75.100	59.111	79
Cl7 187	75.100	59.164	79
Cl8 195	75.100	63.160	84
Cl9 206	75.100	62.577	83
Cl10 209	75.100	65.188	87

Surrogate Recoveries:

Cl3(34)	75
Cl5(112)	76

U - Analyte not detected, ssMDL value inserted.
J - Analyte detected below the ssMDL value.
N - QC value outside the accuracy or precision DQO.

Matrix Spike Data

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Project Name: Ponce Harbor
Project Number: N005561

Client Sample ID:		E-PH03-SC	E-PH03-SC	
Battelle Sample ID:		U8241	U8241MS	
Percent Moisture (%)		32.57	32.57	
Battelle Batch ID:		03-0231	03-0231	
Sample Dry Weight (g):		27.46	13.59	
Sample Wet Weight (g):		40.72	20.16	
Units:	ng FW67	ng/g Dry Wt.	ng/g Dry Wt.	% Recovery
Aldrin	75.100	0.034 U	3.975	72
a Chlordane	75.375	0.039 U	4.487	81
trans Nonachlor	75.025	0.044 U	4.386	79
Dieldrin	75.075	0.052 U	4.259	77
4,4 DDT	75.100	0.050 U	4.622	84
2,4 DDT	75.250	0.068 U	4.940	89
4,4 DDD	75.075	0.044 J	4.863	87
2,4 DDD	75.025	0.058 U	4.155	75
4,4 DDE	75.050	0.022 J	4.091	74
2,4 DDE	75.250	0.072 U	4.751	86
Endosulfan I	75.075	0.055 U	3.874	70
Endosulfan II	75.075	0.054 U	4.367	79
Endosulfan sulfate	75.075	0.056 U	4.441	80
Heptachlor	75.075	0.044 U	4.298	78
Heptachlor epoxide	75.100	0.041 U	4.263	77
Cl2 08	75.100	0.089 U	3.965	72
Cl3 18	75.100	0.042 U	3.602	65
Cl3 28	75.100	0.061 U	4.476	81
Cl4 44	75.100	0.055 U	4.381	79
Cl4 49	75.100	0.064 U	4.654	84
Cl4 52	75.100	0.058 U	4.456	81
Cl4 66	75.100	0.064 U	4.561	83
Cl5 87	75.100	0.048 U	4.511	82
Cl5 101	75.100	0.056 U	4.651	84
Cl5 105	75.100	0.049 U	4.714	85
Cl5 118	75.100	0.063 U	4.237	77
Cl6 128	75.100	0.103 U	4.453	81
Cl6 138	75.100	0.053 U	4.513	82
Cl6 153	75.100	0.018 J	4.370	79
Cl7 170	75.100	0.052 U	4.678	85
Cl7 180	75.100	0.055 U	4.561	83
Cl7 183	75.100	0.049 U	4.605	83
Cl7 184	75.100	0.039 U	4.288	78
Cl7 187	75.100	0.051 U	4.339	79
Cl8 195	75.100	0.048 U	4.641	84
Cl9 206	75.100	0.047 U	4.444	80
Cl10 209	75.100	0.051 U	4.644	84

Surrogate Recoveries:

Cl3(34)	68	77
Cl5(112)	72	75

U - Analyte not detected, ssMDL value inserted.
J - Analyte detected below the ssMDL value.
N - QC value outside the accuracy or precision DQO.

Standard Reference Material Data
Surrogate Corrected

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Project Name: Ponce Harbor
Project Number: N005561

Client Sample ID:	SRM 1944			
Battelle Sample ID:	BB673SRM			
Battelle Batch ID:	03-0231	Values	Values	
Sample Dry Weight (g):	1.03	Lower Range	Upper Range	
Sample Wet Weight (g):	1.04			
Units:	ng/g Dry Wt.	ng/g Dry Wt.	ng/g Dry Wt.	% PD
a Chlordane	21.480	15.680	17.340	24
trans Nonachlor	14.472	7.690	8.710	66 N
4,4 DDT	199.025	108.000	130.000	53 N
Cl2 08	31.189	20.000	24.600	27
Cl3 18	56.083	48.400	53.600	5
Cl3 28	95.995	78.100	83.500	15
Cl4 44	78.603	58.200	62.200	26
Cl4 49	60.408	51.300	54.700	10
Cl4 52	91.090	77.400	81.400	12
Cl4 66	72.165	67.600	76.200	5
Cl5 87	27.145	25.600	34.200	6
Cl5 101	98.939	70.900	75.900	30
Cl5 105	26.665	23.400	25.600	4
Cl5 118	66.544	53.700	62.300	7
Cl6 128	11.050	8.190	8.750	26
Cl6 138	71.636	59.100	65.100	10
Cl6 153	88.644	71.100	76.900	15
Cl7 170	22.336	21.200	24.000	5
Cl7 180	50.306	43.100	45.500	10
Cl7 183	12.688	11.620	12.760	1
Cl7 187	28.477	24.100	26.100	9
Cl8 195	3.574	3.360	4.140	14
Cl9 206	10.353	8.700	9.720	7
Cl10 209	8.622	6.480	7.140	21

Surrogate Recoveries:

Cl3(34)	93
Cl5(112)	84

U - Analyte not detected, ssMDL value inserted.
J - Analyte detected below the ssMDL value.
N - QC value outside the accuracy or precision DQO.
Percent difference calculated vs closest range lower or higher.

Sediment PAH Data

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PAH – SEDIMENT QA/QC SUMMARY
Batch 03-0231

PROJECT: Ponce Harbor
PARAMETER: PAH
LABORATORY: Battelle, Duxbury, MA
MATRIX: Sediment
SAMPLE CUSTODY: The sediment sample composite was received at Battelle Duxbury on March 11, 2003. Sediment was received in good condition. The cooler temperature on arrival was –0.9 °C. Sediment was stored at 4 °C until processing began.

QA/QC DATA QUALITY OBJECTIVES:

	Reference Method	Blank Criteria	Surrogate Recovery	LCS/MS Recovery	SRM % Diff.	Sample Replicate Relative Precision	Achieved Detection Limit (ng/g DW)
PAH	General NS&T	B<3xMDL	30-120% Recovery	50-120% Recovery (for at least 80% of analytes; analyte conc. in MS must be >10x background)	≤25% PD (on average; not to exceed 35% for more than 30% of analytes; measured from range of certified values, for analytes >10x MDL)	≤30% RSD	~ 0.03 – 0.1 Target DL: 10

METHOD: Sediment samples were extracted for PAH following general NS&T methods. Approximately 30 g of sediment was spiked with surrogates and extracted three times with dichloromethane using shaker table techniques. The combined extract was dried over anhydrous sodium sulfate, concentrated, processed through alumina cleanup column, concentrated, and further purified by GPC/HPLC. The post-HPLC extract was concentrated, fortified with recovery internal standards and split for the required analyses. Extracts intended for PAH analysis were analyzed using gas chromatography/mass spectrometry (GC.MS) with the MS operating in the selected ion monitoring (SIM) mode, following general NS&T methods. Sample data were quantified by the method of internal standards, using the Recovery Internal Standard (RIS) compounds.

HOLDING TIMES: Samples were prepared for analysis in one analytical batch and were extracted within 14 days of sample collection and analyzed within 40 days of extraction.

Batch	Extraction Date	Analysis Date
03-0231	03/12/2003	03/18/2003 – 03/19/2003

PAH – SEDIMENT QA/QC SUMMARY
Batch 03-0231

BLANKS:

A procedural blank (PB) was prepared with each analytical batch. Blanks were analyzed to ensure the sample extraction and analysis methods were free of contamination.

03-0231 – Low levels of naphthalene, fluorene, phenanthrene, fluoranthene, and pyrene were detected in the blank.

Comments – All analytes detected were at concentrations < MDL – all are "J" qualified. The concentrations detected were between 0.02 and 0.31 ng/g dry weight – the detection limit goals according to the RTM are 4 ng/g. Therefore, the concentrations in the blank are between a factor of ~10 and 200 times lower than the detection limit requirements of the project.

LABORATORY CONTROL SAMPLE (Blank Spike)

A laboratory control sample (LCS) was prepared with each analytical batch. The percent recoveries of target PAH were calculated to measure data quality in terms of accuracy.

03-0231 – All target analytes were recovered within the laboratory control limits.

Comments – None.

MATRIX SPIKE:

A matrix spike (MS) sample was prepared with each analytical batch. The percent recoveries of target analytes were calculated to measure data quality in terms of accuracy.

03-0231 – Percent recoveries for all target analytes were within data quality criteria.

Comments – None.

SURROGATES:

Three surrogate compounds were added prior to extraction, including naphthalene-d8, phenanthrene-d10, and chrysene-d12. The recovery of each surrogate compound was calculated to measure data quality in terms of accuracy (extraction efficiency).

03-0231 – Percent recoveries for all surrogate compounds were within the laboratory control limits.

Comments – None.

REPLICATES:

Replicate (triplicate) samples for sediments were prepared with each analytical batch. The RSD between triplicate analyses for PAH is calculated to measure data quality in terms of precision.

03-0231 – The RSD between triplicate analyses of several PAH were outside the DQO of <30% RSD.

Comments – The PAH concentrations in these samples are very low, ranging from ~0.2 to 3 ng/g dry weight. The concentrations in the third replicate were higher than the other two replicates. The RSDs are elevated, however this is related to the low concentrations in the sample, not laboratory precision.

PAH – SEDIMENT QA/QC SUMMARY
Batch 03-0231

SRM: A standard reference material (SRM, NIST 1944) was prepared with each analytical batch. The percent difference (PD) between the measured value and the certified range was calculated to measure data quality in terms of accuracy.

03-0231 – SRM PDs were within the control limits for all certified target analytes.

Comments – None.

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Project Name: Ponce Harbor
Project Number: N005561

Procedural Blank Data
Dry Weight
Sediment

Client Sample ID:	Procedural Blank
Battelle Sample ID:	BB671PB
Client Description:	NA
Battelle Batch ID:	03-0231
% Moisture:	43.74
Sample Dry Weight (g):	23
Sample Wet Weight (g):	41
Unit	ng/g
Naphthalene	0.31 J
Biphenyl	0.04 U
Acenaphthylene	0.07 U
Acenaphthene	0.04 U
Fluorene	0.06 U
Phenanthrene	0.06 J
Anthracene	0.06 U
Fluoranthene	0.04 J
Pyrene	0.02 J
Benzo(a)anthracene	0.04 U
Chrysene	0.03 U
Benzo(b)fluoranthene	0.05 U
Benzo(k)fluoranthene	0.03 U
Benzo(a)pyrene	0.06 U
Indeno(1,2,3-c,d)pyrene	0.03 U
Dibenz(a,h)anthracene	0.05 U
Benzo(g,h,i)perylene	0.03 U
Surrogate Recoveries (%)	
Naphthalene-d8	73
Phenanthrene-d10	69
Chrysene-d12	77

U = Analyte undetected MDL Reported
J = Detected, but below the MDL
B = Analyte detected in Blank at level >3*MDL
concentration <3* the level in the blank
NA = Not applicable

Prepared by A CRAM,
5/16/2003

Project Name: Ponce Harbor
Project Number: N005561

Sediment Sample Data
Dry Weight

Client Sample ID:	E-PH03-SC		RS-PH03
Battelle Sample ID:	U8241		U8240
Client Description:	Silty Clay Composite (1-9)		Ref Sample Composite (Harbor Ref)
Battelle Batch ID:	03-0231		03-0231
% Moisture:	32.57		54.91
Sample Dry Weight (g):	27.46		18.35
Sample Wet Weight (g):	40.72		40.7
Unit	ng/g		ng/g
Naphthalene	1.60	J	5.63
Biphenyl	0.36		0.72
Acenaphthylene	0.22		0.86
Acenaphthene	0.17		0.38
Fluorene	0.24		0.38
Phenanthrene	1.25		4.81
Anthracene	0.29		0.87
Fluoranthene	1.57		8.35
Pyrene	1.78		9.22
Benzo(a)anthracene	0.78		4.84
Chrysene	1.08		5.44
Benzo(b)fluoranthene	1.31		5.86
Benzo(k)fluoranthene	0.99		5.08
Benzo(a)pyrene	1.05		5.75
Indeno(1,2,3-c,d)pyrene	0.64		4.12
Dibenz(a,h)anthracene	0.15		0.79
Benzo(g,h,i)perylene	0.83		4.29
Surrogate Recoveries (%)			
Naphthalene-d8	64		57
Phenanthrene-d10	65		64
Chrysene-d12	80		77

U = Analyte undetected MDL Reported
J = Detected, but below the MDL
B = Analyte found in the sample at a concentration <3* the level in the blank
NA = Not applicable
& = QC value outside the accuracy or precision

Prepared by A CRAM,
5/16/2003

Project Name: Ponce Harbor
Project Number: N005561

Sediment Sample Triplicate Data
Dry Weight

Client Sample ID:	E-PH03-SC	E-PH03-SC	E-PH03-SC	
Battelle Sample ID:	U8241	U8241DUP	U8241TRP	
Client Description:	Silty Clay Composite (1-9)	Silty Clay Composite (1-9)	Silty Clay Composite (1-9)	
Battelle Batch ID:	03-0231	03-0231	03-0231	
% Moisture:	32.57	32.57	3257	
Sample Dry Weight (g):	27.46	27.46	27.02	
Sample Wet Weight (g):	40.72	40.72	40.07	
Unit	ng/g	ng/g	ng/g	RSD
				%
Naphthalene	1.60 J	1.38 J	2.14 J	22.7
Biphenyl	0.36	0.34	0.46	16.6
Acenaphthylene	0.22	0.19	0.54	61.6 &
Acenaphthene	0.17	0.15	0.20	14.6
Fluorene	0.24	0.22	0.27	10.8
Phenanthrene	1.25	1.14	1.40	10.2
Anthracene	0.29	0.28	0.40	19.4
Fluoranthene	1.57	1.48	1.89	13.0
Pyrene	1.78	1.66	2.85	31.4 &
Benzo(a)anthracene	0.78	0.62	1.39	44.0 &
Chrysene	1.08	0.90	2.29	53.2 &
Benzo(b)fluoranthene	1.31	1.03	2.24	41.8 &
Benzo(k)fluoranthene	0.99	0.80	2.37	61.9 &
Benzo(a)pyrene	1.05	0.77	2.72	69.7 &
Indeno(1,2,3-c,d)pyrene	0.64	0.47	1.37	57.6 &
Dibenz(a,h)anthracene	0.15	0.12	0.27	44.2 &
Benzo(g,h,i)perylene	0.83	0.71	1.57	44.6 &
Surrogate Recoveries (%)				
Naphthalene-d8	64	54	75	
Phenanthrene-d10	65	66	80	
Chrysene-d12	80	67	103	

U = Analyte undetected MDL Reported
J = Detected, but below the MDL
B = Analyte found in the sample at a concentration <3* the level in the blank
NA = Not applicable
& = QC value outside the accuracy or precision

Prepared by A CRAM,
5/16/2003

Project Name: Ponce Harbor
Project Number: N005561

Laboratory Control Spike Data
Sediment

Client Sample ID:		LCS	
Battelle Sample ID:		BB672LCS	
Client Description:		NA	
Battelle Batch ID:		03-0231	
% Moisture:		NA	
Sample Dry Weight (g):		NA	
Sample Wet Weight (g):	FW67	NA	Percent Recovery
Unit	ng	ng	%
Naphthalene	2501.38	1796.52	72
Biphenyl	2507.50	1806.66	72
Acenaphthylene	2501.00	1830.34	73
Acenaphthene	2501.00	1829.88	73
Fluorene	2501.25	1947.47	78
Phenanthrene	2501.13	1991.94	80
Anthracene	2501.00	1942.57	78
Fluoranthene	2501.00	2124.00	85
Pyrene	2501.00	2111.33	84
Benzo(a)anthracene	2501.38	2186.65	87
Chrysene	2501.25	2311.43	92
Benzo(b)fluoranthene	2501.25	2273.92	91
Benzo(k)fluoranthene	2500.88	2267.04	91
Benzo(a)pyrene	2501.38	2109.11	84
Indeno(1,2,3-c,d)pyrene	2501.50	1880.55	75
Dibenz(a,h)anthracene	2501.00	2023.95	81
Benzo(g,h,i)perylene	2501.25	1846.23	74

Surrogate Recoveries (%)

Naphthalene-d8	71
Phenanthrene-d10	69
Chrysene-d12	85

U = Analyte undetected MDL Reported
J = Detected, but below the MDL
B = Analyte found in the sample at a concentration <3* the level in the blank
NA = Not applicable
& = QC value outside the accuracy or precision

Prepared by A CRAM,
5/16/2003

Project Name: Ponce Harbor
Project Number: N005561

Sediment Matrix Spike Data
Dry Weight

Client Sample ID:		E-PH03-SC	E-PH03-SC	
Battelle Sample ID:		U8241	U8241MS	
Client Description:		Silty Clay Composite (1-9)	Silty Clay Composite (1-9)	
Battelle Batch ID:		03-0231	03-0231	
% Moisture:		32.57	32.57	
Sample Dry Weight (g):		27.46	13.59	
Sample Wet Weight (g):	FW67	40.72	20.16	
Unit	ng	ng/g	ng/g	% Recovery
Naphthalene	2501.38	1.60 J	110.15	59
Biphenyl	2507.50	0.36	113.40	61
Acenaphthylene	2501.00	0.22	112.84	61
Acenaphthene	2501.00	0.17	117.34	64
Fluorene	2501.25	0.24	130.02	71
Phenanthrene	2501.13	1.25	147.73	80
Anthracene	2501.00	0.29	138.79	75
Fluoranthene	2501.00	1.57	164.91	89
Pyrene	2501.00	1.78	164.36	88
Benzo(a)anthracene	2501.38	0.78	158.77	86
Chrysene	2501.25	1.08	171.37	93
Benzo(b)fluoranthene	2501.25	1.31	158.35	85
Benzo(k)fluoranthene	2500.88	0.99	162.02	88
Benzo(a)pyrene	2501.38	1.05	151.99	82
Indeno(1,2,3-c,d)pyrene	2501.50	0.64	126.45	68
Dibenz(a,h)anthracene	2501.00	0.15	135.02	73
Benzo(g,h,i)perylene	2501.25	0.83	128.16	69
Surrogate Recoveries (%)				
Naphthalene-d8		64	58	
Phenanthrene-d10		65	68	
Chrysene-d12		80	84	

U = Analyte undetected MDL Reported
J = Detected, but below the MDL
B = Analyte found in the sample at a concentration <3* the level in the blank
NA = Not applicable

& = QC value outside the accuracy or precision

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5/16/2003

Project Name: Ponce Harbor
Project Number: N005561

Standard Reference Material Data
Surrogate Corrected
Dry Weight
Sediment

Client Sample ID:				SRM1944	
Battelle Sample ID:				BB673SRM	
Client Description:				NA	
Battelle Batch ID:				03-0231	
% Moisture:				98.75	
Sample Dry Weight (g):				1.03	
Sample Wet Weight (g):				1.04	Percent Difference
Unit	SRM 1944 Cert Val	+/- Cert Range		ng/g	%
Naphthalene	1650	310		1399.30	4.4
Phenanthrene	5270	220		5829.19	6.2
Anthracene	1770	330		1161.15	19.4
Fluoranthene	8920	320		10012.73	8.4
Pyrene	9700	420		10441.45	3.2
Benzo(a)anthracene	4720	110		4564.64	1.0
Chrysene	5900	370	A	5956.00	5.0
Benzo(b)fluoranthene	3870	420		3559.98	3.2
Benzo(k)fluoranthene	4390	640	B	3424.92	8.7
Benzo(a)pyrene	4300	130		3568.05	14.4
Indeno(1,2,3-c,d)pyrene	2780	100		2625.67	2.0
Benzo(g,h,i)perylene	2840	100		2620.76	4.4

Surrogate Recoveries (%)

Naphthalene-d8	69
Phenanthrene-d10	84
Chrysene-d12	89

U = Analyte undetected MDL Reported
J = Detected, but below the MDL
B = Analyte found in the sample at a concentration <3* the level in the blank
NA = Not applicable

& = QC value outside the accuracy or precision
A =Combined certified concentrations of Chysene (4860 +/- 100) and Triphenylene (1040+/- 270).
B =Combined concentrations of Benzo(k)fluoranthene (2300+/- 200ug/kg) and and Benzo(j)fluoranthene (2090 +/-440 ug/kg)

Project Name: Ponce Harbor
Project Number: N005561

Independent Check Sample Data

Client Sample ID:		ICC	
Battelle Sample ID:		SQB964\B3253	
Battelle Batch ID:		03-0231	
% Moisture:		NA	
Sample Dry Weight (g):		NA	
Sample Wet Weight (g):	FZ47	NA	PD
Unit	ng/ul	ng/ul	%
Naphthalene	1.002	0.97	3.2
Biphenyl	1.001	0.96	4.1
Acenaphthylene	1.000	1.03	3.0
Acenaphthene	1.001	1.01	0.9
Fluorene	1.000	1.07	7.0
Phenanthrene	1.008	1.06	5.2
Anthracene	1.001	1.11	10.9
Fluoranthene	1.024	1.08	5.5
Pyrene	1.001	1.02	1.9
Benzo(a)anthracene	1.002	0.94	6.2
Chrysene	1.004	0.99	1.4
Benzo(b)fluoranthene	1.002	0.92	8.2
Benzo(k)fluoranthene	1.002	0.93	7.1
Benzo(a)pyrene	1.002	0.91	9.1
Indeno(1,2,3-c,d)pyrene	1.001	0.88	12.1
Dibenz(a,h)anthracene	1.001	0.9	10.0
Benzo(g,h,i)perylene	1.002	0.89	11.2
Surrogate Recoveries (%)			
Naphthalene-d8		94	
Phenanthrene-d10		93	
Chrysene-d12		92	

U = Analyte undetected MDL Reported
J = Detected, but below the MDL
B = Analyte found in the sample at a concentration <3* the level in the blank
NA = Not applicable
& = QC value outside the accuracy or precision

Prepared by A CRAM,
5/16/2003

Sediment Physical Parameters Data

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Ponce Harbor/ODMDS Data Summary Table

Client: Battelle
Client Project #: N005561
Project Name: Ponce Harbor/ODMDS

Client Sample ID	Battelle Sample ID	AMS Sample ID	Gravel >2.00 mm (%)	Sand 2.00-0.0625 mm (%)	Silt 0.0625-0.0039 mm (%)	Clay <0.0039 mm (%)	TOC (dry weight) (%)	Percent Moisture (%)	Wet Bulk Density (g/cm3)	Dry Bulk Density (g/cm3)	Specific Gravity	Plastic Limit	Liquid Limit	Plasticity Index
RS-PH03-1A	U8094	18342	0.00	3.49	40.76	55.75	0.91	60	-	-	-	-	-	-
RS-PH03-1B	U8095	18343	0.00	3.80	41.72	54.48	0.88	61	-	-	-	-	-	-
RS-PH03-1C	U8096	18099	0.12	5.32	41.09	53.47	0.96	61	-	-	-	-	-	-
RS-PH03-2A	U8097	18100	0.00	3.83	41.90	54.27	0.98	60	-	-	-	-	-	-
RS-PH03-2B	U8098	18101	0.13	4.35	41.72	53.79	0.99	61	-	-	-	-	-	-
RS-PH03-2C	U8099	18102	0.25	5.54	40.11	54.11	0.95	60	-	-	-	-	-	-
E-PODS03-1A	U8100	18103	0.13	2.25	48.51	49.11	0.98	59	-	-	-	-	-	-
E-PODS03-1B	U8101	18104	0.00	2.23	48.19	49.58	1.04	59	-	-	-	-	-	-
E-PODS03-1C	U8102	18105	0.00	2.49	48.34	49.17	1.03	60	-	-	-	-	-	-
E-PODS03-2	U8103	18106	0.00	17.74	56.68	25.58	0.59	43	-	-	-	-	-	-
E-PODS03-3A	U8104	18107	0.00	2.88	48.38	48.74	1.08	61	-	-	-	-	-	-
E-PODS03-3B	U8105	18108	0.00	2.85	49.20	47.95	1.06	60	-	-	-	-	-	-
E-PODS03-3C	U8106	18109	0.00	2.96	48.76	48.28	1.08	56	-	-	-	-	-	-
E-PODS03-4A	U8107	18110	0.00	3.21	50.33	46.47	1.03	54	-	-	-	-	-	-
E-PODS03-4B	U8108	18111	0.00	4.03	49.14	46.82	0.97	56	-	-	-	-	-	-
E-PODS03-4C	U8109	18112	0.00	3.23	51.23	45.54	0.97	56	-	-	-	-	-	-
E-PODS03-5	U8110	18113	0.00	6.56	55.96	37.48	0.87	50	-	-	-	-	-	-
E-PODS03-6	U8111	18114	0.00	2.65	48.31	49.04	1.08	59	-	-	-	-	-	-
E-PODS03-7A	U8112	18115	0.00	2.66	50.48	46.86	0.98	59	-	-	-	-	-	-
E-PODS03-8A	U8113	18116	0.00	2.35	48.72	48.93	1.15	58	-	-	-	-	-	-
E-PODS03-8B	U8114	18117	0.09	3.68	47.41	48.82	1.13	59	-	-	-	-	-	-
E-PODS03-8C	U8115	18118	0.00	2.96	47.79	49.25	1.17	59	-	-	-	-	-	-
E-PODS03-7B	U8116	18119	0.00	2.56	49.92	47.53	1.02	55	-	-	-	-	-	-
E-PODS03-7C	U8117	18120	0.00	2.55	48.96	48.49	0.98	57	-	-	-	-	-	-
E-PODS03-9	U8118	18121	0.00	2.76	49.59	47.65	1.08	57	-	-	-	-	-	-
E-PODS03-10	U8119	18122	0.21	3.08	49.21	47.50	1.11	57	-	-	-	-	-	-
E-PODS03-11	U8120	18123	0.00	2.58	49.10	48.32	1.10	60	-	-	-	-	-	-
E-PODS03-12	U8121	18124	0.01	2.95	49.52	47.51	1.08	59	-	-	-	-	-	-
E-PODS03-13	U8122	18125	0.06	3.24	47.85	48.85	1.09	59	-	-	-	-	-	-
E-PODS03-14	U8123	18126	0.00	2.81	49.98	47.22	1.09	58	-	-	-	-	-	-
E-PODS03-15	U8124	18127	0.00	3.02	50.65	46.34	1.03	55	-	-	-	-	-	-
E-PODS03-16	U8125	18128	0.00	3.55	50.56	45.90	1.11	58	-	-	-	-	-	-
E-PODS03-17	U8126	18129	0.83	29.92	40.32	28.93	0.70	47	-	-	-	-	-	-
E-PODS03-18	U8127	18130	0.05	2.92	48.59	48.44	1.10	59	-	-	-	-	-	-
RS-PH03	U8240	18131	0.10	4.88	39.67	55.35	0.91	54	1.42	0.65	2.74	77	30	47
E-PH03-SC	U8241	18132	0.34	9.30	62.24	28.12	0.69	34	1.82	1.21	2.75	46	27	19
E-PH03-CONTROL	U8242	18133	15.51	73.56	3.40	7.52	0.64	30	-	-	-	-	-	-
E-PH03-1SC	U8243	18134	0.74	7.50	57.66	34.10	0.78	34	-	-	-	-	-	-
E-PH03-2SC	U8244	18135	0.08	8.39	57.34	34.20	0.73	32	-	-	-	-	-	-
E-PH03-3SC	U8245	18136	0.10	13.42	63.37	23.11	0.66	31	-	-	-	-	-	-
E-PH03-4SC	U8246	18137	0.03	4.69	64.09	31.19	0.69	32	-	-	-	-	-	-
E-PH03-5SC	U8247	18138	0.55	11.27	64.81	23.37	0.69	30	-	-	-	-	-	-
E-PH03-6SC	U8248	18139	0.13	5.86	65.11	28.89	0.81	34	-	-	-	-	-	-
E-PH03-7SC	U8249	18140	0.00	12.02	64.33	23.64	0.64	33	-	-	-	-	-	-
E-PH03-8SC	U8250	18141	0.04	12.08	65.53	22.35	0.63	31	-	-	-	-	-	-
E-PH03-9SC	U8251	18142	0.30	11.14	58.99	29.58	0.63	38	-	-	-	-	-	-
N/A	U8094	18143	0.01	3.59	39.54	56.86	0.96	60	-	-	-	-	-	-
N/A	U8095	18144	0.00	4.38	40.14	55.48	0.99	62	-	-	-	-	-	-

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Final Data Report

Ponce Harbor Tissue Data

Prepared for:

ppb Environmental Laboratories, Inc.

Prepared by:

Battelle
397 Washington Street
Duxbury, Massachusetts 02332

August 21, 2003

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Ponce, Puerto Rico Tissue Sample Identification Key

Sample ID	Replicate	Sample Description	Battelle ID
RS-PH03	A	*Nereis virens*	T2707
RS-PH03	B	*Nereis virens*	T2708
RS-PH03	C	*Nereis virens*	T2709
RS-PH03	D	*Nereis virens*	T2710
RS-PH03	E	*Nereis virens*	T2711
E-PH03-SC	A	*Nereis virens*	T2712
E-PH03-SC	B	*Nereis virens*	T2713[1]
E-PH03-SC	C	*Nereis virens*	T2714
E-PH03-SC	D	*Nereis virens*	T2715
E-PH03-SC	COMPOSITE	*Nereis virens* Composite made from replicates E – I	T4581[2]
PRETEST N. VIRENS	A	*Nereis virens*	T2721
PRETEST N. VIRENS	B	*Nereis virens*	T2722
PRETEST N.VIRENS	C	*Nereis virens*	T2723
NA	ZIPLOCK BAG SWAB 1	To ensure sampling containers free of PAH contamination	T4578
NA	ZIPLOCK BAG SWAB 2	To ensure sampling containers free of PAH contamination	T4579
NA	BLANK SWAB	To ensure swabs are free of PAH contamination	T4583
RS-PH03	A	*Macoma nasuta*	T2729
RS-PH03	B	*Macoma nasuta*	T2730
RS-PH03	C	*Macoma nasuta*	T2731
RS-PH03	D	*Macoma nasuta*	T2732
RS-PH03	E	*Macoma nasuta*	T2733
E-PH03-SC	A	*Macoma nasuta*	T2734
E-PH03-SC	B	*Macoma nasuta*	T2735[1]
E-PH03-SC	C	*Macoma nasuta*	T2736
E-PH03-SC	D	*Macoma nasuta*	T2737
E-PH03-SC	COMPOSITE	*Macoma nasuta* Composite made from replicates (E – I)	T4582[2]
PRETEST-M.NASUTA	A	*Macoma nasuta*	T2743
PRETEST-M.NASUTA	B	*Macoma nasuta*	T2744
PRETEST-M.NASUTA	C	*Macoma nasuta*	T2745

[1] Matrix Spike made from this sample.

[2] Triplicate analyses done on this sample.

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ShpNo SHP-030530-02

Battelle Project No: N005561

Sample Receipt Form

Approved: ☑ ☑

Project Number:

Client: PPB Environmental

Received by: Smith, Joe

Date/Time Received: Friday, May 30, 2003 12:00 AM

No. of Shipping Containers: 1

SHIPMENT

Method of Delivery: Commercial Carrier

Tracking Number: 628943497971

COC Forms: ☑ **Shipped with samples** ☐ **No Forms**

Cooler(s)/Box(es)

Container	Type	Sealed With	Seal Condition	Container Condition	Temp C	Total Samples
1 of 1	Cooler	Tape	Intact	Intact	2.7	45

Samples

Sample Labels:
☐ Sample labels agree with COC forms
☑ Discrepancies (see Sample Custody Corrective Action Form)

Container Seals:
☐ Tape ☐ Custody Seals ☐ Other Seals (See sample Log)
☑ Seals intact for each shipping container
☐ Seals broken (See sample log for impacted samples)

Condition of Samples:
☑ Sample containers intact
☐ Sample containers broken/leaking (See Custody Corrective Action Form)

Temperature upon receipt (°C): -2.7 Temperature Blank used ☐ Yes ☑ No
(Note: If temperature upon receipt differs from required conditions, see sample log comment field)

Samples Acidified: ☐ Yes ☑ No ☑ Unknown

Initial pH 5-9?: ☐ Yes ☐ No ☑ NA
If no, individual sample adjustments on the Auxiliary Sample Receipt Form

Total Residual Chlorine Present?: ☐ Yes ☐ No ☑ NA
If yes, individual sample adjustments on the Auxiliary Sample Receipt Form

Head Space <1% in samples for water VOC analysis: ☐ Yes ☐ No ☑ NA
Individual sample deviations noted on sample log

Samples Containers:
Samples returned in PC-grade jars: ☐ Yes ☐ No ☑ Unknown /Lot No.: UnKnown

Storage Location: Walk-in Freezer

BDO IDs Assigned: T2702 - T4583

Samples logged in by: Smith, Joe

Date/Time: 05/30/2003 10:50 AM

Approved By: Fahey, Jessica

Approved On: 6/4/2003 2:48:0

Authorized By: Peven-McCarthy, Carole

Authorized On: 6/10/2003 9:51:00

MACTEC Engineering and Consulting, Inc. (formerly Harding ESE)
Toxicology Laboratory
Gainesville, Florida

QA Form Number: 063COCt
Revised: Jan2003

SAMPLE CHAIN OF CUSTODY for TISSUE SAMPLES

Facility: MACTEC Engineering and Consulting, Inc.

Location: 404 SW 140th Ter., Newberry, FL 32669

Other: Phone (352) 332-3318

SAMPLE IDENTIFICATION

Sample Identification	Organism Genus species	#	Replicate Bags	Observations
Ctrl - PHØ3	N. virens	5	A - E	
RFS - PHØ3	N. virens	5	A - E	
E - PHØ3 - SC	N. virens	9	A - I	
Pretest N. virens	N. virens	3	3 bags	
Ctrl - PHØ3	M. nasuta	5	A - E	
RFS - PHØ3	M. nasuta	5	A - E	
E - PHØ3 - SC	M. nasuta	9	A - I	
Pretest M. nasuta	M. nasuta	3	3 bags	

Total Number of Samples: ~~42~~ 44 individual bags

SAMPLE INFORMATION

Toxicity Test Type: Bioaccumulation test

Exposed to Sediment from: Ponce Harbor, PR

Observations/Remarks: Replicate bags packed inside larger ziploc bags

CHAIN OF CUSTODY

Relinquished By:	Received By:	Date	Time
Gary M. Raya	Jessica M. Joy 5/30/03 10:30AM	1500	~~5/28/03~~ 5/29/03 JMRaya

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ShpNo: SHP-030530-02

Battelle Project No: N005561

Report Corrective Actions

Approved:

COC Client: MACTEC ENGINEERING AND CONSULTING

COC Project: PONCE HARBOR

COC Date: 5/30/2003 10:50:

	Description of Problem:	Explanation:
Client Id	Jars and C-O-C do not have matching Ids	For the clams (M. nasuta) COC ID: RFS-PH03 (A-E) SAMPLE ID: PHO3-RS-1 (A-E)
	Other	On the COC the Pretest samples for both species were not given individual letters of identification. We have added consective letters to the ID's to identify the individual samples.

Documentation of project manager notification

Sample Custodian Fahey, Jessica **Date:** 5/30/2003 2:37:00 PM

Laboratory Manager: [signature] **Date:** 6·2·03

Project Manager: [signature] **Date:** 6/2/2003

Documentation of client notification (should be completed by project manager within 24 hrs):

On 5/30/2003 **I contacted** Ann Shostell **at** Mactec (bio lab for ppb)

Results of communication with client (Describe any corrective action directed by the client):

See email Attached.
E-PH03-SC-A, -B, -C...
RS-PH03-A, -B, ...

Date this form was received back to the custodian:

Reference Number:

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ShpNo: SHP-030530-02

Battelle Project No: N005561

Report Corrective Actions

Approved:

COC Client: PPB Environmental

COC Project: PONCE HARBOR

COC Date: 5/30/2003 10:50:

	Description of Problem:	Explanation:
Client Id	Other	The COC did not have a Collection Date listed. Per PM's instructions the Collection Date was added to the LIMS system.

Documentation of project manager notification

Sample Custodian Fahey, Jessica **Date:** 7/2/2003 8:37:00 AM

Laboratory Manager: [signature] **Date:** 7-11-03

Project Manager: [signature] **Date:** 7/3/2003

Documentation of client notification (should be completed by project manager within 24 hrs):

On _____ **I contacted** _____ **at**

Results of communication with client (Describe any corrective action directed by the client):

Used date of shipment to satisfy LIMS - no collection date applicable for these tissues. CJM 7/3/2003

Date this form was received back to the custodian: 7/11/03

Reference Number:

Peven, Carole-Sue

From: Shortelle, Ann [ABSHORTELLE@mactec.com]
Sent: Friday, May 30, 2003 5:04 PM
To: Peven, Carole-Sue
Cc: Barrows, Elisabeth S
Subject: RE: Ponce tissues

The station ID's should be used as the unique identifier for the Ponce samples. Ex. E-PH03-SC, RS-PH03, E-PHO3-CONTROL

The above is from PPB's email clarifying sample nomenclature. Unfortunately, we'd started w/ something similar, but not exact. As you note below...they are the same thing. Glad everything arrived okay.
Ann

Ann B. Shortelle, Ph.D.
Chief Scientist
MACTEC Engineering and Consulting, Inc.
(f/k/a Harding ESE, Inc.)
Phone: 352-332-3318
Fax: 352-333-6622
Email: abshortelle@mactec.com

-----Original Message-----
From: Peven, Carole-Sue [mailto:peven@BATTELLE.ORG]
Sent: Friday, May 30, 2003 4:59 PM
To: Shortelle, Ann
Cc: Barrows, Elisabeth S
Subject: Ponce tissues

Hi Ann --

We received the tissues today -- all were still frozen. I have a quick question about the clam ID's. On the labels, the ID's for the reference site tissues (I think that's what they are anyway) are PH03-RS-1 A through
E. On the custody forms the ID's are RFS-PH03 A through E.

Please let us know which they are so we can report them as you expect.

Thanks!

Enjoy the weekend,

Carole

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Chain of Custody

Proj. No	Proj. Name
	Ponce Harbor

SAMPLERS: Signature

ANALYSIS REQUESTED → "NUMBER OF CONTAINERS"

DATE	TIME	BATTELLE ID	CLIENT ID	SAMPLE DESCRIPTION	PEST	PCB	TPH FINGERPRINT	PAH	VOA	TBT	METALS	OTHER	ACIDIFIED	PRESERVED	Total Number of Containers
		T2719	E-PH03-SC-H	N. Viens (worms)											1
		T2720	E-PH03-SC-I												1
		T2721	Pre-Test N.Viens-A												1
		T2722	Pre-Test N.Viens-B												1
		T2723	Pre-Test N.Viens-C												1
		T2724	CTRL-PH03-A	M nasuta (clams)											1
		T2725	CTRL-PH03-B												1
		T2726	CTRL-PH03-C												1
		T2727	CTRL-PH03-D												1
		T2728	CTRL-PH03-E												1
		T2729	RFS-PH03-A												1
		T2730	RFS-PH03-B												1
		T2731	RFS-PH03-C												1
		T2732	RFS-PH03-D												1
		T2733	RFS-PH03-E												1
		T2734	E-PH03-SC-A												1
		T2735	E-PH03-SC-B												1

Relinquished by:	Date/Time	Received by:	Date/Time
		See Original COC	

Relinquished by:	Date/Time	Received by:	Date/Time

Comments:

ORIGINAL

007

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Chain of Custody

Proj. No	Proj. Name
	Ponce Harbor

SAMPLERS: Signature

ANALYSIS REQUESTED → "NUMBER OF CONTAINERS"

DATE	TIME	BATTELLE ID	CLIENT ID	SAMPLE DESCRIPTION	PEST	PCB	TPH FINGERPRINT	PAH	VOA	TBT	METALS	OTHER	ACIDIFIED	PRESERVED	Total Number of Containers
		T2702	CTRL-PH03-A	N. VIRENS (worms)											1
		T2703	CTRL-PH03-B	N. VIRENS											1
		T2704	CTRL-PH03-C												1
		T2705	CTRL-PH03-D												1
		T2706	CTRL-PH03-E												1
		T2707	RFS-PH03-A												1
		T2708	RFS-PH03-B												1
		T2709	RFS-PH03-C												1
		T2710	RFS-PH03-D												1
		T2711	RFS-PH03-E												1
		T2712	E-PH03-SC-A												1
		T2713	E-PH03-SC-B												1
		T2714	E-PH03-SC-C												1
		T2715	E-PH03-SC-D												1
		T2716	E-PH03-SC-E												1
		T2717	E-PH03-SC-F												1
		T2718	E-PH03-SC-G												1

Relinquished by:	Date/Time	Received by:	Date/Time
		See Original COC	

Relinquished by:	Date/Time	Received by:	Date/Time

Comments:

0080

ORIGINAL

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Chain of Custody

Proj. No	Proj. Name
	Ponce Harbor

SAMPLERS: Signature

ANALYSIS REQUESTED → "NUMBER OF CONTAINERS"

DATE	TIME	BATTELLE ID	CLIENT ID	SAMPLE DESCRIPTION	PEST	PCB	TPH FINGERPRINT	PAH	VOA	TBT	METALS	OTHER	ACIDIFIED	PRESERVED	Total Number of Containers
		T2736	E-PH03-SC-C	M. Nasuta (clams)											1
		T2737	E-PH03-SC-D												1
		T2738	E-PH03-SC-E												1
		T2739	E-PH03-SC-F												1
		T2740	E-PH03-SC-G												1
		T2741	E-PH03-SC-H												1
		T2742	E-PH03-SC-I												1
		T2743	PRETEST-M.nasuta-	A											1
		T2744	PRETEST-M.nasuta-	B											1
		T2745	PRETEST-M.nasuta-	C											1

Relinquished by:	Date/Time	Received by:	Date/Time
		See Original COC	

Relinquished by:	Date/Time	Received by:	Date/Time

Comments:

009

ORIGINAL

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Chain of Custody

Proj. No	Proj. Name
—	PONCE HARBOR

SAMPLERS: Signature

ANALYSIS REQUESTED → "NUMBER OF CONTAINERS"

DATE	TIME	BATTELLE ID	CLIENT ID	SAMPLE DESCRIPTION	PEST	PCB	TPH FINGERPRINT	PAH	VOA	TBT	METALS	OTHER	ACIDIFIED	PRESERVED	Total Number of Containers
6/16/03	—	T4583	Filter Swab	Filter Blank				X							1

Relinquished by:	Date/Time		Received by:	Date/Time	
JM Barber	6/16/03	1:50pm	Jessie N. Joy	6/16/03	15:30
Relinquished by:	Date/Time		Received by:	Date/Time	

Comments:

Logged into ship - 030530-02

010

ORIGINAL

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ShpNo SHP-030530-02

Battelle Project No: N005561

Sample Receipt Form Details

Approved: ☑ Authorized ☑

Project Number:

Client: PPB Environmental

Received by: Smith, Joe

Date/Time Received: Friday, May 30, 2003 12:00 AM

No. of Shipping Containers: 1

BDO Id:	Client Sample ID:	Collection Date:	Login Date:	Ctrs:	Matrix:	Temp:	pH:	TRC:	VOC:	Stored In:	Loc:	No:	Comments:
T2702	E-PH03-CONTROL-A		05/30/03 14:51	1	TISSUE(WORMS	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2703	E-PH03-CONTROL-B		05/30/03 14:58	1	TISSUE (worms)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2704	E-PH03-CONTROL-C		05/30/03 14:59	1	TISSUE (worms)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2705	E-PH03-CONTROL-D		05/30/03 14:59	1	TISSUE (worms)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2706	E-PH03-CONTROL-E		05/30/03 14:59	1	TISSUE (worms)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2707	RS-PH03-A		05/30/03 15:00	1	TISSUE (worms)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2708	RS-PH03-B		05/30/03 15:07	1	TISSUE (worms)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2709	RS-PH03-C		05/30/03 15:07	1	TISSUE (worms)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2710	RS-PH03-D		05/30/03 15:08	1	TISSUE (worms)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2711	RS-PH03-E		05/30/03 15:08	1	TISSUE (worms)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2712	E-PHO3-SC-A		05/30/03 15:08	1	TISSUE (worms)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2713	E-PHO3-SC-B		05/30/03 15:09	1	TISSUE (worms)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2714	E-PHO3-SC-C		05/30/03 15:09	1	TISSUE (worms)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2715	E-PHO3-SC-D		05/30/03 15:09	1	TISSUE (worms)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2716	E-PHO3-SC-E		05/30/03 15:10	1	TISSUE (worms)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2717	E-PHO3-SC-F		05/30/03 15:10	1	TISSUE (worms)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2718	E-PHO3-SC-G		05/30/03 15:10	1	TISSUE (worms)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2719	E-PHO3-SC-H		05/30/03 15:11	1	TISSUE (worms)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2720	E-PHO3-SC-I		05/30/03 15:11	1	TISSUE (worms)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2721	PRETEST N.VIRENS-A		05/30/03 15:11	1	TISSUE (worms)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2722	PRETEST N.VIRENS-B		05/30/03 15:12	1	TISSUE (worms)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2723	PRETEST N.VIRENS-C		05/30/03 15:12	1	TISSUE (worms)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2724	E-PH03-CONTROL-A		05/30/03 15:14	1	TISSUE (clams)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2725	E-PH03-CONTROL-B		05/30/03 15:14	1	TISSUE (clams)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2726	E-PH03-CONTROL-C		05/30/03 15:15	1	TISSUE (clams)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2727	E-PH03-CONTROL-D		05/30/03 15:15	1	TISSUE (clams)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2728	E-PH03-CONTROL-E		05/30/03 15:15	1	TISSUE (clams)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2729	RS-PH03-A		05/30/03 15:15	1	TISSUE (clams)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	

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ShpNo SHP-030530-02

Battelle Project No: N005561

Sample Receipt Form Details

Approved: ☑ Authorized ☑

Project Number:

Client: PPB Environmental

Received by: Smith, Joe

Date/Time Received: Friday, May 30, 2003 12:00 AM

No. of Shipping Containers: 1

BDO Id:	Client Sample ID:	Collection Date:	Login Date:	Ctrs:	Matrix:	Temp:	pH:	TRC:	VOC:	Stored In:	Loc:	No:	Comments:
T2730	RS-PH03-B		05/30/03 15:16	1	TISSUE (clams)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2731	RS-PH03-C		05/30/03 15:16	1	TISSUE (clams)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2732	RS-PH03-D		05/30/03 15:16	1	TISSUE (clams)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2733	RS-PH03-E		05/30/03 15:16	1	TISSUE (clams)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2734	E-PH03-SC-A		05/30/03 15:17	1	TISSUE (clams)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2735	E-PH03-SC-B		05/30/03 15:17	1	TISSUE (clams)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2736	E-PH03-SC-C		05/30/03 15:17	1	TISSUE (clams)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2737	E-PH03-SC-D		05/30/03 15:18	1	TISSUE (clams)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2738	E-PH03-SC-E		05/30/03 15:18	1	TISSUE (clams)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2739	E-PH03-SC-F		05/30/03 15:18	1	TISSUE (clams)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2740	E-PH03-SC-G		05/30/03 15:18	1	TISSUE (clams)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2741	E-PH03-SC-H		05/30/03 15:18	1	TISSUE (clams)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2742	E-PH03-SC-I		05/30/03 15:22	1	TISSUE (clams)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2743	PRETEST-M.NASUTA-A		05/30/03 15:22	1	TISSUE (clams)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2744	PRETEST-M.NASUTA-B		05/30/03 15:23	1	TISSUE (clams)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T2745	PRETEST-M.NASUTA-C		05/30/03 15:23	1	TISSUE (clams)	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	
T4583	Filter Swab	06/16/03 0:00	06/16/03 13:48	1	Filter	-2.7	NA	NA	NA	Walk-in Freezer	BIN	57	

Total Samples: 45

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Sample Split and Transfer Log

Project Number	N005561	Date of Work	6/3/03
Project Title	PONCE HARBOR		
Analysis Type(s)	metals		
Splitting Procedure	~10 g. of bulk sample was poured into SPEC jars, then frozen. 40g. were removed from T2711 and T2733. (include description of amount or weight of split, packaging, storage)		
Name	Theresa Barber	Date	6/3/03

Sample ID	Sample ID	Sample ID	Sample ID	Sample ID	Sample ID
T2702	T2712	T2722	T2731	T2741	
T2703	T2713	T2723	T2732	T2742	
T2704	T2714	S/B 6/3/03 TMB ~~T2273~~	T2733	T2743	
T2705	T2715	T2724	T2734	T2744	
T2706	T2716	T2725	T2735	T2745	
T2707	T2717	T2726	T2736		
T2708	T2718	T2727	T2737		
T2709	T2719	T2728	T2738		
T2710	T2720	T2729	T2739		
T2711	T2721	T2730	T2740		

Released	Received
Signature/Date: Theresa Barber 6/3/03 (1)	Signature/Date: Jessica M. Jay 6/3/03 (1)
Storage Location/Conditions: walk in freezer -24°C	Storage Location/Conditions: Walk-In Freezer -24°C
Released	**Received**
Signature/Date:	Signature/Date:
Storage Location/Conditions:	Storage Location/Conditions:

(1) 1R JMF 6/5/03

ORIGINAL

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ShpNo SHP-030616-03

Battelle Project No: N005561

Sample Receipt Form

Approved: ☑

Project Number: N005561 **Client:** PPB laboratory Inc

Received by: Szleper, Kathleen **Date/Time Received:** Monday, June 16, 2003 10:40 AM

No. of Shipping Containers: 1

SHIPMENT

Method of Delivery: Hand Delivered **Tracking Number:**

COC Forms: ☑ **Shipped with samples** ☐ **No Forms**

Cooler(s)/Box(es)

Container	Type	Sealed With	Seal Condition	Container Condition	Temp C	Total Samples
1 of 1	No Container	None	Not Applicable	Not Applicable	Ambient	2

Samples

Sample Labels:
☑ Sample labels agree with COC forms
☐ Discrepancies (see Sample Custody Corrective Action Form)

Container Seals:
☐ Tape ☐ Custody Seals ☐ Other Seals (See sample Log)
☑ Seals intact for each shipping container
☐ Seals broken (See sample log for impacted samples)

Condition of Samples:
☑ Sample containers intact
☐ Sample containers broken/leaking (See Custody Corrective Action Form)

Temperature upon receipt (°C): Ambient Temperature Blank used ☐ Yes ☑ No
(Note: If temperature upon receipt differs from required conditions, see sample log comment field)

Samples Acidified: ☐ Yes ☐ No ☑ Unknown

Initial pH 5-9?: ☐ Yes ☐ No ☑ NA
If no, individual sample adjustments on the Auxiliary Sample Receipt Form

Total Residual Chlorine Present?: ☐ Yes ☐ No ☑ NA
If yes, individual sample adjustments on the Auxiliary Sample Receipt Form

Head Space <1% in samples for water VOC analysis: ☐ Yes ☐ No ☑ NA
Individual sample deviations noted on sample log

Samples Containers:
Samples returned in PC-grade jars: ☐ Yes ☑ No ☐ Unknown /Lot No.: UnKnown

Storage Location: Walk-in Freezer **BDO IDs Assigned:** T4581 - T4582

Samples logged in by: Szleper, Kathleen **Date/Time:** 06/16/2003 10:38 AM

Approved By: Fahey, Jessica **Approved On:** 6/16/2003 11:5

Authorized By: **Authorized On:**

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Chain of Custody

Proj. No	Proj. Name
N005561	PONCE HARBOR

SAMPLERS: Signature

ANALYSIS REQUESTED → "NUMBER OF CONTAINERS"

DATE	TIME	BATTELLE ID	CLIENT ID	SAMPLE DESCRIPTION	PEST	PCB	TPH FINGERPRINT	PAH	VOA	TBT	METALS	OTHER	ACIDIFIED	PRESERVED	Total Number of Containers
6/16/03	10:30AM	T4581	COMPOSITE-WORMS	COMPOSITE OF BATTELLE ID'S T2716, T2717, T2718, T2719 T2720											1
6/16/03	10:30AM	T4582	COMPOSITE-CLAMS	COMPOSITE OF BATTELLE ID'S T2738, T2739, T2740, T2741 T2742											1

Relinquished by:	Date/Time		Received by:	Date/Time	
Theresa M. Barber	6/16/03	10:40 AM.	Kathleen Slyn	6/16/03	10:40AM
Relinquished by:	Date/Time		Received by:	Date/Time	

Comments:

ORIGINAL

015

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ShpNo SHP-030616-03

Battelle Project No:

Sample Receipt Form Details

Approved: ☐ Authorized ☐

Project Number:

Client: PPB laboratory Inc

Received by: Szleper, Kathleen

Date/Time Received: Monday, June 16, 2003 10:40 AM

No. of Shipping Containers: 1

BDO Id:	Client Sample ID:	Collection Date:	Login Date:	Ctrs:	Matrix:	Temp:	pH:	TRC:	VOC:	Stored In:	Loc:	No:	Comments:
T4581	Composite worms	06/16/03 10:30	06/16/03 10:41	1	tissue		NA	NA	NA	Walk-in Freezer	BIN	57	
T4582	Composite clams	06/16/03 10:30	06/16/03 10:41	1	tissue		NA	NA	NA	Walk-in Freezer	BIN	57	

Total Samples: 2

QA/QC NARRATIVE

PROJECT: Chemical Evaluation of Dredged Material from Ponce Harbor

PARAMETER: Metals

LABORATORY: Battelle Marine Sciences Laboratory, Sequim, Washington

MATRIX: Tissues (*Nereis. virens* and *Macoma nasuta*)

SAMPLE CUSTODY AND PROCESSING: Battelle MSL received thirty-four tissue samples in two shipments on 06/11/03 and 06/17/03. All samples were received in good condition (i.e., all sample containers were intact). Samples were assigned a Battelle Central File (CF) identification number (1982) and were entered into Battelle's laboratory information and sample tracking system.

The sample count included the following samples for each species: 5 replicates of reference tissue, 9 replicates of test tissue and 3 replicates of pretest tissue. The first four replicates of the test tissue were analyzed individually and the remaining five were composited into a single sample for each species (i.e. 1982*49 for *N. virens* and 1982*63 *M. nasuta*). The final sample count included 13 *N. virens* samples and 13 *M. nasuta* samples for metals analyses.

The following lists information on sample receipt and processing activities:

Chemistry Lab ID	1982- 40 through –73
Collection dates [(a)]	See BOS records
Laboratory arrival date	06/11/03 and 06/17/03
Cooler temperatures, on arrival	2.4 and 2.0 °C
Deep Freeze (-58 °C ± 20 °C)	06/11/03 and 06/17/03
Freeze Dryer	06/17/03 to 06/21/03
Digestion (aqua regia)	06/27/03 and 06/30/03
CVAA analysis (Hg)	07/01/03 and 07/02/03
ICP-MS analysis (Ag, As, Cd, Cr, Cu, Ni, Pb, Se, Zn)	07/02/03

(a) Documentation of sample custody as it pertains to metals analysis begins with sample split date at Battelle Ocean Sciences (BOS) Duxbury operations.

QA/QC DATA QUALITY OBJECTIVES:

Analyte	Analytical Method	Range of Recovery	SRM Accuracy	Relative Precision	Laboratory Achieved Detection Limits (µg/g) (WET wt.)[a]	(DRY wt.)[b]
Silver	ICP-MS	75-125%	≤20%	≤20%	0.006	0.04
Arsenic	ICP-MS	75-125%	≤20%	≤20%	0.03	0.2
Cadmium	ICP-MS	75-125%	≤20%	≤20%	0.003	0.02
Chromium	ICP-MS	75-125%	≤20%	≤20%	0.03	0.2
Copper	ICP-MS	75-125%	≤20%	≤20%	0.01	0.1
Mercury	CVAA	75-125%	≤20%	≤20%	0.0003	0.002
Nickel	ICP-MS	75-125%	≤20%	≤20%	0.007	0.07
Lead	ICP-MS	75-125%	≤20%	≤20%	0.004	0.04
Selenium	ICP-MS	75-125%	≤20%	≤20%	0.02	0.06
Zinc	ICP-MS	75-125%	≤20%	≤20%	0.14	1

(a) Tissue wet weight MDLs calculated using laboratory-achieved dry weight MDLs and a representative sample percent dry weight.

(b) Laboratory achieved tissue MDLs are provided from the 2002 MDL study, which were provided in the QAPP as the target detection limits; therefore target and achieved detection limits are identical.

QA/QC NARRATIVE

METHODS:

Ten metals were analyzed: silver (Ag), arsenic (As), cadmium (Cd), chromium (Cr), copper (Cu), mercury (Hg), nickel (Ni), lead (Pb), selenium (Se) and zinc (Zn). Tissue samples were digested according to Battelle SOP MSL-I-024, *Mixed Acid Tissue Digestion*. An approximately 500-mg aliquot of each dried, homogeneous sample was combined with nitric and hydrochloric acids (aqua regia) in a Teflon vessel and heated in an oven at 130°C (±10°C) for a minimum of eight hours. After heating and cooling, deionized water was added to the acid-digested tissue to achieve analysis volume and the digestates were submitted for analysis by two methods: CVAA and ICP-MS.

Sample digestates were analyzed for Hg using cold-vapor atomic absorption spectroscopy (CVAA) according to Battelle SOP MSL-I-016, *Total Mercury in Tissues and Sediments by Cold Vapor Atomic Absorption*, which is based on EPA Method 245.5, *Determination of Mercury in Sediments by Cold Vapor Atomic Absorption Spectrometry*.

Sample digestates were analyzed for Ag, As, Cd, Cr, Cu, Ni, Pb, Se and Zn using inductively coupled plasma-mass spectrometry (ICP-MS) according to Battelle SOP MSL-I-022, *Determination of Elements in Aqueous and Digestate Samples by ICP/MS*. This procedure is based on two methods modified and adapted for analysis of solid sample digestates: EPA Method 1638, *Determination of Trace Elements in Ambient Waters by Inductively Coupled Plasma-Mass Spectrometry* and EPA Method 1640, *Determination of Trace Elements in Water by Preconcentration and Inductively Coupled Plasma-Mass Spectrometry*.

All results were determined and reported in units of µg/g on a dry-weight basis and converted to µg/g on a wet-weight basis calculated using the percent dry weight of each sample.

HOLDING TIMES:

The recommended holding times for metals analyses are 28 days from sample collection for Hg analysis, and 6 months from sample collection for analysis of all other metals. The Quality Assurance Plan for Chemical Evaluation of Dredged Material from Ponce Harbor also allows for frozen tissue samples to be analyzed within one year of collection. *M. nasuta* and *N. virens* samples were frozen on receipt and then freeze dried and analyzed within 1 year.

DETECTION LIMITS:

Laboratory target and achieved method detection limit are provided in the QAPP. Sample concentrations were evaluated and flagged to the following criteria:

- **U** Analyte not detected, achieved MDL reported
- **B** Analyte detected in the method blank at a level greater than 3 times the MDL.
- **&** QC value outside the accuracy or precision criteria goal

METHOD BLANKS:

One method blank was analyzed with each of the two digestion batches. Analytes in the method blanks were either undetected above the achieved MDL or less than 3 times the MDL, with the exception of one method blank for Se. Samples associated with the digestion batch were flagged with a "B". Data were evaluated for potential impact due to the detected blank concentration. The impact to the digestion batch is minimal as all QC samples are within their respective criterion and the concentration of the samples is greater than 10 times the level of Se detected in the blank. No corrective action was initiated. The data were not blank-corrected.

QA/QC NARRATIVE

LABORATORY CONTROL SAMPLE/BLANK SPIKE ACCURACY:

Two replicates of blank spike/laboratory control samples (LCS) were analyzed with the set of samples. LCS samples were fortified at approximately 2 µg/g for As, Cd, Cr, Cu, Ni, Hg, Se, and Zn. LCS recoveries among all metals ranged from 91% to 114% and were within the QC acceptance criteria of 75% to 125% for all metals, with the exception of one replicate for Hg (70%). Acceptable analytical accuracy for Hg was demonstrated in the alternate replicate of the LCS and all other QC samples, including the matrix spiked samples and SRMs. No corrective action was initiated.

MATRIX SPIKE ACCURACY:

Two tissue samples, one of each species, were selected as matrix spike samples. Each species was spiked at approximately 2 µg/g or 25 µg/g, analyte specific. MS recoveries ranged from 75% to 108% and were within the QC acceptance criteria of 75% to 125%, with the exception of one replicate for Cu (157%). The high spike recovery results from an inappropriate spiking level relative to the native Cu concentration. Acceptable analytical accuracy for Cu was demonstrated in the alternate matrix spike (with appropriate spiking level), the two replicates of the LCS samples, and all four SRM replicates.

REPLICATE PRECISION:

Two sets of triplicate samples, one for each species, were analyzed with the set of samples. Precision of triplicate analyses is expressed as the relative standard deviation (RSD) of replicate results. RSDs for all metals ranged from 1% to 16% and were within the QC limits of ≤20% for metals, with the exception of one set of triplicate samples for Ag (22%). The sample concentration is less than 10 times the MDL; therefore the QC sample is not an appropriate indicator of analytical precision. Acceptable precision was demonstrated in the alternate set of triplicates. No corrective action was initiated.

STANDARD REFERENCE MATERIAL ACCURACY:

SRM accuracy was expressed as the percent difference (PD) between the measured and certified SRM concentrations. Recovery of a particular analyte exceeded QC criteria if the PD, determined from either end of the acceptable range or 95% confidence interval for the particular analyte, exceeded 20% PD. SRMs are reported for analytes certified greater than 10 times the achieved MDL.

Two replicates of SRMs 1566b and DORM-2 were analyzed with the set of samples. Multiple SRMs were selected because no single SRM is certified for all metals of interest at appropriate concentration ranges. The PD recoveries for SRM 1566b ranged from 6% to 20% and were within QC acceptance criterion. The SRM 1566b is not certified for Cr.

The PD recoveries for SRM DORM-2 ranged from 0% to 19% PD and were within the QC acceptance criterion.

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PONCE HARBOR DREDGE EVALUATION
METALS IN TISSUES

(concentrations in µg/g WET wt. - data are not blank corrected)

Client ID	BOS Code	Matrix	MSL Sample ID	Percent Solids	Ag	As	Cd	Cr	Cu	Hg	Ni	Pb	Se	Zn
RS-PH03-A	T2707	*N. virens*	1982-40	13.8	0.0322	1.52	0.0300	1.01	51.3	0.0328	0.697	0.574	0.431 B	13.8
RS-PH03-B	T2708	*N. virens*	1982-41	14.7	0.0295	1.45	0.0315	0.817	71.1	0.0388	0.566	1.063	0.478 B	23.0
RS-PH03-C	T2709	*N. virens*	1982-42	18.3	0.0414	2.11	0.0423	1.48	31.0	0.0441	0.781	1.791	0.689 B	27.3
RS-PH03-D	T2710	*N. virens*	1982-43	14.2	0.0304	1.55	0.0332	0.939	89.4	0.0333	0.733	0.335	0.432 B	11.0
RS-PH03-E	T2711	*N. virens*	1982-44	20.0	0.0403	2.10	0.0433	1.39	90.0	0.0372	1.14	0.497	0.589 B	36.3
E-PH03-SC-A	T2712	*N. virens*	1982-45	8.60	0.0205	0.767	0.0221	0.869	67.2	0.0227	0.533	0.345	0.329 B	9.38
E-PH03-SC-B	T2713	*N. virens*	1982-46	9.75	0.0204	0.877	0.0228	0.881	66.8	0.0283	0.527	0.334	0.371 B	8.38
E-PH03-SC-C	T2714	*N. virens*	1982-47	11.7	0.0281	1.08	0.0318	0.343	66.1	0.0207	0.322	0.250	0.359 B	13.9
E-PH03-SC-D	T2715	*N. virens*	1982-48	11.9	0.0237	1.03	0.0289	0.395	25.3	0.0192	0.403	0.242	0.439 B	21.1
E-PH03-SC-E (composite)	T2716	*N. virens*	1982-49 r1	10.5	0.0241	0.878	0.0263	0.633	51.7	0.0208	0.573	0.304	0.330 B	11.2
E-PH03-SC-E (composite)	T2716	*N. virens*	1982-49 r2	10.5	0.0237	0.851	0.0248	0.623	51.1	0.0208	0.566	0.289	0.321 B	14.4
E-PH03-SC-E (composite)	T2716	*N. virens*	1982-49 r3	10.5	0.0247	0.832	0.0242	0.614	54.2	0.0210	0.551	0.301	0.356 B	10.9
PRETEST N.VIRENS-A	T2721	*N. virens*	1982-71	18.9	0.0423	2.21	0.0455	1.26	2.21	0.0538	0.650	0.467	0.548 B	11.7
PRETEST N.VIRENS-B	T2722	*N. virens*	1982-72	9.94	0.0113	0.829	0.0030 U	0.667	1.44	0.0244	0.286	4.99	0.339 B	9.72
PRETEST N.VIRENS-C	T2723	*N. virens*	1982-73	10.6	0.0117	0.819	0.0209	0.834	1.35	0.0265	0.343	2.85	0.333 B	8.92
		MEAN Dry Weight N. virens		*12.9*										
RS-PH03-A	T2729	*M. nasuta*	1982-54	14.1	0.0187	2.46	0.0309	0.288	1.73	0.0136	0.368	0.145	0.393	10.3
RS-PH03-B	T2730	*M. nasuta*	1982-55	8.55	0.0109	1.72	0.0168	0.135	0.881	0.00551	0.245	0.0771	0.259	6.69
RS-PH03-C	T2731	*M. nasuta*	1982-56	9.91	0.0137	2.08	0.0153	0.110	0.939	0.00628	0.241	0.0750	0.287	6.52
RS-PH03-D	T2732	*M. nasuta*	1982-57	9.92	0.0145	2.02	0.0175	0.117	1.04	0.00652	0.220	0.0770	0.261	6.81
RS-PH03-E	T2733	*M. nasuta*	1982-58	6.96	0.0120	1.26	0.0162	0.124	0.967	0.00487	0.207	0.0710	0.205	5.32
E-PH03-SC-A	T2734	*M. nasuta*	1982-59	9.71	0.0155	1.89	0.0221	0.498	1.854	0.00711	0.440	0.127	0.287	7.06
E-PH03-SC-B	T2735	*M. nasuta*	1982-60	9.08	0.0103	2.10	0.0181	0.130	0.843	0.00557	0.244	0.0694	0.296	5.79
E-PH03-SC-C	T2736	*M. nasuta*	1982-61	10.5	0.0119	1.74	0.0202	0.120	1.12	0.00756	0.286	0.0737	0.291	6.65
E-PH03-SC-D	T2737	*M. nasuta*	1982-62	8.69	0.0109	1.69	0.0154	0.164	0.921	0.00636	0.227	0.0704	0.287	5.62
E-PH03-SC-E (composite)	T2738	*M. nasuta*	1982-63 r1	8.66	0.0143	1.70	0.0181	0.139	1.08	0.00583	0.231	0.0759	0.271	6.49
E-PH03-SC-E (composite)	T2738	*M. nasuta*	1982-63 r2	8.66	0.00909	1.52	0.0163	0.138	0.935	0.00603	0.219	0.0630	0.246	5.53
E-PH03-SC-E (composite)	T2738	*M. nasuta*	1982-63 r3	8.66	0.0119	1.76	0.0182	0.150	1.07	0.00631	0.263	0.0739	0.268	6.33
PRETEST-M-NASUTA-A	T2743	*M. nasuta*	1982-68	9.20	0.0141	1.45	0.0265	0.109	7.31	0.00909	0.375	0.0795	0.278	10.9
PRETEST-M-NASUTA-B	T2744	*M. nasuta*	1982-69	12.6	0.0149	2.37	0.0369	0.226	8.67	0.0100	0.690	0.137	0.368	11.4
PRETEST-M-NASUTA-C	T2745	*M. nasuta*	1982-70	7.83	0.00838	1.58	0.0225	0.0901	6.50	0.00673	0.393	0.0627	0.253	7.07
		MEAN Dry Weight M. nasuta		*9.53*										
Target and Achieved Method Detection Limit (WET Wt.) (1)					**0.006**	**0.03**	**0.003**	**0.03**	**0.01**	**0.0003**	**0.007**	**0.004**	**0.02**	**0.14**
Target and Achieved Method Detection Limit (Dry Wt.) (2)					0.04	0.2	0.02	0.2	0.1	0.002	0.07	0.04	0.06	1
Method Blank														
Blank (1)		*N. virens Batch*	blk 062703		0.006 U	0.03 U	0.003 U	0.03 U	0.0295	0.000390	0.007 U	0.004 U	0.0430 B	0.14 U
Blank (2)		*M. nasuta Batch*	blk 063003		0.006 U	0.03 U	0.003 U	0.03 U	0.0113	0.000500	0.007 U	0.004 U	0.02 U	0.14 U

U Not detected; Achieved MDL reported
& QC value outside the accuracy or precision criteria goal (spike recovery ±25%; SRM ±20% PD; triplicate precision <20% RSD).
B Analyte detected in the method blank at level greater than 3 x MDL.
(1) Tissue wet weight target MDLs calculated using laboratory-achieved dry weight MDLs and a representative sample percent dry weight of 14%.
(2) Achieved dry weight tissue MDLs listed in QAPP

020

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PONCE HARBOR DREDGE EVALUATION
METALS IN QC TISSUE SAMPLES

(concentrations in µg/g Dry wt. - data are not blank corrected)

Client ID	BOS Code	Matrix	MSL Sample ID	Percent Solids	Ag	As	Cd	Cr	Cu	Hg	Ni	Pb	Se	Zn
Method Blank														
Blank (1)		*N. virens Batch*	blk 062703		0.04 U	0.2 U	0.02 U	0.2 U	0.229	0.00300	0.07 U	0.04 U	0.333 B	1 U
Blank (2)		*M. nasuta Batch*	blk 063003		0.04 U	0.2 U	0.02 U	0.2 U	0.119	0.00520	0.07 U	0.04 U	0.06 U	1 U
Target and Achieved Method Detection Limit (WET Wt.)(1)					0.006	0.03	0.003	0.03	0.01	0.0003	0.007	0.004	0.02	0.14
Target and Achieved Method Detection Limit (Dry Wt.)(2)					**0.04**	**0.2**	**0.02**	**0.2**	**0.1**	**0.002**	**0.07**	**0.04**	**0.06**	**1**
Laboratory Control Sample Results														
LCS (1)		*N. virens Batch*	LCS L1 062703		2.02	2.10	2.02	2.08	2.05	1.83	1.96	2.16	2.41	1.89
Blank (1)		*N. virens Batch*	blk 062703		0.04 U	0.2 U	0.02 U	0.2 U	0.229	0.00300	0.07 U	0.04 U	0.333 B	1 U
Spike Concentration					2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00
Percent Recovery					**101%**	**105%**	**101%**	**104%**	**91%**	**91%**	**98%**	**108%**	**104%**	**95%**
LCS (2)		*M. nasuta Batch*	BS L1 063003		2.02	2.07	2.03	2.08	2.01	1.41	1.89	2.27	2.02	1.94
Blank (2)		*M. nasuta Batch*	blk 063003		0.04 U	0.2 U	0.02 U	0.2 U	0.119	0.00520	0.07 U	0.04 U	0.06 U	1 U
Spike Concentration					2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00
Percent Recovery					**101%**	**104%**	**102%**	**104%**	**95%**	**70% &**	**95%**	**114%**	**101%**	**97%**
Standard Reference Material Results														
SRM 1 R1		*N. virens Batch*	1566b 062703		0.590	6.72	2.26	0.796	66.5	0.0323	0.936	0.285	2.47 B	1260
SRM 1 R2		*M. nasuta Batch*	1566b 063003		0.569	6.46	2.22	0.757	66.2	0.0345	0.904	0.289	2.46	1280
certified value					0.666	7.65	2.48	NC	71.6	0.0371	1.04	0.308	2.06	1424
range					±0.009	±0.65	±0.08		±1.6	±0.0013	±0.09	±0.009	±0.15	±46
percent difference					**11%**	**12%**	**9%**	**NA**	**7%**	**13%**	**10%**	**7%**	**20%**	**12%**
					15%	**16%**	**10%**	**NA**	**8%**	**7%**	**13%**	**6%**	**19%**	**10%**
SRM 2 R1		*N. virens Batch*	dorm2 062703		0.047	16.7	0.0457	30.5	2.07	4.33	16.0	0.0555	1.60 B	21.7
SRM 2 R2		*M. nasuta Batch*	dorm2 063003		0.041	16.5	0.0468	32.6	2.07	4.43	17.1	0.0671	1.78	21.7
certified value					0.041	18.0	0.043	34.7	2.34	4.64	19.4	0.065	1.49	25.6
range					±0.013	±1.1	±0.008	±5.5	±0.16	±0.26	±3.1	±0.007	±0.09	±2.3
percent difference					**13%**	**7%**	**6%**	**12%**	**12%**	**7%**	**18%**	**15%**	**7%** (3)	**15%**
					0%	**8%**	**9%**	**6%**	**12%**	**4%**	**12%**	**3%**	**19%** (3)	**15%**

021

BATTELLE MARINE SCIENCES LABORATORY
1529 West Sequim Bay Road
Sequim, Washington 98382
(360) 681-4564

PONCE HARBOR DREDGE EVALUATION
METALS IN QC TISSUE SAMPLES

(concentrations in µg/g Dry wt. - data are not blank corrected)

Client ID	BOS Code	Matrix	MSL Sample ID	Percent Solids	Ag	As	Cd	Cr	Cu	Hg	Ni	Pb	Se	Zn
Matrix Spike Results														
MS (1)	T2716	*N. virens*	1982-49 MS		1.97	31.7	2.10	28.8	532	2.25	27.0	4.84	5.27 B	181
E-PH03-SC-E (composite)	T2716	*N. virens*	1982-49 r1	10.5	0.230	8.38	0.251	6.04	493	0.198	5.47	2.90	3.15	107
Spike Concentration					1.97	24.9	1.97	24.9	24.9	1.97	24.9	1.97	1.97	99.6
Percent Recovery					**88%**	**94%**	**94%**	**91%**	**157% &, SL**	**104%**	**86%**	**98%**	**108%**	**75%**
MS (2)	T2738	*M. nasuta*	1982-63 MS		1.92	46.3	2.08	3.27	35.3	2.13	25.5	2.87	4.70	161
E-PH03-SC-E (composite)	T2738	*M. nasuta*	1982-63 r1	8.66	0.165	19.6	0.209	1.60	12.5	0.0673	2.67	0.877	3.13	74.9
Spike Concentration					1.97	24.8	1.97	1.97	24.8	1.97	24.8	1.97	1.97	99.0
Percent Recovery					**89%**	**108%**	**95%**	**85%**	**92%**	**105%**	**92%**	**101%**	**80%**	**87%**
Replicate Analysis Results														
E-PH03-SC-E (composite)	T2716	*N. virens*	1982-49 r1	10.5	0.230	8.38	0.251	6.04	493	0.198	5.47	2.90	3.15 B	107
E-PH03-SC-E (composite)	T2716	*N. virens*	1982-49 r2	10.5	0.226	8.12	0.237	5.94	488	0.198	5.40	2.76	3.06 B	137
E-PH03-SC-E (composite)	T2716	*N. virens*	1982-49 r3	10.5	0.236	7.94	0.231	5.86	517	0.201	5.26	2.87	3.40 B	104
Mean					0.231	8.15	0.240	5.95	499	0.199	5.38	2.84	3.20 B	116
RSD					**2%**	**3%**	**4%**	**2%**	**3%**	**1%**	**2%**	**3%**	**5%**	**16%**
E-PH03-SC-E (composite)	T2738	*M. nasuta*	1982-63 r1	8.66	0.165	19.6	0.209	1.60	12.5	0.0673	2.67	0.877	3.13	74.9
E-PH03-SC-E (composite)	T2738	*M. nasuta*	1982-63 r2	8.66	0.105	17.6	0.188	1.59	10.8	0.0696	2.53	0.728	2.84	63.9
E-PH03-SC-E (composite)	T2738	*M. nasuta*	1982-63 r3	8.66	0.138	20.3	0.210	1.73	12.4	0.0729	3.04	0.854	3.09	73.1
Mean					0.136	19.2	0.202	1.64	11.9	0.0699	2.75	0.82	3.02	71
RSD					**22% &, (4)**	**7%**	**6%**	**5%**	**8%**	**4%**	**10%**	**10%**	**5%**	**8%**

U Not detected; Achieved MDL reported
& QC value outside the accuracy or precision criteria goal (spike recovery ±25%; SRM ±20% PD; triplicate precision <20% RSD).
B Analyte detected in the method blank at level greater than 3 x MDL.
RSD Relative Standard Deviation
PD Percent Difference
NC SRM not certified for this analyte.
NA Not Applicable
SL Insufficient spiking level relative to native sample concentration
(1) Tissue wet weight target MDLs calculated using laboratory-achieved dry weight MDLs and a representative sample percent dry weight.
(2) Achieved dry weight tissue MDLs listed in QAPP
(3) SRM percent difference calculated from the upper 95% confience interval of the certified range
(4) Analyte less than 10x the MDL; therefore not appropriate QC sample for evaluating analytical precision

022

PAH – TISSUE QA/QC SUMMARY
Batch 03-0539

PROJECT: Ponce Harbor
PARAMETER: PAH
LABORATORY: Battelle, Duxbury, MA
MATRIX: Tissue (Nereis)
SAMPLE CUSTODY: The tissue samples were received at Battelle Duxbury on May 30, 2003. The cooler temperature on arrival was –2.7 °C. Tissue was stored at -20 °C until processing began. The tissues arrived frozen in plastic bags. In order to determine if the tissues were impacted by the close proximity to the plastic, the insides of two individual bags were swiped with clean, muffled glass fiber filter papers and these filter papers extracted with sample batch 03-0539. Sample ID discrepancies were noted between the sample bags and the custody records. Mactec Engineering representatives responded quickly to the request for clarification. The information is included with the custody records. Selected samples were composited and returned to the sample custodian. These composites were given new unique Battelle IDs. All samples were in the walk-in freezer until sample preparation could begin.

	Reference Method	Method Blank	Surrogate Recovery	LCS/MS Recovery	SRM % Diff.	Sample Replicate Relative Precision	Detection Limits (ng/g wet wt)
PAH	General NS&T	B<3xMDL	30-150% Recovery	50-120% Recovery (for at least 80% of analytes; analyte conc. in MS must be >10x background)	≤30% PD (on average; not to exceed 35% for more than 30% of analytes; measured from range of certified values, for analytes >10x MDL)	≤30% RSD	Achieved MDLs: ~ 0.03 – 0.1 Region 2 Target MDL: 4 QAPP Target MDLs: 0.02 – 0.2

METHOD: Tissue samples were extracted for PAH following general NS&T methods. Approximately 30 g of tissue was spiked with surrogates and extracted three times with dichloromethane using tissuemizer and shaker table techniques. The combined extract was dried over anhydrous sodium sulfate, concentrated, processed through alumina cleanup column, concentrated, and further purified by GPC/HPLC. The post-HPLC extract was concentrated and fortified with RIS. Extracts were analyzed for PAH using gas chromatography/mass spectrometry (GC/MS), following general NS&T methods. Sample data were quantified by the method of internal standards, using the Recovery Internal Standard (RIS) compounds. In addition to the Nereis samples and associated QC samples that were extracted in this batch, three wipes, taken to ensure the sample containers were free of target analytes, were processed with this batch. These samples (T4578, T4579, and T4583), were processed identical to all other samples associated with batch 03-0539 except they did not go through alumina column cleanup or GPC/HPLC cleanup.

HOLDING TIMES: Samples were prepared for analysis in one analytical batch. Extracts were analyzed within 40 days of extraction.

Batch	Extraction Date	Analysis Date
03-0539	06/23/2003	06/28/2003 – 06/29/2003

PAH – TISSUE QA/QC SUMMARY
Batch 03-0539

BLANK:

A procedural blank (PB) was prepared with each analytical batch. Blanks were analyzed to ensure the sample extraction and analysis methods were free of contamination.

03-0539 – All target analytes were recovered within the laboratory control limits specified by the client.

Comments – Three target PAH were detected in the blank, however at levels < MDL.

LABORATORY CONTROL SAMPLE:

A laboratory control sample (LCS) was prepared with each analytical batch. The percent recoveries of target analytes were calculated to measure data quality in terms of accuracy.

03-0539 – All target analytes were recovered within the specified laboratory control limits.

Comments – None.

MATRIX SPIKE:

A matrix spike (MS) sample was prepared with each analytical batch. The percent recoveries of target analytes were calculated to measure data quality in terms of accuracy.

03-0539 – All analytes were recovered within the specified laboratory control limits.

Comments – None.

REPLICATES:

Replicate (triplicate) samples were prepared with each analytical batch. The RSD between triplicate analyses for PAH is calculated to measure data quality in terms of precision.

03-0539 – One exceedence noted.

Comments – One RSD was calculated to be in exceedence amongst T4581, T4581DUP, and T481TRIP. Anthracene was calculated to have an RSD of 34.4%. However this analyte was detected below the reporting limit in all three samples, therefore it was not suitable for data quality assessment. The exceedence was qualified with an "N" and no further corrective action was taken.

PAH – TISSUE QA/QC SUMMARY
Batch 03-0539

SRM: A standard reference material (SRM, NIST 1944) was prepared with each analytical batch. The percent difference (PD) between the measured value and the certified range was calculated to measure data quality in terms of accuracy. If the detected value fell within the certified range then the PD was calculated using the nearest end of the certified range.

03-0539 – Four exceedences noted.

Comments – Seven percent differences were calculated to be greater than 30% for sample BC749SRM. Three compounds that were calculated to have exceeding RPDs, Phenanthrene, Benzo(a)pyrene, and Indeno(1,2,3-cd)pyrene, also had certified target concentrations not greater than ten times the MDL. Because the certified target concentration was not greater than ten times the MDL, these data were not suitable for data quality assessment. These exceedences were qualified with an "n" to indicate that the QC value is outside the accuracy or precision data quality objective, but meets contingency criteria. The other four exceedences, Naphthalene, Benzo(b)fluoranthene, Benzo(k)fluoranthene, and Benzo(g,h,i)perylene, all had certified target concentrations greater than ten times the MDL. Results for these compounds were re-analyzed, but results were not improved. The exceeding data was reported and these exceedences have been qualified with an "N".

SURROGATES: Three surrogate compounds were added prior to extraction, including naphthalene-d8, phenanthrene-d10, and chryses-d12. The recovery of each surrogate compound was calculated to measure data quality in terms of accuracy (extraction efficiency).

03-0539 – Percent recoveries for all surrogate compounds were within the laboratory control limits specified by the method (30 – 150% recovery).

Comments – None.

CALIBRATIONS: The GC/MS is calibrated with a minimum of a 5-point curve. The RSD between response factors for the individual target analytes must be <20%. Each batch of samples analyzed is bracketed by a calibration check sample, run at a frequency of minimally every 10 samples. This PD between the initial calibration RF and the check should be <20% for individual analytes; 15% on average.

03-0539– No calibration exceedences.

Comments – None.

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Project Client: PPB Environmental Laboratories
Project Name: Ponce Harbor
Project Number: N005561

Client ID	Procedural Blank
Battelle ID	BC747PB
Sample Type	PB
Collection Date	06/23/03
Extraction Date	06/23/03
Analysis Date	06/28/03
Analytical Instrument	MS
% Moisture	88.22
% Lipid	NA
Matrix	
Samp_WGT_VOL	25.22
Samp_WGT_VOL_UNIT	G_WET
Units	NG/G_WET
1,4-Dichlorobenzene	0.07 J
Naphthalene	0.11
Biphenyl	0.03 U
Acenaphthylene	0.03 U
Acenaphthene	0.05 U
Fluorene	0.04 U
Phenanthrene	0.02 J
Anthracene	0.08 U
Fluoranthene	0.05 U
Pyrene	0.04 U
Benzo(a)anthracene	0.08 U
Chrysene	0.08 U
Benzo(b)fluoranthene	0.10 U
Benzo(k)fluoranthene	0.06 U
Benzo(a)pyrene	0.09 U
Indeno(1,2,3-cd)pyrene	0.06 U
Dibenz(a,h)anthracene	0.05 U
Benzo(g,h,i)perylene	0.05 U

Surrogate Recoveries (%)

Naphthalene-d8	77
Phenanthrene-d10	71
Chrysene-d12	82

J = Analytes detected below sample-specific MDL

U = Analyte not detected at a 3:1 signal:noise ratio. Sample-specific MDL

N = QC value outside of accuracy or precision MQO

Not Surrogate Corrected

Analyzed by Krahforst, Kerylynn
8/19/2003

PB: T03-0539MS-NYD_PAH-Final rev 4 FINAL

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Project Client: PPB Environmental Laboratories
Project Name: Ponce Harbor
Project Number: N005561

Client ID	PRETEST N.VIRENS-A	PRETEST N.VIRENS-B	PRETEST N.VIRENS-C
Battelle ID	T2721	T2722	T2723
Sample Type	SA	SA	SA
Collection Date	05/29/03	05/29/03	05/29/03
Extraction Date	06/23/03	06/23/03	06/23/03
Analysis Date	06/28/03	06/29/03	06/29/03
Analytical Instrument	MS	MS	MS
% Moisture	82.58	90.97	90.58
% Lipid	1.06	0.58	0.47
Matrix	Tissue (Nereis)	Tissue (Nereis)	Tissue (Nereis)
Samp_WGT_VOL	30.41	32.66	30.68
Samp_WGT_VOL_UNIT	G_WET	G_WET	G_WET
Units	NG/G_WET	NG/G_WET	NG/G_WET
1,4-Dichlorobenzene	4.68	0.32	0.16
Naphthalene	5.49	1.07	0.85
Biphenyl	0.49	0.21	0.18
Acenaphthylene	0.20	0.07	0.10
Acenaphthene	0.35	0.21	0.19
Fluorene	0.36	0.22	0.28
Phenanthrene	1.52	1.52	2.69
Anthracene	0.37	0.18	0.19
Fluoranthene	3.74	4.22	5.06
Pyrene	4.63	4.68	5.12
Benzo(a)anthracene	1.31	1.22	1.57
Chrysene	2.57	2.51	3.05
Benzo(b)fluoranthene	1.05	1.09	1.44
Benzo(k)fluoranthene	1.50	1.57	2.03
Benzo(a)pyrene	1.41	1.21	1.71
Indeno(1,2,3-cd)pyrene	0.98	0.78	1.16
Dibenz(a,h)anthracene	0.11	0.09	0.14
Benzo(g,h,i)perylene	1.31	1.00	1.23
Surrogate Recoveries (%)			
Naphthalene-d8	89	88	77
Phenanthrene-d10	82	82	72
Chrysene-d12	89	91	80

J = Analytes detected below sample-specific MDL

U = Analyte not detected at a 3:1 signal:noise ratio.
Sample-specific MDL reported

N = QC value outside of accuracy or precision MQO

Not Surrogate Corrected

Analyzed by Krahforst, Kerylynn
8/19/2003

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Project Client: PPB Environmental Laboratories
Project Name: Ponce Harbor
Project Number: N005561

Client ID	RS-PH03-A	RS-PH03-B	RS-PH03-C	RS-PH03-D	RS-PH03-E
Battelle ID	T2707	T2708	T2709	T2710	T2711
Sample Type	SA	SA	SA	SA	SA
Collection Date	05/29/03	05/29/03	05/29/03	05/29/03	05/29/03
Extraction Date	06/23/03	06/23/03	06/23/03	06/23/03	06/23/03
Analysis Date	06/28/03	06/28/03	06/28/03	06/28/03	06/28/03
Analytical Instrument	MS	MS	MS	MS	MS
% Moisture	87.53	87.05	83.56	87.39	81.18
% Lipid	0.75	0.72	0.87	0.72	1.24
Matrix	Tissue (Nereis)	Tissue (Nereis)	Tissue (Nereis)	Tissue (Nereis)	Tissue (Nereis)
Samp_WGT_VOL	30.04	30.29	30.63	30.08	30.94
Samp_WGT_VOL_UNIT	G_WET	G_WET	G_WET	G_WET	G_WET
Units	NG/G_WET	NG/G_WET	NG/G_WET	NG/G_WET	NG/G_WET
1,4-Dichlorobenzene	2.27	1.36	2.34	1.62	2.58
Naphthalene	2.81	1.65	2.97	1.92	3.62
Biphenyl	0.25	0.22	0.24	0.23	0.37
Acenaphthylene	0.10	0.08	0.17	0.13	0.22
Acenaphthene	0.15	0.19	0.25	0.17	0.33
Fluorene	0.18	0.2	0.27	0.21	0.30
Phenanthrene	1.05	1.30	2.10	1.48	1.23
Anthracene	0.13	0.13	0.37	0.20	0.14
Fluoranthene	2.52	3.70	4.80	3.17	4.70
Pyrene	2.72	4.32	4.98	3.81	4.44
Benzo(a)anthracene	0.75	0.66	1.96	0.77	0.64
Chrysene	1.75	2.18	3.15	1.73	2.66
Benzo(b)fluoranthene	0.70	0.92	1.68	0.89	1.06
Benzo(k)fluoranthene	1.06	1.05	1.77	0.96	1.10
Benzo(a)pyrene	0.93	0.93	2.02	1.06	1.01
Indeno(1,2,3-cd)pyrene	0.60	0.53	1.24	0.64	0.64
Dibenz(a,h)anthracene	0.08	0.06	0.16	0.06	0.06
Benzo(g,h,i)perylene	0.70	0.73	1.34	0.76	0.84
Surrogate Recoveries (%)					
Naphthalene-d8	83	79	92	88	90
Phenanthrene-d10	77	71	84	81	84
Chrysene-d12	86	79	92	91	94

J = Analytes detected below sample-specific MDL

U = Analyte not detected at a 3:1 signal:noise ratio.
Sample-specific MDL reported

N = QC value outside of accuracy or precision MQO

Not Surrogate Corrected

Analyzed by Krahforst, Kerylynn
8/19/2003

Main: T03-0539MS-NYD_PAH-Final rev 4 FINAL

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Project Client: PPB Environmental Laboratories
Project Name: Ponce Harbor
Project Number: N005561

Client ID	E-PH03-SC-A	E-PH03-SC-B	E-PH03-SC-C	E-PH03-SC-D	Composite worms
Battelle ID	T2712	T2713	T2714	T2715	T4581
Sample Type	SA	SA	SA	SA	SA
Collection Date	05/29/03	05/29/03	05/29/03	05/29/03	06/16/03
Extraction Date	06/23/03	06/23/03	06/23/03	06/23/03	06/23/03
Analysis Date	06/28/03	06/28/03	06/28/03	06/28/03	06/29/03
Analytical Instrument	MS	MS	MS	MS	MS
% Moisture	93.91	91.16	89.98	89.9	91.13
% Lipid	0.52	0.73	0.95	1.08	0.77
Matrix	Tissue (Nereis)	Tissue (Nereis)	Tissue (Nereis)	Tissue (Nereis)	Tissue (Nereis)
Samp_WGT_VOL	31.21	30.24	30.54	32.01	30.26
Samp_WGT_VOL_UNIT	G_WET	G_WET	G_WET	G_WET	G_WET
Units	NG/G_WET	NG/G_WET	NG/G_WET	NG/G_WET	NG/G_WET
1,4-Dichlorobenzene	0.27	0.34	0.38	0.45	0.33
Naphthalene	0.96	1.29	2.02	2.32	1.50
Biphenyl	0.16	0.21	0.31	0.26	0.17
Acenaphthylene	0.04	0.08	0.12	0.14	0.09
Acenaphthene	0.11	0.11	0.34	0.41	0.21
Fluorene	0.13	0.15	2.52	0.27	0.17
Phenanthrene	0.73	0.85	8.46	1.22	0.84
Anthracene	0.09	0.09	7.35	0.17	0.13
Fluoranthene	1.80	2.06	3.74	3.90	2.54
Pyrene	1.84	2.10	2.81	3.33	2.28
Benzo(a)anthracene	0.50	0.54	1.42	0.52	0.42
Chrysene	1.17	1.31	6.96	2.23	1.59
Benzo(b)fluoranthene	0.59	0.67	0.77	0.64	0.61
Benzo(k)fluoranthene	0.72	0.87	1.10	1.01	0.81
Benzo(a)pyrene	0.67	0.70	0.59	0.63	0.54
Indeno(1,2,3-cd)pyrene	0.38	0.42	0.45	0.46	0.45
Dibenz(a,h)anthracene	0.04	0.06	0.07	0.06	0.06
Benzo(g,h,i)perylene	0.52	0.54	0.65	0.68	0.59
Surrogate Recoveries (%)					
Naphthalene-d8	82	80	85	91	84
Phenanthrene-d10	77	75	79	84	77
Chrysene-d12	86	83	86	92	86

J = Analytes detected below sample-specific MDL

U = Analyte not detected at a 3:1 signal:noise ratio.
Sample-specific MDL reported

N = QC value outside of accuracy or precision MQO

Not Surrogate Corrected

Analyzed by Krahforst, Kerylynn
8/19/2003

Main: T03-0539MS-NYD_PAH-Final rev 4 FINAL

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Project Client: PPB Environmental Laboratories
Project Name: Ponce Harbor
Project Number: N005561

Client ID	ziplock bag 1	ziplock bag 2	Filter Swab
Battelle ID	T4578	T4579	T4583
Sample Type	SA	SA	SA
Collection Date	06/23/03	06/23/03	06/16/03
Extraction Date	06/23/03	06/23/03	06/23/03
Analysis Date	06/29/03	06/29/03	06/29/03
Analytical Instrument	MS	MS	MS
% Moisture	NA	NA	NA
% Lipid	NA	NA	NA
Matrix			cotton swab
Samp_WGT_VOL	1.19	1.17	1.17
Samp_WGT_VOL_UNIT	G_WET	G_WET	G_WET
Units	NG/G_WET	NG/G_WET	NG/G_WET
1,4-Dichlorobenzene	0.58 J	0.62 J	0.94
Naphthalene	1.24	1.26	1.69
Biphenyl	0.40 U	0.42	0.41 U
Acenaphthylene	0.38 U	0.39 U	0.39 U
Acenaphthene	0.67 U	0.68 U	0.68 U
Fluorene	0.48 U	0.49 U	0.49 U
Phenanthrene	2.95	3.23	3.80
Anthracene	1.01 U	1.03 U	1.03 U
Fluoranthene	2.91	2.79	3.14
Pyrene	2.29	1.84	2.50
Benzo(a)anthracene	1.01 U	1.02 U	1.02 U
Chrysene	1.02 U	1.04 U	1.04 U
Benzo(b)fluoranthene	1.23 U	1.25 U	1.25 U
Benzo(k)fluoranthene	0.81 U	0.82 U	0.82 U
Benzo(a)pyrene	1.09 U	1.11 U	1.11 U
Indeno(1,2,3-cd)pyrene	0.76 U	0.78 U	0.78 U
Dibenz(a,h)anthracene	0.70 U	0.71 U	0.71 U
Benzo(g,h,i)perylene	0.63 U	0.64 U	0.64 U
Surrogate Recoveries (%)			
Naphthalene-d8	82	86	85
Phenanthrene-d10	84	84	87
Chrysene-d12	88	88	93

J = Analytes detected below sample-specific MDL

U = Analyte not detected at a 3:1 signal:noise ratio.
Sample-specific MDL reported

N = QC value outside of accuracy or precision MQO

Not Surrogate Corrected

Analyzed by Krahforst, Kerylynn
8/19/2003

Main: T03-0539MS-NYD_PAH-Final rev 4 FINAL

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Project Client: PPB Environmental Laboratories
Project Name: Ponce Harbor
Project Number: N005561

Client ID	Composite worms	Composite worms	Composite worms			
Battelle ID	T4581	T4581DUP	T4581TRP			
Sample Type	SA	QADU	QATP			
Collection Date	06/16/03	6/16/2003	6/16/2003			
Extraction Date	06/23/03	6/23/2003	6/23/2003			
Analysis Date	06/29/03	6/29/2003 7:50	6/29/2003 9:06			
Analytical Instrument	MS	MS	MS			
% Moisture	91.13	91.13	91.13			
% Lipid	0.77	0.74	0.78			
Matrix	Tissue (Nereis)					
Samp_WGT_VOL	30.26	30.05	30.56			
Samp_WGT_VOL_UNIT	G_WET	G_WET	G_WET			
Units	NG/G_WET	NG/G_WET	NG/G_WET		RSD	Qualifier
1,4-Dichlorobenzene	0.33	0.34	0.33		1.7	
Naphthalene	1.50	1.48	1.43		2.5	
Biphenyl	0.17	0.19	0.19		6.3	
Acenaphthylene	0.09	0.08	0.08		6.9	
Acenaphthene	0.21	0.22	0.21		2.7	
Fluorene	0.17	0.17	0.16		3.5	
Phenanthrene	0.84	0.70	0.67		12.3	
Anthracene	0.13	0.08	0.07	J	34.4	n
Fluoranthene	2.54	2.32	2.32		5.3	
Pyrene	2.28	2.08	2.09		5.2	
Benzo(a)anthracene	0.42	0.34	0.35		11.8	
Chrysene	1.59	1.41	1.44		6.5	
Benzo(b)fluoranthene	0.61	0.51	0.60		9.6	
Benzo(k)fluoranthene	0.81	0.77	0.70		7.3	
Benzo(a)pyrene	0.54	0.46	0.50		8.0	
Indeno(1,2,3-cd)pyrene	0.45	0.36	0.39		11.5	
Dibenz(a,h)anthracene	0.06	0.05 J	0.05	J	10.8	
Benzo(g,h,i)perylene	0.59	0.52	0.48		10.5	

Surrogate Recoveries (%)

Naphthalene-d8	84	83	93
Phenanthrene-d10	77	77	94
Chrysene-d12	86	86	95

J = Analytes detected below sample-specific MDL

U = Analyte not detected at a 3:1 signal:noise ratio. Sample-specific MDL

N = QC value outside of accuracy or precision MQO

Not Surrogate Corrected

Analyzed by Krahforst, Kerylynn
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TRP: T03-0539MS-NYD_PAH-Final rev 4 FINAL

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Project Client: PPB Environmental Laboratories
Project Name: Ponce Harbor
Project Number: N005561

Client ID	Laboratory Control Sample			
Battelle ID	BC748LCS			
Sample Type	LCS			
Collection Date	06/23/03			
Extraction Date	06/23/03			
Analysis Date	06/28/03			
Analytical Instrument	MS			
% Moisture	NA			
% Lipid	NA			
Matrix				
Samp_WGT_VOL	1.00			
Samp_WGT_VOL_UNIT	G_WET			
Units	NG/G_WET	Target	% Recovery	Qualifier
1,4-Dichlorobenzene	186.76	250.75	74	
Naphthalene	1921.06	2501.38	77	
Biphenyl	1930.64	2507.50	77	
Acenaphthylene	1959.61	2501.00	78	
Acenaphthene	1998.72	2501.00	80	
Fluorene	2073.57	2501.25	83	
Phenanthrene	2163.86	2501.13	87	
Anthracene	2186.05	2501.00	87	
Fluoranthene	2394.21	2501.00	96	
Pyrene	2428.53	2501.00	97	
Benzo(a)anthracene	2352.06	2501.38	94	
Chrysene	2342.31	2501.25	94	
Benzo(b)fluoranthene	2194.86	2501.25	88	
Benzo(k)fluoranthene	2409.75	2500.88	96	
Benzo(a)pyrene	2157.34	2501.38	86	
Indeno(1,2,3-cd)pyrene	1957.3	2501.50	78	
Dibenz(a,h)anthracene	2260.44	2501.00	90	
Benzo(g,h,i)perylene	2240.11	2501.25	90	

Surrogate Recoveries (%)

Naphthalene-d8	81
Phenanthrene-d10	76
Chrysene-d12	90

J = Analytes detected below sample-specific MDL

U = Analyte not detected at a 3:1 signal:noise ratio. Sample-specific MDL

N = QC value outside of accuracy or precision MQO

Not Surrogate Corrected

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Project Client: PPB Environmental Laboratories
Project Name: Ponce Harbor
Project Number: N005561

Client ID	E-PH03-SC-B	E-PH03-SC-B			
Battelle ID	T2713	T2713MS			
Sample Type	SA	MS			
Collection Date	05/29/03	5/29/2003			
Extraction Date	06/23/03	6/23/2003			
Analysis Date	06/28/03	6/28/2003 19:04			
Analytical Instrument	MS	MS			
% Moisture	91.16	91.16			
% Lipid	0.73	0.69			
Matrix	Tissue (Nereis)				
Samp_WGT_VOL	30.24	30.95			
Samp_WGT_VOL_UNIT	G_WET	G_WET			
Units	NG/G_WET	NG/G_WET	Target	% Recovery	Qualifier
1,4-Dichlorobenzene	0.34	6.29	8.10	73	
Naphthalene	1.29	65.7	80.82	80	
Biphenyl	0.21	66.79	81.02	82	
Acenaphthylene	0.08	68.06	80.81	84	
Acenaphthene	0.11	68.51	80.81	85	
Fluorene	0.15	70.84	80.82	87	
Phenanthrene	0.85	73	80.81	89	
Anthracene	0.09	73.31	80.81	91	
Fluoranthene	2.06	81.78	80.81	99	
Pyrene	2.1	83.15	80.81	100	
Benzo(a)anthracene	0.54	78.95	80.82	97	
Chrysene	1.31	77.04	80.82	94	
Benzo(b)fluoranthene	0.67	73.29	80.82	90	
Benzo(k)fluoranthene	0.87	78.97	80.80	97	
Benzo(a)pyrene	0.7	69.2	80.82	85	
Indeno(1,2,3-cd)pyrene	0.42	63.5	80.82	78	
Dibenz(a,h)anthracene	0.06	71.74	80.81	89	
Benzo(g,h,i)perylene	0.54	72.74	80.82	89	

Surrogate Recoveries (%)

Naphthalene-d8	80	83
Phenanthrene-d10	75	78
Chrysene-d12	83	90

J = Analytes detected below sample-specific MDL

U = Analyte not detected at a 3:1 signal:noise ratio. Sample-specific MDL

N = QC value outside of accuracy or precision MQO

Not Surrogate Corrected

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MS: T03-0539MS-NYD_PAH-Final rev 4 FINAL

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Project Client: PPB Environmental Laboratories
Project Name: Ponce Harbor
Project Number: N005561

Client ID	020814-01: 1974a Mussel Tissue				
Battelle ID	BC749SRM				
Sample Type	SRM				
Collection Date	06/23/03				
Extraction Date	06/23/03				
Analysis Date	06/28/03				
Analytical Instrument	MS				
% Moisture	89.4				
% Lipid	NA				
Matrix					
Samp_WGT_VOL	5.69				
Samp_WGT_VOL_UNIT	G_WET				
Units	NG/G_WET		Target	% Difference	Qualifier
1,4-Dichlorobenzene	0.65				
Naphthalene	1.1		2.68	59.0	N
Biphenyl	0.13	J			
Acenaphthylene	0.18				
Acenaphthene	0.38				
Fluorene	0.4				
Phenanthrene	2.07		2.53	18.2	
Anthracene	0.24	J	0.69	65.2	n
Fluoranthene	18.09		18.60	2.7	
Pyrene	17.07		17.26	1.1	
Benzo(a)anthracene	2.31		3.71	37.7	
Chrysene	8.29		10.81	23.3	
Benzo(b)fluoranthene	2.28		5.28	56.8	N
Benzo(k)fluoranthene	2.05		4.63	55.7	N
Benzo(a)pyrene	1.08		1.78	39.3	n
Indeno(1,2,3-cd)pyrene	0.75		1.62	53.7	n
Dibenz(a,h)anthracene	0.24	U			
Benzo(g,h,i)perylene	1.34		2.50	46.4	N

Surrogate Recoveries (%)

Naphthalene-d8	77
Phenanthrene-d10	76
Chrysene-d12	85

J = Analytes detected below sample-specific MDL

U = Analyte not detected at a 3:1 signal:noise ratio. Sample-specific MDL

N = QC value outside of accuracy or precision MQO

Surrogate Corrected

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Glossary of Data Qualifiers

Flag:	Application:
B	Amount found in Blank Outside of Acceptable Limits.
D	Reported from dilution. Initial run outside linear calibration range of instrument.
E	Estimate, result is greater than the highest concentration level in the calibration.
H	Surrogate diluted out. Used when surrogate recovery is affected by excessive dilution of the sample extract.
J	Analyte detected below the sample-specific Reporting Limit (RL).
ME	Significant Matrix Interference - value reported is estimated.
MI	Significant Matrix Interference - value could not be determined or estimated.
n	Quality Control (QC) value is outside the accuracy or precision Data Quality Objective (DQO), but meets the contingency criteria.
N	Quality Control (QC) value is outside the accuracy or precision Data Quality Objective (DQO)
NA	Not applicable
T	Holding Time (HT) exceeded.
U	Analyte not detected at 3:1 signal:noise ratio. The sample-specific method detection limit (MDL) reported.

PAH – TISSUE QA/QC SUMMARY
Batch 03-0540

PROJECT: Ponce Harbor
PARAMETER: PAH
LABORATORY: Battelle, Duxbury, MA
MATRIX: Tissue (*Macoma nasuta*)
SAMPLE CUSTODY: The tissue samples were received at Battelle Duxbury on May 30, 2003. The cooler temperature on arrival was –2.7 °C. Tissue was stored at -20 °C until processing began. The tissues arrived frozen in plastic bags. In order to determine if the tissues were impacted by the close proximity to the plastic, the insides of two individual bags were swiped with clean, muffled glass fiber filter papers and these filter papers extracted with sample batch 03-0539. Sample ID discrepancies were noted between the sample bags and the custody records. Mactec Engineering representatives responded quickly to the request for clarification. The information is included with the custody records.

	Reference Method	Method Blank	Surrogate Recovery	LCS/MS Recovery	SRM % Diff.	Sample Replicate Relative Precision	Detection Limits (ng/g wet wt)
PAH	General NS&T	B<3xMDL	30-120% Recovery	50-120% Recovery (for at least 80% of analytes; analyte conc. in MS must be >10x background)	≤30% PD (on average; not to exceed 35% for more than 30% of analytes; measured from range of certified values, for analytes >10x MDL)	≤30% RSD	Achieved MDLs: ~ 0.03 – 0.1 Region 2 Target MDL: 4 QAPP Target MDLs: 0.02 – 0.2

METHOD: Tissue samples were extracted for PAH following general NS&T methods. Approximately 30 g of tissue was spiked with surrogates and extracted three times with dichloromethane using tissuemizer and shaker table techniques. The combined extract was dried over anhydrous sodium sulfate, concentrated, processed through alumina cleanup column, concentrated, and further purified by GPC/HPLC. The post-HPLC extract was concentrated and fortified with RIS. Extracts were analyzed for PAH using gas chromatography/mass spectrometry (GC/MS), following general NS&T methods. Sample data were quantified by the method of internal standards, using the Recovery Internal Standard (RIS) compounds.

HOLDING TIMES: Samples were prepared for analysis in one analytical batch. Extracts were analyzed within 40 days of extraction.

Batch	Extraction Date	Analysis Date
03-0540	06/17/2003	06/21/2003 – 06/22/2003

PAH – TISSUE QA/QC SUMMARY
Batch 03-0540

BLANK:

A procedural blank (PB) was prepared with each analytical batch. Blanks were analyzed to ensure the sample extraction and analysis methods were free of contamination.

03-0540 – All target analytes were recovered within the laboratory control limits specified by the client.

Comments – Two target PAH were detected in the blank, however both at levels < MDL.

LABORATORY CONTROL SAMPLE:

A laboratory control sample (LCS) was prepared with each analytical batch. The percent recoveries of target analytes were calculated to measure data quality in terms of accuracy.

03-0540 – All target analytes were recovered within the specified laboratory control limits.

Comments – None.

MATRIX SPIKE:

A matrix spike (MS) sample was prepared with each analytical batch. The percent recoveries of target analytes were calculated to measure data quality in terms of accuracy.

03-0540 – All analytes were recovered within the specified laboratory control limits.

Comments – None.

REPLICATES:

Replicate (triplicate) samples were prepared with each analytical batch. The RSD between triplicate analyses for PAH is calculated to measure data quality in terms of precision.

03-0540 – The RSD between triplicate analyses of all target analytes were within the DQO of <30% RSD.

Comments – None.

SRM:

A standard reference material (SRM, NIST 1944) was prepared with each analytical batch. The percent difference (PD) between the measured value and the certified range was calculated to measure data quality in terms of accuracy. If the detected value fell within the certified range then the PD was calculated using the nearest end of the certified range.

03-0540 – SRM PDs were within the control limits for all certified target analytes with the exception of naphthalene.

Comments – Naphthalene was slightly over-recovered with a PD of 62%. Naphthalene recoveries in the LCS and MS were acceptable. Calibration was acceptable for this analyte and no issues were noted with calculations. The certified concentration range in the SRM is less than the target detection limit for the project.

PAH – TISSUE QA/QC SUMMARY
Batch 03-0540

SURROGATES: Three surrogate compounds were added prior to extraction, including naphthalene-d8, phenanthrene-d10, and chryses-d12. The recovery of each surrogate compound was calculated to measure data quality in terms of accuracy (extraction efficiency).

03-0540 – Percent recoveries for all surrogate compounds were within the laboratory control limits specified by the method (30 – 120% recovery).

Comments – None.

CALIBRATIONS: The GC/MS is calibrated with a minimum of a 5-point curve. The RSD between response factors for the individual target analytes must be <20%. Each batch of samples analyzed is bracketed by a calibration check sample, run at a frequency of minimally every 10 samples. This PD between the initial calibration RF and the check should be <20% for individual analytes; 15% on average.

03-0540 – No calibration exceedences.

Comments – None.

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Project Client: PPB Environmental Laboratories
Project Name: Ponce Harbor
Project Number: N005561

Client ID	Procedural Blank
Battelle ID	BC750PB
Sample Type	PB
Collection Date	06/17/03
Extraction Date	06/17/03
Analysis Date	06/21/03
Analytical Instrument	MS
% Moisture	91.51
% Lipid	NA
Matrix	
Samp_WGT_VOL	30.33
Samp_WGT_VOL_UNIT	G_WET
Units	NG/G_WET
1,4-Dichlorobenzene	0.03 J
Naphthalene	0.07
Biphenyl	0.03 U
Acenaphthylene	0.02 U
Acenaphthene	0.04 U
Fluorene	0.03 U
Phenanthrene	0.03 U
Anthracene	0.07 U
Fluoranthene	0.04 U
Pyrene	0.03 U
Benzo(a)anthracene	0.07 U
Chrysene	0.07 U
Benzo(b)fluoranthene	0.08 U
Benzo(k)fluoranthene	0.05 U
Benzo(a)pyrene	0.07 U
Indeno(1,2,3-cd)pyrene	0.05 U
Dibenz(a,h)anthracene	0.05 U
Benzo(g,h,i)perylene	0.04 U

Surrogate Recoveries (%)

Naphthalene-d8	85
Phenanthrene-d10	75
Chrysene-d12	85

J = Analytes detected below sample-specific MDL

U = Analyte not detected at a 3:1 signal:noise ratio. Sample-specific MDL

N = QC value outside of accuracy or precision MQO

Not Surrogate Corrected

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Project Client: PPB Environmental Laboratories
Project Name: Ponce Harbor
Project Number: N005561

Client ID	PRETEST-M.NASUTA-A	PRETEST-M.NASUTA-B	PRETEST-M.NASUTA-C
Battelle ID	T2743	T2744	T2745
Sample Type	SA	SA	SA
Collection Date	05/29/03	05/29/03	05/29/03
Extraction Date	06/17/03	06/17/03	06/17/03
Analysis Date	06/21/03	06/21/03	06/21/03
Analytical Instrument	MS	MS	MS
% Moisture	91.77	88.89	92.7
% Lipid	0.58	0.85	0.38
Matrix	Tissue (Macoma)	Tissue (Macoma)	Tissue (Macoma)
Samp_WGT_VOL	30.12	30.26	30.68
Samp_WGT_VOL_UNIT	G_WET	G_WET	G_WET
Units	NG/G_WET	NG/G_WET	NG/G_WET
1,4-Dichlorobenzene	0.4	0.66	0.34
Naphthalene	0.77	1.23	0.63
Biphenyl	0.12	0.13	0.07
Acenaphthylene	0.03 U	0.03 U	0.03 U
Acenaphthene	0.07	0.08	0.04 J
Fluorene	0.11	0.15	0.08
Phenanthrene	0.63	0.92	0.44
Anthracene	0.05 J	0.09	0.04 J
Fluoranthene	2.66	5.19	1.86
Pyrene	1.55	2.92	1.06
Benzo(a)anthracene	0.13	0.35	0.12
Chrysene	0.72	1.28	0.56
Benzo(b)fluoranthene	0.2	0.36	0.14
Benzo(k)fluoranthene	0.11	0.32	0.1
Benzo(a)pyrene	0.09	0.16	0.1
Indeno(1,2,3-cd)pyrene	0.06 U	0.05 J	0.05 U
Dibenz(a,h)anthracene	0.05 U	0.05 U	0.05 U
Benzo(g,h,i)perylene	0.03	0.06	0.02 J
Surrogate Recoveries (%)			
Naphthalene-d8	78	80	81
Phenanthrene-d10	75	74	77
Chrysene-d12	81	79	83

J = Analytes detected below sample-specific MDL

U = Analyte not detected at a 3:1 signal:noise ratio. Sample-specific MDL

N = QC value outside of accuracy or precision MQO

Not Surrogate Corrected

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8/19/2003

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Project Client: PPB Environmental Laboratories
Project Name: Ponce Harbor
Project Number: N005561

Client ID	RS-PH03-A	RS-PH03-B	RS-PH03-C	RS-PH03-D	RS-PH03-E
Battelle ID	T2729	T2730	T2731	T2732	T2733
Sample Type	SA	SA	SA	SA	SA
Collection Date	05/29/03	05/29/03	05/29/03	05/29/03	05/29/03
Extraction Date	06/17/03	06/17/03	06/17/03	06/17/03	06/17/03
Analysis Date	06/21/03	06/21/03	06/21/03	06/21/03	06/21/03
Analytical Instrument	MS	MS	MS	MS	MS
% Moisture	87.57	92.52	91.22	92.28	94.17
% Lipid	0.58	0.34	0.38	0.34	0.32
Matrix	Tissue (Macoma)	Tissue (Macoma)	Tissue (Macoma)	Tissue (Macoma)	Tissue (Macoma)
Samp_WGT_VOL	30.10	30.35	30.89	30.94	30.74
Samp_WGT_VOL_UNIT	G_WET	G_WET	G_WET	G_WET	G_WET
Units	NG/G_WET	NG/G_WET	NG/G_WET	NG/G_WET	NG/G_WET
1,4-Dichlorobenzene	0.09	0.09	0.07	0.08	0.08
Naphthalene	0.51	0.31	0.31	0.31	0.31
Biphenyl	0.13	0.07	0.08	0.07	0.06
Acenaphthylene	0.03 U	0.03 U	0.03 U	0.03 U	0.03 U
Acenaphthene	0.04 J	0.05 U	0.05 U	0.05 U	0.05 U
Fluorene	0.13	0.08	0.08	0.07	0.06
Phenanthrene	0.72	0.5	0.48	0.39	0.42
Anthracene	0.04 J	0.04 J	0.03 J	0.02 J	0.07 U
Fluoranthene	2.2	1.48	1.3	0.94	1.34
Pyrene	1.55	1.13	0.9	0.81	0.93
Benzo(a)anthracene	0.21	0.14	0.11	0.09	0.14
Chrysene	0.85	0.63	0.59	0.46	0.59
Benzo(b)fluoranthene	0.24	0.22	0.15	0.18	0.18
Benzo(k)fluoranthene	0.25	0.14	0.13	0.13	0.1
Benzo(a)pyrene	0.15	0.1	0.1	0.08	0.1
Indeno(1,2,3-cd)pyrene	0.06 U	0.06 U	0.05 U	0.05 U	0.05 U
Dibenz(a,h)anthracene	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U
Benzo(g,h,i)perylene	0.05	0.04 J	0.03 J	0.03 J	0.04 J
Surrogate Recoveries (%)					
Naphthalene-d8	83	88	88	91	85
Phenanthrene-d10	76	80	81	84	78
Chrysene-d12	82	87	89	91	84

J = Analytes detected below sample-specific MDL

U = Analyte not detected at a 3:1 signal:noise ratio. Sample-specific MDL

N = QC value outside of accuracy or precision MQO

Not Surrogate Corrected

Analyzed by Krahforst, Kerylynn
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Project Client: PPB Environmental Laboratories
Project Name: Ponce Harbor
Project Number: N005561

Client ID	E-PH03-SC-A	E-PH03-SC-B	E-PH03-SC-C	E-PH03-SC-D	E-PH03-SC Composite
Battelle ID	T2734	T2735	T2736	T2737	T4582
Sample Type	SA	SA	SA	SA	SA
Collection Date	05/29/03	05/29/03	05/29/03	05/29/03	06/16/03
Extraction Date	06/17/03	06/17/03	06/17/03	06/17/03	06/17/03
Analysis Date	06/21/03	06/21/03	06/21/03	06/21/03	06/22/03
Analytical Instrument	MS	MS	MS	MS	MS
% Moisture	91.08	91.92	90.69	93.13	91.68
% Lipid	0.37	0.35	0.48	0.38	0.37
Matrix	Tissue (Macoma)	Tissue (Macoma)	Tissue (Macoma)	Tissue (Macoma)	Tissue (Macoma)
Samp_WGT_VOL	30.34	28.49	30.62	30.52	30.18
Samp_WGT_VOL_UNIT	G_WET	G_WET	G_WET	G_WET	G_WET
Units	NG/G_WET	NG/G_WET	NG/G_WET	NG/G_WET	NG/G_WET
1,4-Dichlorobenzene	0.07	0.07 J	0.08	0.06	0.06 J
Naphthalene	0.31	0.29	0.35	0.24	0.31
Biphenyl	0.08	0.07	0.1	0.06	0.08
Acenaphthylene	0.03 U	0.03 U	0.03 U	0.03 U	0.03 U
Acenaphthene	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U
Fluorene	0.06	0.06	0.08	0.06	0.07
Phenanthrene	0.38	0.39	0.4	0.32	0.37
Anthracene	0.07 U	0.08 U	0.02 J	0.07 U	0.07 U
Fluoranthene	1.16	1.09	0.96	0.84	1.3
Pyrene	0.77	0.73	0.72	0.65	0.79
Benzo(a)anthracene	0.12	0.08	0.09	0.07	0.13
Chrysene	0.59	0.48	0.56	0.41	0.56
Benzo(b)fluoranthene	0.15	0.14	0.16	0.11	0.14
Benzo(k)fluoranthene	0.13	0.09	0.11	0.11	0.12
Benzo(a)pyrene	0.09	0.09	0.08 J	0.07 J	0.06 J
Indeno(1,2,3-cd)pyrene	0.06 U	0.06 U	0.05 U	0.06 U	0.06 U
Dibenz(a,h)anthracene	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U
Benzo(g,h,i)perylene	0.02 J	0.05 U	0.03	0.04 J	0.05 U
Surrogate Recoveries (%)					
Naphthalene-d8	86	81	82	81	76
Phenanthrene-d10	79	74	77	75	70
Chrysene-d12	87	79	84	82	77

J = Analytes detected below sample-specific MDL

U = Analyte not detected at a 3:1 signal:noise ratio. Sample-specific MDL

N = QC value outside of accuracy or precision MQO

Not Surrogate Corrected

Analyzed by Krahforst, Kerylynn
8/19/2003

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Project Client: PPB Environmental Laboratories
Project Name: Ponce Harbor
Project Number: N005561

Client ID	Composite clams		Composite clams		Composite clams			
Battelle ID	T4582		T4582DUP		T4582TRP			
Sample Type	SA		QADU		QATP			
Collection Date	06/16/03		6/16/2003		6/16/2003			
Extraction Date	06/17/03		6/17/2003		6/17/2003			
Analysis Date	06/22/03		6/22/2003 2:24		6/22/2003 3:40			
Analytical Instrument	MS		MS		MS			
% Moisture	91.68		91.68		91.68			
% Lipid	0.37		0.43		0.4			
Matrix	Tissue (Macoma)							
Samp_WGT_VOL	30.18		30.49		30.51			
Samp_WGT_VOL_UNIT	G_WET		G_WET		G_WET			
Units	NG/G_WET		NG/G_WET		NG/G_WET		RSD	Qualifier
1,4-Dichlorobenzene	0.06	J	0.07		0.07		8.7	
Naphthalene	0.31		0.3		0.34		6.6	
Biphenyl	0.08		0.08		0.08		0.0	
Acenaphthylene	0.03	U	0.03	U	0.03	U	NA	
Acenaphthene	0.05	U	0.05	U	0.05	U	NA	
Fluorene	0.07		0.07		0.07		0.0	
Phenanthrene	0.37		0.39		0.4		4.0	
Anthracene	0.07	U	0.07	U	0.07	U	NA	
Fluoranthene	1.3		1.34		1.44		5.3	
Pyrene	0.79		0.85		0.87		5.0	
Benzo(a)anthracene	0.13		0.13		0.1		14.4	
Chrysene	0.56		0.61		0.59		4.3	
Benzo(b)fluoranthene	0.14		0.15		0.12		11.2	
Benzo(k)fluoranthene	0.12		0.11		0.12		4.9	
Benzo(a)pyrene	0.06	J	0.09		0.1		25.0	
Indeno(1,2,3-cd)pyrene	0.06	U	0.06	U	0.06	U	NA	
Dibenz(a,h)anthracene	0.05	U	0.05	U	0.05	U	NA	
Benzo(g,h,i)perylene	0.05	U	0.05	U	0.05	U	NA	

Surrogate Recoveries (%)

Naphthalene-d8	76	79	100
Phenanthrene-d10	70	72	102
Chrysene-d12	77	80	101

J = Analytes detected below sample-specific MDL

U = Analyte not detected at a 3:1 signal:noise ratio. Sample-specific MDL

N = QC value outside of accuracy or precision MQO

Not Surrogate Corrected

Analyzed by Krahforst, Kerylynn
8/19/2003

TRP: T03-0540MS-NYD_PAH-Final rev 3 FINAL

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Project Client: PPB Environmental Laboratories
Project Name: Ponce Harbor
Project Number: N005561

Client ID	Laboratory Control Sample			
Battelle ID	BC751LCS			
Sample Type	LCS			
Collection Date	06/17/03			
Extraction Date	06/17/03			
Analysis Date	06/21/03			
Analytical Instrument	MS			
% Moisture	NA			
% Lipid	NA			
Matrix				
Samp_WGT_VOL	1.00			
Samp_WGT_VOL_UNIT	G_WET			
Units	NG/G_WET	Target	% Recovery	Qualifier
1,4-Dichlorobenzene	211.03	250.75	84	
Naphthalene	2174.12	2501.38	87	
Biphenyl	2180.36	2507.50	87	
Acenaphthylene	2263.19	2501.00	90	
Acenaphthene	2265.95	2501.00	91	
Fluorene	2339.46	2501.25	94	
Phenanthrene	2388.2	2501.13	95	
Anthracene	2461.34	2501.00	98	
Fluoranthene	2603.07	2501.00	104	
Pyrene	2631.68	2501.00	105	
Benzo(a)anthracene	2579.27	2501.38	103	
Chrysene	2522.56	2501.25	101	
Benzo(b)fluoranthene	2405.66	2501.25	96	
Benzo(k)fluoranthene	2646.2	2500.88	106	
Benzo(a)pyrene	2398.59	2501.38	96	
Indeno(1,2,3-cd)pyrene	2329.19	2501.50	93	
Dibenz(a,h)anthracene	2568.8	2501.00	103	
Benzo(g,h,i)perylene	2470.29	2501.25	99	

Surrogate Recoveries (%)

Naphthalene-d8	88
Phenanthrene-d10	82
Chrysene-d12	95

J = Analytes detected below sample-specific MDL

U = Analyte not detected at a 3:1 signal:noise ratio. Sample-specific MDL

N = QC value outside of accuracy or precision MQO

Not Surrogate Corrected

Analyzed by Krahforst, Kerylynn
8/19/2003

LCS: T03-0540MS-NYD_PAH-Final rev 3 FINAL

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Project Client: PPB Environmental Laboratories
Project Name: Ponce Harbor
Project Number: N005561

Client ID	E-PH03-SC-B	E-PH03-SC-B		
Battelle ID	T2735	T2735MS		
Sample Type	SA	MS		
Collection Date	05/29/03	5/29/2003		
Extraction Date	06/17/03	6/17/2003		
Analysis Date	06/21/03	6/21/2003 17:28		
Analytical Instrument	MS	MS		
% Moisture	91.92	91.92		
% Lipid	0.35	0.36		
Matrix	Tissue (Macoma)			
Samp_WGT_VOL	28.49	28.89		
Samp_WGT_VOL_UNIT	G_WET	G_WET		
Units	NG/G_WET	NG/G_WET	Target	% Recovery	Qualifier
1,4-Dichlorobenzene	0.07 J	6.14	8.68	70	
Naphthalene	0.29	65.84	86.58	76	
Biphenyl	0.07	67.96	86.79	78	
Acenaphthylene	0.03 U	69.17	86.57	80	
Acenaphthene	0.05 U	70.18	86.57	81	
Fluorene	0.06	72.76	86.58	84	
Phenanthrene	0.39	74.58	86.57	86	
Anthracene	0.08 U	74.3	86.57	86	
Fluoranthene	1.09	81.43	86.57	93	
Pyrene	0.73	81.93	86.57	94	
Benzo(a)anthracene	0.08	77.77	86.58	90	
Chrysene	0.48	78.11	86.58	90	
Benzo(b)fluoranthene	0.14	73.44	86.58	85	
Benzo(k)fluoranthene	0.09	78.46	86.57	91	
Benzo(a)pyrene	0.09	70.13	86.58	81	
Indeno(1,2,3-cd)pyrene	0.06 U	61.48	86.59	71	
Dibenz(a,h)anthracene	0.05 U	71.21	86.57	82	
Benzo(g,h,i)perylene	0.05 U	72.47	86.58	84	

Surrogate Recoveries (%)

Naphthalene-d8	81	73
Phenanthrene-d10	74	72
Chrysene-d12	79	83

J = Analytes detected below sample-specific MDL

U = Analyte not detected at a 3:1 signal:noise ratio. Sample-specific MDL

N = QC value outside of accuracy or precision MQO

Not Surrogate Corrected

Analyzed by Krahforst, Kerylynn
8/19/2003

MS: T03-0540MS-NYD_PAH-Final rev 3 FINAL

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Project Client: PPB Environmental Laboratories
Project Name: Ponce Harbor
Project Number: N005561

Client ID	020814-01: 1974a Mussel Tissue				
Battelle ID	BC752SRM				
Sample Type	SRM				
Collection Date	06/17/03				
Extraction Date	06/17/03				
Analysis Date	06/21/03				
Analytical Instrument	MS				
% Moisture	89.4				
% Lipid	NA				
Matrix					
Samp_WGT_VOL	6.24				
Samp_WGT_VOL_UNIT	G_WET				
Units	NG/G_WET		Target	% Difference	Qualifier
1,4-Dichlorobenzene	0.52				
Naphthalene	1.01		2.68	62.3	N
Biphenyl	0.24				
Acenaphthylene	0.22				
Acenaphthene	0.28				
Fluorene	0.42				
Phenanthrene	1.78		2.53	29.6	
Anthracene	0.29	J	0.69	58.0	
Fluoranthene	22.15		18.60	19.1	
Pyrene	20.81		17.26	20.6	
Benzo(a)anthracene	2.59		3.71	30.2	
Chrysene	9.9		10.81	8.4	
Benzo(b)fluoranthene	3.56		5.28	32.6	
Benzo(k)fluoranthene	3.14		4.63	32.2	
Benzo(a)pyrene	2.01		1.78	12.9	
Indeno(1,2,3-cd)pyrene	1.05		1.62	35.2	
Dibenz(a,h)anthracene	0.21	J			
Benzo(g,h,i)perylene	2.07		2.50	17.2	

Surrogate Recoveries (%)

Naphthalene-d8	80
Phenanthrene-d10	79
Chrysene-d12	85

J = Analytes detected below sample-specific MDL

U = Analyte not detected at a 3:1 signal:noise ratio. Sample-specific MDL

N = QC value outside of accuracy or precision MQO

Surrogate Corrected

Analyzed by Krahforst, Kerylynn
8/19/2003

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Glossary of Data Qualifiers

Flag:	Application:
B	Amount found in Blank Outside of Acceptable Limits.
D	Reported from dilution. Initial run outside linear calibration range of instrument.
E	Estimate, result is greater than the highest concentration level in the calibration.
H	Surrogate diluted out. Used when surrogate recovery is affected by excessive dilution of the sample extract.
J	Analyte detected below the sample-specific Reporting Limit (RL).
ME	Significant Matrix Interference - value reported is estimated.
MI	Significant Matrix Interference - value could not be determined or estimated.
n	Quality Control (QC) value is outside the accuracy or precision Data Quality Objective (DQO), but meets the contingency criteria.
N	Quality Control (QC) value is outside the accuracy or precision Data Quality Objective (DQO)
NA	Not applicable
T	Holding Time (HT) exceeded.
U	Analyte not detected at 3:1 signal:noise ratio. The sample-specific method detection limit (MDL) reported.

Ponce Harbor
N005561
Lipid Data - Bligh and Dyer

Sample ID	Sample Description	% Total Lipid (wet wt)
BC880PB	Blank	0.03
T2722	Nereis virens	0.90
T2722-DUP	Nereis virens	0.72
T2722-TRIP	Nereis virens	0.83
T2745	Macoma nasuta	0.57
T2745-DUP	Macoma nasuta	0.69
T2745-TRIP	Macoma nasuta	0.52

Appendix F

Toxicity Testing Report with Bench Sheets

Final Report
Toxicity Testing Results
Ponce Harbor Puerto Rico

Prepared for:

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Project Number: 312253
June 2003

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1.0 Introduction

Ponce Harbor is located on the southern coast of Puerto Rico in Puerto Rico's second largest city, Ponce. The harbor is an inlet off the Caribbean Sea. The Ponce ODMDS, 4.5 nautical miles south of the harbor in the Caribbean Sea, is the proposed offshore disposal location.

The purpose of the project is to determine whether sediment dredged from Ponce Harbor can be disposed under open water disposal conditions in the Ponce ODMDS without causing significant adverse environmental impacts, or to determine the need for mitigation of potential adverse impacts. Toxicity testing and evaluation of the test results is Tier III, i.e., it is only required if sediments or sediment elutriates contain toxic substances at concentrations greater than levels recognized to cause little or no stress to aquatic biota. Toxicity testing is required when bulk sediments or elutriates from the proposed area to be dredged exceed screening levels, and have the potential to cause exceedances of water quality standards. The toxicity testing is designed to determine if sediments will actually cause toxicity to aquatic biota, and the extent of bioaccumulation of sediment toxicants in benthic organisms.

The toxicity testing was conducted in accordance with requirements of Section 103 of Public Law 92-532 (the Marine Protection, Research, and Sanctuaries Act of 1972); implementing regulations published by the Environmental Protection Agency (EPA) in Title 40 of the Code of Federal Regulations, Parts 220-228 (40 CFR 220-228); and guidance prepared by USACE and USEPA (1991) and USACE New York District and USEPA Region II (1992). This report addresses only the toxicity testing element of these requirements. Section 2 summarizes the procedures used, and Section 3 provides the bioassay test results.

2.0 General Procedures

A suite of bioassays was conducted in accordance with the U.S. Environmental Protection Agency, Region II and the U.S. Army Corps of Engineers, New York District, Standard Testing Manual entitled: *Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal* , Draft (December, 1992), and the QAPP for this project. Elutriate bioassays were conducted on sediments collected from the Ponce Harbor area to determine the potential impact of dissolved and suspended contaminants on organisms exposed to the elutriate after conducting an initial mixing period. The test organisms used for the elutriate tests included the inland silverside, *Menidia beryllina*, the mysid shrimp, *Mysidopsis bahia* (a crustacean), and gametes of the mussel, *Mytilus edulis*. Sediment bioassays were conducted to determine the effects of the site contaminants on the infaunal amphipod, *Leptocheirus plumulosus*[1] and the mysid shrimp, *Mysidopsis bahia*. Sediment bioaccumulation tests were also conducted with the polychaete, *Nereis virens* and the bivalve, *Macoma nasuta*. General procedures are summarized below. Specifics are further documented in the QAPP.

The test samples (one site sediment and one reference sediment) were collected March 4, 2003 and were received at the Harding ESE Toxicology Laboratory on March 11, 2003. Samples received at the Harding ESE Toxicology Laboratory were identified as RS-PH03 and E-PH03-SC. Additionally, approximately 25 gallons of site water, labeled as Site Water, was received at Harding ESE Toxicology Laboratory on March 14, 2003. Additional reference sediment (RS-PH03) was also received at Harding ESE Toxicology Laboratory on March 14, 2003. Control sediment (E-PH03-Control) was collected by Harding ESE personnel from the Atlantic Ocean near Marineland, Florida, on March 16, 2003, for use in the control exposures of the bioassay tests. The same control sediment was used for all the bioassay tests. All samples were stored in a refrigerator at $4 \pm 2^{\circ}C$ to test initiation, and unused portions of test samples were stored similarly during the testing period. Sample chain-of-custody and other traffic information are provided in Appendix A.

Prior to use in testing, sediments were thoroughly homogenized in their original containers and sifted or hand-sorted to remove any organic debris, small rocks, and indigenous organisms. Prior to initiating sediment tests, pore water ammonia concentrations were measured for the site sample, the reference sample, and the control sediment. The sediment samples were centrifuged and the porewater decanted off to obtain sufficient volume for the ammonia analyses. Ammonia porewater data are included with the whole sediment biassay data (see Section 3.2).

[1]This was a species substitution due to the lack of availability of *Ampelisca,* approved upon consultation with USEPA.

2.1 Elutriate Bioassay Procedures

Two of the test species used for the elutriate bioassays, *M. beryllina* and *M. bahia*, were received from Aquatic Research Organisms, Hampton, New Hampshire. *M. beryllina* were 10 days and *M. bahia* were 2 days old at test initiation. Gravid adult mussels, *M. edulis*, were received from Aquatic Research Organisms, Hampton, New Hampshire.

The elutriate bioassays were conducted at the Harding ESE Toxicology Laboratory from March 21 through March 25 for *M. bahia,* from March 24 through March 28 for *M. beryllina*, and from May 23 through 25 for *M. edulis*[2] with laboratory control sediment and sediment from the one sample station.

Elutriates were prepared by mixing 1 part of sediment to 4 parts of site water to achieve a sediment-to-water ratio of 1:4 by volume. The mixtures were mechanically stirred for 30 minutes at room temperature on a magnetic stirrer, with additional mixing by hand every 10 minutes. The mixtures were allowed to settle for at least 1 hour and then the supernatant was siphoned off as the 100% elutriate. Dilutions of the 100% elutriate were made to obtain the 50% and 10% elutriates on a volume-to-volume basis.

Natural filtered seawater collected from the Atlantic Ocean, near Marineland, Florida, was used as dilution and laboratory control water. The seawater was adjusted to a salinity of 30 parts per thousand (ppt) prior to use in preparing the three different elutriate concentrations for the *M. beryllina* and *M. bahia* tests. The salinity for the *M. edulis* was maintained at 30 ± 1 ppt. Five replicates each of the three elutriate concentrations (10%, 50%, and 100%) of the one site sediment, and laboratory control sediment were tested. The laboratory control water was also tested as 0% elutriate. The *M. beryllina* tests were conducted in 600-mL beakers containing 200 mL of elutriate or control solution and the *M. bahia* tests were conducted in 1 liter beakers containing 750 mL of elutriate or control solution. Twenty organisms were placed in each of the five replicate test chambers. *M. bahia* were fed sufficient brine shrimp nauplii (*Artemia* sp.) daily to prevent cannibalism. The *M. edulis* test was conducted in 250 ml beakers with five replicate test chambers. Additionally, a dummy set of one replicate per concentration was setup to record water quality measurements during the *M. edulis* elutriate test. *M. beryllina* and *M. bahia* tests were performed at a temperature of 20 ± 1°C and under ambient laboratory illumination (~740 lux). Test temperature for the *M. edulis* elutriate test was maintained at 16 ± 2°C, in a chilled waterbath, under ambient laboratory illumination.

Water quality parameters were measured daily during the 96-hour *M. bahia,* and *M. beryllina* tests, and daily during the 48 hour *M. edulis* test. Water quality parameters include dissolved

[2]This apparently delayed date allowed for timing and readiness of spawning, and for the laboratory to complete necessary preliminary testing.

oxygen, pH, temperature, and salinity. Dissolved oxygen was measured with a YSI Model 55 DO meter, temperature was measured with a VWR thermocouple, pH was measured with an Orion Model SA 290A pH meter, and salinity was measured with an Aquatic Eco-systems CL893 refractometer. All instruments were calibrated daily before use, and at the end of each day. Survival counts were performed daily and at test conclusion for the *M. beryllina* and *M. bahia* elutriate tests.

M. edulis eggs and sperm were obtained from adults by thermal shock methodology (ASTM Method E724.94). Sperm and eggs were collected in dilution water. The eggs were passed through a 75 µm screen to concentrate the eggs. A 1 ml sample of the eggs was placed on a Sedgwick-Rafter cell and counted. The egg density was adjusted to the range of 20 to 50 eggs/mL before adding sperm. Sufficient sperm cells were added to egg suspension to yield approximately 5 million sperm per mL. Fertilization was then confirmed. Approximately 1 hour after adding sperm suspension to the egg suspension, a 1 mL sample of the embryo concentration was observed and adjusted to 20 to 30 embryos per mL. Within 4 hours of fertilization, equal volumes of embryo suspension were added to each elutriate test vessel. The embryo suspension was homogeneously mixed during this procedure. The same volume of embryo suspension was added to each of several surrogate test chambers and preserved with buffered formalin to determine the initial, midpoint, and ending embryo density. Forty-eight hours after the beginning of the test, the solution in each test chamber was carefully mixed, volume reduced. Subsamples were removed and placed in a Sedgwick-Rafter cell for enumeration.

ANOVAs, 2 Sample t-Tests (or nonparametric equivalent for data which did not meet the assumptions for the parametric test) were used to compare mean survivorship or fertilization in the control sediment (100% elutriate) or the control elutriate (0% elutriate) versus the 100% elutriate from each of the Ponce Harbor site sediments. Data were first checked for normality and homogeneity of variance. If either of these assumptions was not met, the data were transformed in an attempt to normalization prior to analysis by Dunnett's procedure. When data could not be normalized by transformation, the data were analyzed by an equivalent nonparametric test. Median lethal concentration (LC_{50}) values for survival of *M. bahia* and *M. beryllina* and exposure concentration (EC_{50}) values for larval development for *M. edulis* were calculated, if necessary. The LC_{50} and EC_{50} are defined as the concentration of elutriate or reference toxicant that kills or inhibits 50% of the exposed test organisms under the specified conditions of exposure. The LC_{50} and EC_{50} values for all of the sediment elutriates with less than 50% mortality (or development) were estimated as greater than 100% in accordance with EPA guidelines (EPA/503/8-91/001).

2.2 Sediment Bioassay Procedures

Two test species were used for the sediment bioassays, *Leptocheirus plumulosus* and *M. bahia*. Juvenile *L. plumulosus* (3-5 mm in length, with no mature males) and *M. bahia* (3 days old at test initiation) were obtained from Aquatic Research Organisms, Hampton, Hew Hampshire.

The 10-day sediment test with *L. plumulosus* was conducted April 11 through April 21, 2003. The 10-day sediment test with *M. bahia* was conducted March 28 through April 7, 2003. Both tests had a daily photoperiod cycle of 16-hour light and 8-hour dark cycle under fluorescent lighting conditions (~840 lux) for the duration of the tests.

The sediment tests were conducted at the Harding ESE Toxicology Laboratory using five replicates each for the laboratory control sediment, one site sediment, and one reference sediment from the Ponce Harbor area. Test chambers were 2 quart glass jars for the *L. plumulosus* test and 1.6-liter glass Carolina bowls for the *M. bahia* test. Twenty *M. bahia* or *L. plumulosus* were loaded in each of the five replicate test chambers at test initiation. All test chambers were aerated at approximately 60-80 bubbles per minute. During testing, *M. bahia* were fed 0.2 ml per replicate of brine shrimp nauplii (*Artemia* sp.) daily to prevent cannibalism, and *L. plumulosus* were not fed for the duration of the 10-day exposure period.

Water quality parameters measured daily during the 10-day *M. bahia* and *L. plumulosus* sediment tests were dissolved oxygen (DO), pH, temperature, and salinity. Dissolved oxygen was measured with a YSI Model 55 DO meter, temperature was measured with a VWR thermocouple, pH was measured with an Orion Model SA 290A pH meter, and salinity was measured with an Aquatic Eco Systems CL893 refractometer. Total ammonia in pore water and overlying water samples was determined utilizing an Orion Model SA290A meter with an Orion 95-12 ammonia probe. Unionized ammonia was calculated. Pore water samples were collected on Day 0 from control, reference, and client sediments and total ammonia was determined. For samples with initial pore water ammonia levels >20 ppm, pore water samples were collected from dummy test replicates (approximately 15 per concentration and for *L. plumulosus* , and overlying water was collected from *M. bahia* test vessels, for the duration of the test. Test water was renewed twice a day, and vessels aerated to purge ammonia. Light intensity was measured with a lux meter. All instruments were calibrated daily before use, and at the end of each day. Survival counts for *L. plumulosus* and *M. bahia* were performed at termination of the sediment tests.

Prior to test initiation (Day 0), natural seawater (salinity of 30 ppt for *M. bahia* and 28 ppt for *L. plumulosus*) and sediments were introduced to each test chamber at a ratio of 1 part sediment to 4 parts seawater and allowed to settle overnight. The overlying water was siphoned from each of the replicate test chambers after 24 hours and new overlying water was added. Water quality parameters were measured prior to adding the test organisms. Water renewals were performed at 48-hour intervals after taking water quality measurements. Water was siphoned from the test chambers and placed in a glass beaker to ensure removal of test organisms had not occurred. Any test organisms inadvertently siphoned out were immediately returned to the appropriate test chamber. Fresh seawater was added back into the test chambers, taking care not to resuspend the sediment. Dead brine shrimp were removed from the *M. bahia* test chambers on a daily basis.

Sediment bioassay data were evaluated by a statistical comparison of mean survivorship in the sample station sediment relative to the field reference or laboratory control average survivorship using parametric procedures (EPA/600/4-89/001). Data were first checked for normality and homogeneity of variance. If either of these assumptions was not met, the data were transformed in an attempt to normalization prior to analysis by parametric procedures. When data could not be normalized by transformation, the data were analyzed by an equivalent nonparametric test.

2.3 Bioaccumulation Procedures

The polychaete, *N. virens*, and the bivalve, *M. nasuta* used in the bioaccumulation study were obtained from Aquatic Research Organisms, Hampton, New Hampshire.

The bioaccumulation tests were performed for 28 days in 10 gallon test aquaria were used for the *N. virens* and *M. nasuta* tests with each aquaria representing an exposure replicate for a total of 5 replicates for each sample. The exposure period was from April 10, 2003 through May 8, 2003 for N. virens and April 5, 2003 through May 3, 2003 for M. nasuta. The laboratory control sediment sample, site sediment sample, and reference sediment sample were tested without dilution. Approximately 5 centimeters depth of sediment were added to each replicate test chamber, and the aquaria was filled with at least 20 L natural seawater with a salinity of 30 ± 2 ppt. Prior to test initiation, *M. nasuta* were held for two to four days in natural filtered seawater (30 ppt), and fed Algae Spat Formula (Innovative Aquaculture Products Ltd., British Columbia, Canada) once or twice daily. At test initiation, 20 *N. virens* and 20 *M. nasuta* were placed in each test chamber (the two species were tested in separate tanks). During the first exposure, the test failed due to mechanical problems. The test was reinitiated as soon as adequate test organisms were received and prepped. Test organisms were not fed at any time during the testing period. Reference toxicant tests were performed on both species.

The tests were performed in a temperature-controlled room. Salt water chillers were utilized to maintain test temperature for each species: *N. virens*, 20 ± 2°C , and *M. nasuta*, 12 to 14°C. A flow-through water system was utilized. Ambient laboratory illumination was set on a timer, 16 hours light and 8 hours dark. Aeration was provided to all the test chambers at approximately 100-120 bubbles per minute.

Water quality parameters measured daily during the 28-day sediment bioaccumulation tests were dissolved oxygen (DO), pH, temperature, and salinity. Dissolved oxygen was measured with a YSI Model 55 DO meter, temperature was measured with a VWR thermocouple, pH was measured with an Orion Model SA 290A pH meter, and salinity was measured with an Aquatic Eco Systems CL893 refractometer. All instruments were calibrated daily before use, and at the end of each day. Observations were made daily for organism behavior and mortality. Survival counts were performed at test termination for all of the bioaccumulation tests.

After 28 days of exposure, survival and mortality of test organisms was determined and recorded. Test organisms from each replicate were removed from the aquaria and allowed to depurate in clean seawater for 24 hours. After depuration, organisms from each replicate were rinsed in deionized water, placed into Ziploc® bags, and stored in a freezer at -10 ± 2°C. Frozen *N. virens* and *M. nasuta* tissues were shipped to Battelle Laboratories on 05/29/03.

3.0 Results and Discussion

3.1 Elutriate Bioassay Data

Elutriate bioassay test conditions, including temperature, DO, pH, and salinity were maintained at acceptable levels throughout the testing period. The relevant laboratory raw data pertaining to the elutriate tests are provided in Appendix B for *M. bahia, M. beryllina*, and *M. edulis*.

Mysidopsis bahia

M. bahia survivorship was 93% in the laboratory control seawater. Survivorship in the laboratory control sediment (Table 1) was 96%, 95%, and 97% in the 10 percent, 50 percent, and 100 percent concentrations, respectively. Survivorship of *M. bahia* in the one site sediment (E-PH03-SC) was 97%, 95%, and 87% in the 10 percent, 50 percent, and 100 percent concentrations, respectively. Surviving *M. bahia* appeared healthy at test termination. *M. bahia* survivorship among replicates was relatively uniform (Appendix B).

The 100% elutriate survival dataset was found to be both normal and with homogeneity of variance. Analysis of variance followed by Dunnett's comparison indicated that the survival of *M. bahia* in the laboratory control seawater was not significantly different from either the 100% control sediment elutriate exposure or the site sample (Table 2). Results for the 2 sample t-Test between the 100% elutriate exposures for the control sediment and the site sample showed a statistically significant difference between these two samples (Table 2). LC_{50}s were >100% (Table 7).

Menidia beryllina

M. beryllina survivorship was 98% in the laboratory control seawater. Survivorship in the laboratory control sediment (Table 3) was 97%, 96%, and 95% in the 10 percent, 50 percent, and 100 percent concentrations, respectively. Survivorship of *M. beryllina* in the one site sediment (E-PH03-SC) was 94%, 85%, and 72% in the 10 percent, 50 percent, and 100 percent concentrations, respectively. Surviving *M. beryllina* appeared healthy at test termination. *M. beryllina* survivorship among replicates was relatively uniform, except in sample E-PH03-SC which exhibited some variance timing replicates (Appendix B).

This 100% elutriate survival dataset did not meet the assumptions for a parametric test, and a suitable transformation was not found. The non parametric Kruskal-Wallis comparison showed a statistically significant difference between the survival in the control seawater and the 100% elutriate of E-PH03-SC (Table 4). With the removal of the control seawater data, the dataset met the assumptions for a parametric comparison and a t-Test was conducted. Survival in sample E-PH03-SC was found to be statistically different from the 100% control sediment elutriate survival in this comparison (Table 4). LC_{50}s were >100% (Table 7).

Mytilus edulis

M. edulis development (expressed as percentage of embryos resulting in live larvae with complete shell development) was 75% in the laboratory control seawater, with acceptable survivorship (Table 5, Appendix B). Development in the laboratory control sediment (Table 5) was 72%, 71%, and 69% in the 10 percent, 50 percent, and 100 percent concentrations, respectively. Develpment of *M. edulis* in the one site sediment (E-PH03-SC) was 72%, 70%, and 60% in the 10 percent, 50 percent, and 100 percent concentrations, respectively. *M. edulis* development among replicates was relatively uniform (Appendix B).

The 100% elutriate development dataset was found to be both normal and with homogeneity of variance. Analysis of variance followed by Dunnett's comparison indicated that the development of *M. edulis* in the laboratory control seawater was significantly different from both the 100% control sediment elutriate exposure or the site sample (Table 6). Results for the 2 sample t-Test between the 100% elutriate exposures for the control sediment and the site sample showed a statistically significant difference between these two samples (Table 6). EC_{50}s were estimated as >100% (Table 7).

Reference Toxicant Tests

Concurrent reference toxicant tests were conducted to determine the general health of each test species. The reference toxicant for the *M. bahia* and *M. beryllina* tests was sodium dodecyl sulfate (SDS) with a test duration of 48 hours. The reference toxicant for the *M. edulis* test was copper sulfate (CuSO4) with a duration of 48 hours and was performed concurrently with the fertilization tests. The 48-hour LC50 result for *M. bahia* was 8.91 mg SDS/L (95% confidence limits of 6.88 to 11.53 mg SDS/L) and for *M. beryllina* was 2.18 mg SDS/L (95% confidence limits of 1.78 and 2.66 mg SDS/L). The current LC50 values were within the upper and lower control limits (±2S) of the Harding ESE control charts running average LC50, and indicated that the test organisms were within their normal sensitivity ranges (USEPA, 1994). The reference toxicant EC_{50} for the mussel was also consistent with others performed by the laboratory. The reference toxicant data sheets and the LC50 and EC_{50} calculations for the elutriate tests are presented in Appendix B.

3.2 Sediment Bioassay Data

Sediment bioassay test conditions, including temperature, DO, pH, and salinity were maintained at acceptable levels throughout the testing period. Ammonia results are discussed below. The relevant laboratory raw data pertaining to the sediment tests are provided in Appendix C for *M. bahia* and *L. plumulosus*.

Mysidopsis bahia

Pore water samples were collected on Day 0 and total ammonia (NH_3-N) values were 10.8 ppm (Control PH03), 28.8 ppm (E-PH03-SC), and 7.2 ppm (RS-PH03). As per the guidance, samples were aerated and overlying water samples were collected on Day 0 through 10 for sample E-PH03-SC, and total ammonia ranged from 7.3 ppm (Day 0) to 0.408 ppm (Day 10). Ammonia for other samples remained low throughout the test duration (Appendix C).

M. bahia survivorship was 96% in the laboratory control sediment and 92% in the reference sediment, RS-PH03 (Table 8). Survivorship of *M. bahia* in the one site sediment was 96% (Station E-PH03-SC). Surviving *M. bahia* appeared healthy at test termination. *M. bahia* survivorship among replicates was relatively uniform (Appendix C).

Statistical analysis indicated that the survival of *M. bahia* in the laboratory control sediment was not significantly different (P=0.05) from survival in the Ponce Harbor sample or reference sample (Table 9). Survival of *M. bahia* in the reference sediment was not significantly different (p=0.05) from survival in the site sample (Table 9).

Leptocheirus plumulosus

Pore water samples were collected on Day 0 and total ammonia (NH_3-N) values were 7.3 ppm (Control PH03), 38.0 ppm (E-PH03-SC), and 7.5 ppm (RS-PH03). Overlying waters were vigorously aerated to reduce the ammonia to less that 20 ppm prior to initiation of testing. Additional replicates ("dummy" replicates, without organisms) of the site sediment were assembled for porewater monitoring throughout the test duration. Total ammonia in pore water samples ranged from 23.2 ppm (Day 0) to 2.92 ppm (Day 10).

L. plumulosus survivorship was 95% in the laboratory control sediment and 95% in the reference sediment (Table 10). Survivorship of *L. plumulosus* in the site sediment was 84% (station E-PH03-SC). It should be noted, at test termination and prior to counting of organisms, one replicate of the laboratory control was accidentally broken, and is not included in the survivorship. Surviving *L. plumulosus* appeared healthy at the termination of the tests.

Statistical analysis using Dunnett's T-test indicated that the survival of *L. plumulosus* in the laboratory control sediment was not significantly different (P=0.05) from survival in the reference sediment, but was significantly different from the Ponce Harbor survival (Table 11). However, survival of *L. plumulosus* in the reference sediment was not statistically different from the site sample (Table 11).

Reference Toxicant Tests

Reference toxicant tests were conducted on each species tested in the sediment tests. The reference toxicant used for *M. bahia* was SDS and the duration of the test was 96 hours. The

reference toxicant used for *L. plumulosus* was cadmium chloride (CdCl2), measured as Cd, for a duration of 96 hours. The 96-hour LC50 result for *M. bahia* was 4.39 mg SDS/L (95% confidence limits of 1.69 to 11.39mg SDS/L) and the 96-hour LC50 result for *L. plumulosus* was 1.93 mg Cd/L (95% confidence limits of 1.56 to 2.37 mg Cd/L). The current LC50 values were within the upper and lower control limits (±2S) of the Harding ESE control charts running average LC50, and indicated that the test organisms were within their normal sensitivity ranges (USEPA, 1994). The reference toxicant data sheets and LC50 calculations for the sediment tests are presented in Appendix C.

3.3 Bioaccumulation Data

The bioaccumulation test conditions, including temperature, DO, pH, and salinity were maintained at acceptable levels throughout the 28-day testing period. Flow through conditions were maintained with minimal interruptions to adjust flows, and on two occasions to replace parts. The laboratory raw data and reference toxicant data are provided in Appendix C. Results of the reference toxicant testing confirm that the organisms were in good health at test initiation and are consistent with results of similar testing by the laboratory (Appendix C).

Macoma nasuta

Data for the survival of *M. nasuta* in the bioaccumulation tests are presented in Table 12. *M. nasuta* survivorship in the laboratory control sediment was 92. Survival of *M. nasuta* in the site sediment was 87% (sample station E-PH03-SC) (Table 12). Survival of *M. nasuta* in the reference sediment was 94% (sample station RS-PH03) (Table 12). Adequate mass of *M. nasuta* tissue was available for chemical analyses.

Nereis virens

Data for the survival of *N. virens* in the bioaccumulation tests are also presented in Table 12. *N. virens* survivorship in the laboratory control sediment was 95%. Survival of *N. virens* in the site sediment was 88% (sample station E-PH03-SC) (Table 12). Survival of *N. virens* in the reference sediment was 95%. Adequate mass of *N. virens* tissue was available for chemical analyses in the remaining samples.

4.0 References

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Tables

Table 1. 96-Hour *Mysidopsis bahia* Survival in Three Elutriate Concentrations Prepared from Sediments Collected from Ponce Harbor, March 2003 (Page 1 of 1)

Sample	Replicate	Number of Survivors[a]
CONTROL SEAWATER	A	19
	B	18
	C	18
	D	19
	E	19
	Total	93
	Percent	93%

Sample	Replicate	Elutriate Concentration		
		10%	50%	100%
CONTROL SEDIMENT	A	19	17	19
	B	18	19	20
	C	20	20	20
	D	19	19	19
	E	20	20	19
	Total	96	95	97
	Percent	96%	95%	97%
E-PH03-SC	A	19	19	19
	B	19	20	16
	C	20	19	19
	D	20	18	17
	E	19	19	16
	Total	97	95	87
	Percent	97%	95%	87%

a - Based upon 100 organisms exposed.

Source: Harding ESE.

Table 2. Summary of ANOVA and Dunnett's Tests of Control Seawater and ANOVA and 2 Sample t-Test for Control Sediment and Test Sediment Survival for Ponce Harbor Elutriate of *Mysidopsis bahia*, April 2003 (Page 1 of 2)

Mysidopsis bahia 100% Elutriates vs. Control Seawater

ANOVA for Differences Between Means

Source of Variation	df	Sum of Squares	Mean Square	F
Between Means	2	10.13	5.07	5.24
Within Means	12	11.60	0.97	
Total	14	21.73		

Critical F = 3.89 (a = 0.05, df = 2,12)
Since F is > Critical F reject H_o: all groups equal;
with a = 0.05, and 2,12 df.

Dunnett's Test

Critical value = 2.11

Sample ID	a = 0.05	difference between means	T Stat
CONTROL SEAWATER	–	–	–
E-PH03-CONTROL	=	-0.80	-1.29
E-PH03-SC	=	1.20	1.93

= Indicates no significant difference between the sample station and the control seawater
* Indicates a significant difference exists between the sample station and the control seawater

Table 2. Summary of ANOVA and Dunnett's Tests of Control Seawater and ANOVA and 2 Sample t-Test for Control Sediment and Test Sediment Survival for Ponce Harbor Elutriate of *Mysidopsis bahia*, April 2003 (Page 2 of 2)

Mysidopsis bahia 100% Elutriates vs. Control Sediment

ANOVA for Differences Between Means

Source of Variation	df	Sum of Squares	Mean Square	F
Between Means	1	10.00	10.00	7.69
Within Means	8	10.40	1.30	
Total	9	20.40		

Critical F = 5.32 (a = 0.05, df = 1,8)
Since F is > Critical F reject H_o: all groups equal;
with a = 0.05, and 1,8 df.

2 Sample t-Test

Critical T = 1.86

Sample ID	a = 0.05	difference between means	T Stat
E-PH03-CONTROL		-	-
E-PH03-SC	*	2.00	2.77

= Indicates no significant difference between the sample station and the reference sediment
* Indicates a significant difference exists between the sample station and the reference sediment

Table 3. 96-Hour *Menidia beryllina* Survival in Three Elutriate Concentrations Prepared from Sediments Collected from Ponce Harbor, Puerto Rico, March 2003 (Page 1 of 1)

Sample	Replicate	Number of Survivors[a]
CONTROL SEAWATER	A	20
	B	20
	C	20
	D	20
	E	19
	Total	99
	Percent	99%

Sample	Replicate	Elutriate Concentration		
		10%	50%	100%
E-PH03-CONTROL SEDIMENT	A	19	20	19
	B	20	20	20
	C	19	20	18
	D	20	19	19
	E	19	17	19
	Total	97	96	95
	Percent	97%	96%	95%
E-PH03-SC	A	19	12	14
	B	19	19	16
	C	18	16	18
	D	19	18	14
	E	19	20	10
	Total	94	85	72
	Percent	94%	85%	72%

a - Based upon 100 organisms exposed.

Source: Harding ESE, Inc., 2003

Table 4. Summary of Kruskal – Wallis ANOVA by Ranks of Control Seawater and ANOVA with 2 Sample t-Test for Control Sediment and Test Sediment Survival for Ponce Harbor Elutriate of *Menidia beryllina*, April 2003 (Page 1 of 2)

Menidia beryllina 100% Elutriate vs. Control Seawater

Kruskal – Wallis ANOVA by Ranks

Sample	Transformed Mean	Rank Sum
Control Seawater	0.0505	19.50
E-PH03-SC	0.0722	64.50
E-PH03-Control	0.0527	36.00

Critical H = 5.78 (a = 0.05, df = 2,)
Since Calc H is > Critical H reject H_o: all groups equal;
with a = 0.05, and 2 df.

Dunn's Multiple Comparison

Table q value = 2.3940

Sample ID	a = 0.05
CONTROL SEAWATER	=
E-PH03-SC	*
E-PH03-CONTROL	=

= Indicates no significant difference between the sample station and the control seawater
* Indicates a significant difference exists between the sample station and the control seawater

Table 4. Summary of Kruskal – Wallis ANOVA by Ranks of Control Seawater and ANOVA with 2 Sample t-Test for Control Sediment and Test Sediment Survival for Ponce Harbor Elutriate of *Menidia beryllina*, April 2003 (Page 2 of 2)

Menidia beryllina 100% Elutriate vs. Control Sediment

ANOVA for Differences Between Means

Source of Variation	df	Sum of Squares	Mean Square	F
Between Means	1	52.90	52.90	11.38
Within Means	8	37.20	4.65	
Total	9	90.10		

Critical F = 5.32 (a = 0.05, df = 1,8)
Since F is > Critical F reject H_o: all groups equal;
with a = 0.05, and 1,8 df.

2 Sample t-Test

Critical T = 1.86

Sample ID	a = 0.05	difference between means	T Stat
E-PH03-CONTROL		-	-
E-PH03-SC	*	4.60	3.37

= Indicates no significant difference between the sample station and the reference sediment
* Indicates a significant difference exists between the sample station and the reference sediment

Table 5. 48-Hour *Mytilus edilus* Development in Three Elutriate Concentrations Prepared from Sediments Collected from Ponce Harbor, Puerto Rico, May 2003 (Page 1 of 1)

Sample	Replicate	Percentage of Full Development[a]
CONTROL SEAWATER	A	72
	B	80
	C	79
	D	71
	E	75
	Total Percent	75%

Sample	Replicate	Elutriate Concentration		
		10%	50%	100%
E-PH03-CONTROL	A	70	75	68
	B	66	81	62
	C	80	62	77
	D	72	70	67
	E	71	69	70
	Total Percent	72%	71%	69%
E-PH03-SC	A	76	73	60
	B	62	78	62
	C	75	63	58
	D	68	68	67
	E	80	68	54
	Total Percent	72%	70%	60%

a – Expressed as percentage of mussel embryos resulting in live larvae with complete shell development.

Source: Harding ESE

Table 6. Summary of ANOVA and Dunnett's Tests of Control Seawater and ANOVA and 2 Sample t-Test for Control Sediment and Test Sediment Larval Development for Ponce Harbor Elutriate of *Mytilus edulis*, May2003 (Page 1 of 2)

M. edulis 100% Elutriates vs. Control Seawater

ANOVA for Differences Between Means

Source of Variation	df	Sum of Squares	Mean Square	F
Between Means	2	580.93	290.47	12.59
Within Means	12	276.80	23.07	
Total	14	857.73		

Critical F = 3.89 (a = 0.05, df = 2,12)
Since F is > Critical F reject H_o: all groups equal;
with a = 0.05, and 2,12 df.

Dunnett's Test

Critical value = 2.11

Sample ID	a = 0.05	difference between means	T Stat
CONTROL SEAWATER	–	–	–
E-PH03-CONTROL	*	6.6	2.17
E-PH03-SC	*	15.2	5.00

= Indicates no significant difference between the sample station and the control seawater
* Indicates a significant difference exists between the sample station and the control seawater

Table 6. Summary of ANOVA and Dunnett's Tests of Control Seawater and ANOVA and 2 Sample t-Test for Control Sediment and Test Sediment Larval Development for Ponce Harbor Elutriate of *Mytilus edulis*, May2003 (Page 2 of 2)

Mytilus edulis 100% Elutriates vs. Control Sediment

ANOVA for Differences Between Means

Source of Variation	df	Sum of Squares	Mean Square	F
Between Means	1	184.90	184.90	6.99
Within Means	8	211.60	26.45	
Total	9	396.50		

Critical F = 5.32 (a = 0.05, df = 1,8)
Since F is > Critical F reject H_o: all groups equal;
with a = 0.05, and 1,8 df.

2 Sample t-Test

Critical T = 1.86

Sample ID	a = 0.05	difference between means	T Stat
E-PH03-CONTROL		-	-
E-PH03-SC	*	8.60	2.64

= Indicates no significant difference between the sample station and the reference sediment
* Indicates a significant difference exists between the sample station and the reference sediment

Table 7. LC_{50} or EC_{50} (*Mysidopsis bahia*, *Menidia beryllina* and *Mytilus edulis*) Values[a] for Elutriate Bioassays Conducted for Ponce Harbor (Page 1 of 1)

Sample ID	*Mysidopsis bahia*	*Menidia beryllina*	*Mytilus edulis*
E-PH03-CONTROL	>100	>100	>100
E-PH03-SC	>100	>100	>100
RS-PH03	>100	>100	>100

[a] LC_{50} values recorded as greater than 100% had greater than 50% survivorship. In the case of *Mytilus edulis*, an EC_{50} value greater than 100% indicated development greater than 50%.

Table 8. 10-Day Sediment *Mysidopsis bahia* Survival, Ponce Harbor Sediments, April 2003 (Page 1 of 1)

Sample	Replicate	Number of Survivors[a]
E-PH03 – CONTROL	A	19
	B	18
	C	20
	D	20
	E	19
	Total	96
	Percent	96%
RS-PH03	A	17
	B	19
	C	19
	D	17
	E	20
	Total	92
	Percent	92%
E-PH03-SC	A	20
	B	20
	C	20
	D	17
	E	19
	Total	96
	Percent	96%

a - Based upon 100 organisms exposed.

Source: Harding ESE

Table 9. Summary of ANOVA, Dunnett's, and 2-Sample t-Tests of Control Sediment or Reference Sediment and Test Sediment Survival for Ponce Harbor Whole Sediment of *Mysidopsis bahia*, April 2003 (Page 1 of 2)

Mysidopsis bahia Whole Sediment vs. Control

ANOVA for Differences Between Means

Source of Variation	df	Sum of Squares	Mean Square	F
Between Means	2	2.13	1.07	0.76
Within Means	12	16.80	1.40	
Total	14	18.93		

Critical F = 3.89 (a = 0.05, df = 2,12)
Since F is < Critical F fail to reject H_o: all groups equal;
with a = 0.05, and 2,12 df.

Dunnett's Test

Critical T = 2.11

Sample ID	a = 0.05	difference between means	T Stat
E-PH03-CONTROL		–	–
E-PH03-SC	=	0.00	0.00
RS-PH03	=	0.80	1.07

= Indicates no significant difference between the sample station and the control sediment
* Indicates a significant difference exists between the sample station and the control sediment

Table 9. Summary of ANOVA, Dunnett's, and 2-Sample t-Tests of Control Sediment or Reference Sediment and Test Sediment Survival for Ponce Harbor Whole Sediment of *Mysidopsis bahia*, April 2003 (Page 2 of 2)

Mysidopsis bahia Reference Sediment vs. Control

ANOVA for Differences Between Means

Source of Variation	df	Sum of Squares	Mean Square	F
Between Means	1	1.60	1.60	0.91
Within Means	8	14.00	1.75	
Total	9	15.60		

Critical F = 5.32 (a = 0.05, df = 1,8)
Since F is < Critical F fail to reject H_o: all groups equal;
with a = 0.05, and 1,8 df.

2 Sample t-Test

Critical T = 1.86

Sample ID	a = 0.05	difference between means	T Stat
RS-PH03		–	–
E-PH03-SC	=	-0.80	-0.96

= Indicates no significant difference between the sample station and the reference sediment
* Indicates a significant difference exists between the sample station and the reference sediment

Table 10. 10- Day Sediment *Leptocheirus plumulosus* Survival, Ponce Harbor Sediments, April 2003 (Page 1 of 1)

Sample	Replicate	Number of Survivors[a]
PH03 - CONTROL	A	19
	B	17
	C	-
	D	20
	E	20
	Total	76
	Percent	95%
RS-PH03	A	18
	B	19
	C	20
	D	19
	E	19
	Total	95
	Percent	95%
E-PH03- SC	A	16
	B	16
	C	17
	D	17
	E	18
	Total	84
	Percent	84%

a - Based upon 100 organisms exposed, except sample PH03-Control, based on 80 organisms exposed as Replicate C test vessel was broken during testing.

SOURCE: Harding ESE 2003

Table 11. Summary of ANOVA, Bonferroni, and 2 Sample t-Tests of Control Sediment or Reference Sediment and Test Sediment Survival for Ponce Harbor Whole Sediment of *L. plumulosus*, April 2003 (Page 1 of 2)

L. plumulosus Whole Sediment vs. Control

ANOVA for Differences Between Means

Source of Variation	df	Sum of Squares	Mean Square	F
Between Means	2	15.56	7.78	7.92
Within Means	11	10.80	0.98	
Total	13	26.36		

Critical F = 3.98 (a = 0.05, df = 2,11)
Since F is > Critical F reject H_o: all groups equal;
with a = 0.05, and 2,11 df.

Bonferroni Test

Critical T = 2.20

Sample ID	a = 0.05	difference between means	T Stat
E-PH03-CONTROL		–	–
E-PH03-SC	*	2.20	3.31
RS-PH03	=	–	–

= Indicates no significant difference between the sample station and the control sediment
* Indicates a significant difference exists between the sample station and the control sediment

Table 11. Summary of ANOVA, Bonferroni, and 2 Sample t-Tests of Control Sediment or Reference Sediment and Test Sediment Survival for Ponce Harbor Whole Sediment of *L. plumulosus*, April 2003 (Page 2 of 2)

L. plumulosus Reference Sediment vs. Control

ANOVA for Differences Between Means

Source of Variation	df	Sum of Squares	Mean Square	F
Between Means	1	12.10	12.10	20.17
Within Means	8	4.80	0.60	
Total	9	16.90		

Critical F = 5.32 (a = 0.01, df = 1,8)
Since F is > Critical F reject H_o: all groups equal;
with a = 0.05, and 1,8 df.

2 Sample t-Test

Critical T = 2.43

Sample ID	a = 0.05	difference between means	T Stat
RS-PH03	=	–	–
E-PH03-SC	*	2.20	4.49

= Indicates no significant difference between the sample station and the reference sediment
* Indicates a significant difference exists between the sample station and the reference sediment

Table 12. Survivorship of *Macoma nasuta* and *Nereis virens* during 28-Day Bioaccumulation Bioassays with Sediments from Ponce Harbor, May 2003 (Page 1 of 1)

Sample ID	Replicate	*Macoma nasuta*[a]	*Nereis virens*[a]
E-PH03 -Control Sediment	A	18	20
	B	18	19
	C	19	18
	D	19	18
	E	18	20
	Total	92	95
	Percent	92%	95%
RS-PH03A	18	20	
	B	18	19
	C	20	20
	D	19	17
	E	19	19
	Total	94	95
	Percent	94%	95%
E-PH03-SC	A	16	16
	B	18	20
	C	18	16
	D	19	18
	E	18	17
	F	16	20
	G	17	17
	H	18	16
	I	16	18
	Total	156	158
	Percent	87%	88%

a – based upon 100 organisms, except for sample E-PH03-SC for which extra replicates were exposed to ensure sufficient tissue for triplicates, spikes, etc.

Deviations/Qualifiers Ponce Harbor Study

ELUTRIATE STUDIES

M. beryllina
Reference Toxicant Test
Date: 3/27/03, Day 3
Re: Control, 1.25 and 2.50 concentrations. Salinity not recorded on Day 3. Salinity was 19 and 20 ppt on Day 2, and 19 ppt on Day 4 for these concentrations, therefore we believe the salinity remained within range on Day 3. Salinity for all other concentrations recorded on 3/27/03.

M. beryllina
96-hour Test
Date: 3/28/03, Day 4 (last day).
Re: Control sediment, 50% concentration, Replicate E. Dissolved oxygen recorded at 4.3 mg/L. This occurred on final day of test, and did not appear to have an impact on organism survival.

M. bahia
96-hr Test.
Date: 3/21/03, Day 0.
Re: Concentrations 5, 10, 20, and 40. Temperature recorded as 21.5, 21.5, 21.6, and 21.6 °C, respectively. Guidance requires temperature range of 19 to 21 °C. This minor deviation did not appear to have an effect on survival. Temperature for the remaining days of the test remained within range.

M. edulis
Day 0; seawater control temperature of 17.5.

WHOLE SEDIMENT STUDIES

M. bahia
10-day Test.
Date: 4/7/03, Day 10.
Re: Replicate A, sample E-PH03-SC. Dissolved oxygen recorded at 4.5 mg/L.
This occurred on final day of test, and did not appear to have an impact on organism survival.

Substituted *Leptocheirus* for *Ampelisca* following consultation with USEPA 2

BIOACCUMULATION STUDIES

Both test species restarted due to control mortality (consulted USEPA II).

Appendix A

ppb
ENVIRONMENTAL LABORATORIES, INC.
6821 SW Archer Road, Gainesville, FL 32608
(352) 377-2349 · FAX (352) 395-6639

CHAIN-OF-CUSTODY RECORD

LABTROL CLIENT No.	BATCH NO.	FOR LAB USE ONLY
		Subcontractor
		Shipping Method

CLIENT NAME: AFI – Ponce

CLIENT PROJECT: Ponce Harbor

SITE NAME & ADDRESS: Ponce, Puerto Rico

SAMPLERS: *(Signature)* CSA

LAB REPORT GOES TO (Client contact person):

IDENTIFY PARAMETERS DESIRED AND NO. OF CONTAINERS: As per Contract

PRESERVATION

CF	Chilled-Filtered
SF	Sulfuric-Filtered
NF	Nitric-Filtered
C	Chilled
S	Sulfuric
N	Nitric
B	Basic/NaOH
Z	Zinc
T	Thiosulfate
H	HCL
Ot	Other (see Remarks)

NUMBER	DATE	TIME	COMP.	GRAB	STATION LOCATION / NUMBER	SAMPLE MATRIX	NUMBER OF CONTAINERS	As per Contract							Lab I.D. Number
			✓		RS-PH03 (extra)	S	2	✓							
				✓	Site Water	W	12	✓							

Precleaned Containers Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Date / Time
		BC	3/14/03
Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Date / Time
[illegible]	3/14/03 1500	Craig [illegible]	3/14/03 1500
Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Date / Time
Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Date / Time

Remarks and Observations

Matrix Types S Soil or Sediment WW Wastewater T Animal Tissue SW Surface Water SL Sludge or Solid Waste MW Marine Water GW Ground Water DW Drinking Water P Plant Tissue F Filter A Air Ot Other (See Remarks)

ppb
ENVIRONMENTAL LABORATORIES, INC.
6821 SW Archer Road, Gainesville, FL 32608
(352) 377-2349 · FAX (352) 395-6639

CHAIN-OF-CUSTODY RECORD

LABTROL CLIENT No.	BATCH NO.	FOR LAB USE ONLY
		Subcontractor
		Shipping Method

CLIENT NAME: AFI - Ponce Harbor

CLIENT PROJECT: Ponce Harbor

SITE NAME & ADDRESS: Ponce, Puerto Rico

SAMPLERS: (Signature) CSA/PPB

LAB REPORT GOES TO (Client contact person):

IDENTIFY PARAMETERS DESIRED AND NO. OF CONTAINERS: As per contract

PRESERVATION: CF Chilled-Filtered; SF Sulfuric-Filtered; NF Nitric-Filtered; C Chilled; S Sulfuric; N Nitric; B Basic/NaOH; Z Zinc; T Thiosulfate; H HCL; Ot Other (see Remarks)

NUMBER	DATE	TIME	COMP.	GRAB	STATION LOCATION / NUMBER	SAMPLE MATRIX	NUMBER OF ~~CONTAINERS~~ Coolers	Lab I.D. Number
	3/4/03	1257	✓		RS-PH03	S	8	
	3/4/03	1233	✓		E-PH03-SC	S	5	

Precleaned Containers Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Date / Time
[signature]	3/10/03 1200	[signature]	
Relinquished by: (Signature) [signature]	Date / Time 3/11/03 ~~1000~~ 1145	Received by: (Signature) [signature]	Date / Time 3/11/03 1145
Relinquished by: (Signature)	Date / Time 3/11/03	Received by: (Signature)	Date / Time
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Date / Time

Remarks and Observations

Note: Actually receipt, 5 coolers of RS-PHØ3, and 8 coolers of E-PHØ3-SC. [signature] 3/11/03

Matrix Types S Soil or Sediment; WW Wastewater; T Animal Tissue; SW Surface Water; SL Sludge or Solid Waste; MW Marine Water; GW Ground Water; DW Drinking Water; P Plant Tissue; F Filter; A Air; Ot Other (See Remarks)

MACTEC Engineering and Consulting, Inc. (formerly Harding ESE)
Toxicology Laboratory
Gainesville, Florida

QA Form Number: 063
Revised: Jan2003

SAMPLE CHAIN OF CUSTODY

Facility: Mactec

Location: Gainesville, FL

Permit Number: N/A Outfall Number: N/A

SAMPLE IDENTIFICATION

	Sample Identification	Sample Type Grab	Sample Type Comp	Date	Time	Observations
1	CH 03 - Ctrl - Sed	✓		4/16/03		
2	CH 03 - ctrl - Sed	✓		4/16/03		
3	CH 03 - ctrl - Sed	✓		4/16/03		
Total Number of Samples:		3				

SAMPLE INFORMATION

Sampler(s): Craig Godman

Sampling Point (Description): St. Augustine

Water Quality Measurements (optional): —

Temp: pH: Diss. Oxygen: Salinity/Cond.: Chlorine (TRC):

Water Quality Measured by: Date: Time:

Observations/Remarks:

CHAIN OF CUSTODY

Relinquished By:	Received By:	Date	Time
[signature]	[signature]	4-21-03	1602

MACTEC Engineering and Consulting, Inc. (formerly Harding ESE)
Toxicology Laboratory
Gainesville, Florida

QA Form Number: 063
Revised: Jan2003

SAMPLE CHAIN OF CUSTODY

Facility: Toxicology lab

Location:

Permit Number: Outfall Number:

SAMPLE IDENTIFICATION

Sample Identification	Sample Type Grab	Sample Type Comp	Date	Time	Observations
Cooler 013	✓		03/9/03	10:00	Sediment Ctrl
" 014	✓		"	"	Sediment Ctrl
~~" 015~~	~~JA ✓~~		~~"~~	~~"~~	~~JA~~
	✓				
Total Number of Samples:	~~3~~ 2 ~~JA~~				

SAMPLE INFORMATION

Sampler(s): James H. Ares

Sampling Point (Description): Marineland

Water Quality Measurements (optional):

Temp: 20.1 pH: Diss. Oxygen: Salinity/Cond.: Chlorine (TRC):

Water Quality Measured by: JA Date: 03/9/03 Time: 10:00

Observations/Remarks:

CHAIN OF CUSTODY

Relinquished By:	Received By:	Date	Time
	James H. Ares	03/9/03	11:00
James H. Ares	Craig [illegible]	3/9/03	1400

MACTEC Engineering and Consulting, Inc. (formerly Harding ESE)
Toxicology Laboratory
Gainesville, Florida

QA Form Number: 063
Revised: Jan2003

SAMPLE CHAIN OF CUSTODY

Facility: Toxicology lab

Location:

Permit Number: Outfall Number:

SAMPLE IDENTIFICATION

Sample Identification	Sample Type Grab	Sample Type Comp	Date	Time	Observations
Cooler 007	✓		3/9/03	10:00	Control Sediment
" 008	✓		"	"	
" 009	✓		"	"	
" 010	✓		"	"	
" 011	✓		"	"	
" 012	✓		"	"	
Total Number of Samples:	6				

SAMPLE INFORMATION

Sampler(s): James H. Ares

Sampling Point (Description): Marine land

Water Quality Measurements (optional):

Temp: 20.1 pH: Diss. Oxygen: Salinity/Cond.: Chlorine (TRC):

Water Quality Measured by: JA Date: 3/9/03 Time: 10:00

Observations/Remarks:

CHAIN OF CUSTODY

Relinquished By:	Received By:	Date	Time
	James H. Ares	03/9/03	11:00
James H. Ares	Craig [illegible]	3/9/03	1400

MACTEC Engineering and Consulting, Inc. (formerly Harding ESE)
Toxicology Laboratory
Gainesville, Florida

QA Form Number: 063
Revised: Jan2003

SAMPLE CHAIN OF CUSTODY

Facility: Toxicology lab

Location:

Permit Number: Outfall Number:

SAMPLE IDENTIFICATION

Sample Identification	Sample Type Grab	Sample Type Comp	Date	Time	Observations
cooler 001	✓		03/09/03	10:00	Control Sediment
cooler 002	✓		"	"	
" 003	✓		"	"	
" 004	✓		"	"	
" 005	✓		"	"	
" 006 JA	✓		"	"	
Total Number of Samples:	6				

SAMPLE INFORMATION

Sampler(s): James H. Ares

Sampling Point (Description): Marineland

Water Quality Measurements (optional):

Temp: 20.1 pH: Diss. Oxygen: Salinity/Cond.: Chlorine (TRC):

Water Quality Measured by: JA Date: 3/9/03 Time: 10:00

Observations/Remarks:

CHAIN OF CUSTODY

Relinquished By:	Received By:	Date	Time
	James H Ares	03/9/03	11:00
James H Ares	Craig [illegible]	3/9/03	1400

Carbon copy

MACTEC Engineering and Consulting, Inc. (formerly Harding ESE)
Toxicology Laboratory
Gainesville, Florida

QA Form Number: 063COCt
Revised: Jan2003

SAMPLE CHAIN OF CUSTODY for TISSUE SAMPLES

Facility: MACTEC Engineering and Consulting, Inc.

Location: 404 SW 140th Terr., Newberry, FL 32669

Other: Phone (352)332-3318

SAMPLE IDENTIFICATION

Sample Identification	Organism Genus species	#	Replicate Bags	Observations
Ctrl-PHØ3	N. virens	5	A - E	
RFS-PHØ3	N. virens	5	A - E	
E-PHØ3-SC	N. virens	9	A - I	
Pretest N. virens	N. virens	3	3 bags	
Ctrl-PHØ3	M. nasuta	5	A - E	
RFS-PHØ3	M. nasuta	5	A - E	
E-PHØ3-SC	M. nasuta	9	A - I	
Pretest M. nasuta	M. nasuta	3	3 bags	

Total Number of Samples: ~~42~~ 44 individual bags

SAMPLE INFORMATION

Toxicity Test Type: Bioaccumulation test

Exposed to Sediment from: Ponce Harbor, PR

Observations/Remarks: Replicate bags packed inside larger ziploc bags

CHAIN OF CUSTODY

Relinquished By:	Received By:	Date	Time
Jerry M. Ryan		1500	~~5/28/03~~ 5/29/03 JMRyan

Appendix B

MACTEC Engineering & Consulting, Inc.
Toxicology Laboratory
Gainesville, Florida

Page: 1 of 1
QA Form No. 018
Revised: October 2002

Project No: 312253.0300 Client: Ponce Harbor/ PPB Test: Elutriate, M. bahia

DAILY LOG

3/21/03. elutriates prepared by mixing 1 part sediment with 4 parts site water for 30 minutes. Mixture allowed to settle for 1 hour and the overlying water siphoned off. Dilutions were made and loaded into test vessels. Airlines were set up. Water quality monitored. Organisms were fed while in holding. Organisms loaded into test vessels. Ref Tox initiated.

4 hours after elutriate test initiated, organisms were observed and counted

3/22/03. Test monitored, organisms observed and fed, Airlines checked. Ref Tox test monitored and observed.

3/23/03. Water quality monitored and organisms observed. Airlines checked. Fed BS.

3/24/03. Water quality monitored and organisms observed. Airlines checked. Organisms fed brine shrimp.

3/25/03. Water quality monitored. Organisms fed B.S. Organisms observed and counted. Test terminated.

MACTEC Engineering and Consulting, Inc.
Gainesville, Florida

QA Form No. 106
Effective: July 2002

DAILY FEEDING LOG

Sponsor: Ponce Harbor/ PPB	Project No.:
Test Substance: elutriate	Test Species: M. bahia

TEST DAY	DATE	TIME	INITIALS	FOOD TYPE	QUANTITY	COMMENTS
0	3/21/03	1130	HEB	Artemia nauplii	.2m	in holding
1	3/22/03	1800	HEB	Bs	.2m	
2	3/23/03	1900	HEB	Bs	.2m	
3	3/24/03	1500	JM	BS	.2mL	
4	3/25/03	1200	JM	BS	.2mL	

BS= Brine Shrimp nauplii YTC= Yeast/Trout Chow/Cereal Leaves Algae= Green Algae (*Selenastrum capricornutum*)

JM

MACTEC Engineering and Consulting, Inc.
Gainesville, Florida

QA Form No.: 097
Revised: March 2001

SUBJECT: TOXICITY TEST SYSTEM DESCRIPTION SHEET

Sponsor: Ponce Harbor

Project No.:

TEST MATERIAL

Test Material: Sediment

See Page 652-657 of Effluent Log for Test Material Information.

PROTOCOL/ GUIDELINES FOLLOWED:

SOP - S007

Test Concentrations Based on: ( ) Active Ingredient
(x) Whole Material

TEST ORGANISM HISTORY

Test Species: Mysidopis bahia

Lot Number: 2003-17

Age/ Life Stage: 2 days

Date Acclimation/ Maintenance Began: 3/21/03

See Page 107 of organism receipt Log for Data.

Mortality (%) During 48 Hours Prior to Test Initiation: <5%

TEST CONDITIONS

( ) Range Finder
( ) Screening
(X) Definitive

(X) Static
( ) Flow-Through

Lighting: (x) Fluorescent ( ) Incandescent

Photoperiod: 16 Hours Light
8 Hours Dark

Test Duration: 96 hrs

Test Area Used: Water bath

Test Temperature (°C): 20 +/- 2

Test Salinity (ppt): 30 +/- 2

Replicates Per Concentration: 5

Organisms Per Replicate: 20

Dilution Water: Natural filtered seawater

TEST VESSELS

Dimensions (cm): Length x Width x Height

Test Solution Height (cm) :

Test Vessels: ~~( ) Open~~ (X) Covered
HB 3/21/03

Test Container Volume: 1 L

Test Solution Volume: 750 mL

TEST SOLUTION PREPARATION

Test Concentration %	Control	0	10	50	100				
Volume of Dilution Water Added (mL)	1500	1500	1350	750	-				
Volume of Effluent/ Stock Added (mL)	-	-	150	750	1500				

ADDITIONAL OBSERVATIONS:

DATA BY: HB DATE: 3/21/03

MACTEC
Gainesville, Florida

QA Form Number: 016
Revised: March 2001

ACUTE TOXICITY DATA SHEET - WATER QUALITY DATA

SPONSOR: Ponce Harbor/ PPB

PROJECT NUMBER: 312253

TEST SUBSTANCE: Elutriate

TEST SPECIES: M. bahia

		Day 0				Day 1				Day 2				Day 3				Day 4			
DATE		3/21/03				3/22/03				3/23/03				3/24/03				3/25/03			
TIME		1530				1730				1900				1205				1630			
INIT.		HEB				HEB				KG				DD				KG			
NOMIN CONCN % mg/L µg/L	REP	TEMP	PH	DO	SL or CN	TEMP	pH	DO	SL or CN	TEMP	pH	DO	SL or CN	TEMP	pH	DO	SL or CN	TEMP	pH	DO	SL or CN
Seawater Control 100%	A	21.6	8.4	7.2	30			~~7.5~~	~~30~~	HB 3/22/03							~~30~~ 30				
	B					19.3	8.3	7.5	30												
	C									19.5	8.2	7.9	30								
	D													19.0	8.1	7.6	30				
	E																	19.3	8.1	7.5	30
Control Sediment 100%	A	20.0	8.2	7.5	30																
	B					19.4	8.3	7.4	30												
	C									19.5	8.1	7.9	30								
	D													18.8	8.3	7.7	30				
	E																	19.0	8.2	7.8	30
Control Sediment 50%	A	20.7	8.3	7.5	30																
	B					19.5	8.3	7.3	30												
	C									19.6	8.2	7.8	30				~~30~~				
	D													18.7	8.3	7.8	30				
	E																	19.1	8.2	7.8	30
INST/METH		FS6	SA 290A	DO3	Ref10	FS6	SA 290A	DO3	Ref10	FS6	SA290A	DO3	Ref9	FS6	SA290A	DO3	Ref9	FS6	SA290A	DO3	Ref9

KEY

TEMP - Temperature
SL - Salinity (ppt)
pH - standard units

DO - Dissolved oxygen (mg/L)
CN - Conductivity (µmhos/cm)
INST - Instrument
NOMIN CONCN - Nominal concentration

INIT. - Initials
REP - Replicate
METH - Method

JM

MACTEC
Gainesville, Florida

QA Form Number: 016
Revised: March 2001

ACUTE TOXICITY DATA SHEET - WATER QUALITY DATA

SPONSOR: Ponce Harbor/ PPB

PROJECT NUMBER: 312253

TEST SUBSTANCE: Elutriate

TEST SPECIES: M. bahia

		Day 0				Day 1				Day 2				Day 3				Day 4			
DATE		3/21/03				3/22/03				3/23/03				3/24/03				3/25/03			
TIME		1530				1730				1900				1205				1630			
INIT.		HEB				HEB				KG				DD				KG			
NOMIN CONCN % mg/L µg/L	REP	TEMP	PH	DO	SL or CN	TEMP	pH	DO	SL or CN	TEMP	pH	DO	SL or CN	TEMP	pH	DO	SL or CN	TEMP	pH	DO	SL or CN
Control Sediment 10%	A	21.2	8.4	7.5	30																
	B					19.4	8.3	7.4	30												
	C									19.7	8.2	7.8	30		~~8.2~~		~~30~~				
	D													18.8	8.2	7.7	30		KG		
	E																	19.0	8.3	7.8	30
E-PH03-SC 100%	A	19.8	8.1	7.4	30																
	B					19.4	8.4	7.5	30	~~19.7~~	~~8.3~~	~~7.7~~				7.7					
	C									19.7	8.3	7.7	30	~~18.6~~	~~8.5~~	~~8.5~~	~~30~~				
	D													18.6	8.5	7.7	30				
	E																	18.7	8.4	7.9	30
E-PH03-SC 50%	A	20.9	8.3	7.5	30																
	B					19.4	8.5	7.4	30												
	C									19.8	8.4	7.7	30			7.9					
	D													18.1	8.4	~~8.4~~	30				
	E																	18.6	8.3	7.9	30
INST/METH		FS6	SA 290A	DO3	Ref10	FS6	SA 290A	DO3	Ref10	FS6	SA290A	DO3	Ref9	FS6	SA 290A	DO3	Ref10	FS6	SA290A	DO3	Ref9

KEY

TEMP - Temperature
SL - Salinity (ppt)
pH - standard units

DO - Dissolved oxygen (mg/L)
CN - Conductivity (µmhos/cm)
INST - Instrument
NOMIN CONCN - Nominal concentration

INIT. - Initials
REP - Replicate
METH - Method

MACTEC
Gainesville, Florida

QA Form Number: 016
Revised: March 2001

ACUTE TOXICITY DATA SHEET - WATER QUALITY DATA

SPONSOR: Ponce Harbor/ PPB

PROJECT NUMBER: 312253

TEST SUBSTANCE: Elutriate

TEST SPECIES: M. bahia

	Day 0	Day 1	Day 2	Day 3	Day 4
DATE	3/21/03	3/22/03	3/23/03	3/24/03	3/25/03
TIME	3/21/03 1530	1730	1900	1200	1635
INIT.	HEB	HEB	KG	DS	KG

NOMIN CONCN % mg/L µg/L	REP	TEMP	PH	DO	SL or CN	TEMP	pH	DO	SL or CN	TEMP	pH	DO	SL or CN	TEMP	pH	DO	SL or CN	TEMP	pH	DO	SL or CN
E-PH03-SC 10%	A	21.1	8.3	7.9	30																
	B					19.4	8.4	~~7.5~~ 7.4	~~30~~ 30	HB 3/22/03											
	C									19.7	8.2	7.8	30								
	D													18.5	8.3	7.8	30				
	E													~~18.5~~	~~8.3~~	~~7.8~~	~~30~~ KG 3/25/03	18.8	8.3	8.0	30
	A																				
	B																				
	C																				
	D																				
	E																				
	A																				
	B																				
	C																				
	D																				
	E																				
INST/METH		FS6	SA 290A	DO3	Ref10	FS6	SA 290A	DO3	Ref10	FS6	SA290A	DO3	Ref10	FS6	SA290A	DO3	Ref10	FS6	SA290A	DO3	Ref10

KEY

TEMP - Temperature
SL - Salinity (ppt)
pH - standard units

DO - Dissolved oxygen (mg/L)
CN - Conductivity (µmhos/cm)
INST - Instrument
NOMIN CONCN - Nominal concentration

INIT. - Initials
REP - Replicate
METH - Method

MACTEC Engineering & Consulting, Inc. (formerly Harding ESE)
Gainesville, Florida

QA Form: 015d Region II
Revised: November 2002

Subject: Acute Toxicity Data Sheet – Survival

Sponsor: PPB/Ponce Harbor **Project No.:** 312253

Test Substance: elutriate **Test Species:** M. bahia

Test Hour		0-Hour		4-Hours		24-Hours		48-Hours		72-Hours		96-Hours	
Date		3/21/03		3/21/03		3/22/03		3/23/03		3/24/03		3/25/03	
Feeding		BS		–		BS		BS		BS		BS	
Time		1830		2200		1530		1900		1220		1700	
Technician		HEB		Jm		Jm		JA		SB		KG	
Nominal Concentration mg/L %	REP	Observ.	No. Alive	Observ.	No. Alive	Observ.	No. Alive	Observ.	No. Alive	Observ.	No. Alive	Observ.	No. Alive
Seawater Control 100%	A	Loaded	20	N	20	1d	19	N	19	N	19	N	19
	B			N	20	1d	19	N	19	N	19	1d	18
	C			N	20	2d	18	N	18	N	18	N	18
	D			N	20	N	20	1d	19	N	19	N	19
	E			N	20	N	20	1d	19	N	19	N	19
Control Sediment 10%	A			~~2d N~~	~~20~~	1d	19	N	19	N	19	N	19
	B			~~1d N~~	~~20~~	1d	19	N	19	N	19	1d	18
	C			~~1d N~~	~~20~~	N	20	N	20	N	20	N	20
	D			N	20	N	20	N	20	N	20	1d	19
	E			N	20	N	20	N	20	N	20	N	20
Control Sediment 50%	A			N	20	2d	18	N	18	1d	17	N	17
	B			N	~~19~~ 20	N	20	1d	19	N	19	N	19
	C			N	20	N	20	N	20	N	20	N	20
	D			N	20	N	20	N	20	N	20	1d	19
	E			N	20	N	20	N	20	N	20	N	20
Control Sediment 100%	A			N	20	N	20	N	20	N	20	1d	19
	B			N	20	N	20	N	20	N	20	N	20
	C			N	20	N	20	N	20	N	20	N	20
	D			N	20	1d	19	N	19	N	19	N	19
	E			N	20	1d	19	N	19	N	19	N	19
E-PH03-SC 10%	A			N	20	N	20	N	20	1d	19	N	19
	B			N	20	N	20	N	20	N	20	1d	19
	C			N	20	N	20	N	20	N	20	N	20
	D			N	20	N	20	N	20	N	20	N	20
	E			N	20	N	20	N	20	N	20	1d	19

Observation Key

AS = Organisms At Surface of Water	LE = Loss of Equilibrium	OBS = Observations
BS = Brine Shrimp	LETH = Lethargic	PRE = Precipitate
ERR = Erratic	LT = Light Pigmentation	RAR = Rapid Respiration
FOS = Film on Surface of Water	N (NONE) = Normal Conditions	REP = Replicate
G = Gulping	NF = Not Found	UC = Undissolved Chemical
GY = Gyrating	NO. ALIVE = Number of Organisms Alive (survival)	YTC/ALG = YTC and green algae (daphnids)
HYP = Hyperactive		

MACTEC Engineering & Consulting, Inc. (formerly Harding ESE)
Gainesville, Florida

QA Form: 015d Region II
Revised: November 2002

Subject: Acute Toxicity Data Sheet – Survival

Sponsor: PPB/Ponce Harbor **Project No.:** 312253

Test Substance: elutriate **Test Species:** ~~M. beryllina~~ jmr M. bahia

Test Hour		0-Hour		4-Hours		24-Hours		48-Hours		72-Hours		96-Hours	
Date		3/21/03		3/21/03		3/22/03		3/23/03		3/24/03		3/25/03	
Feeding		BS		–		BS		BS		BS		BS	
Time		1830		2200		1530		1900		1220		1700	
Technician		HEB		jmr		jmr		JA		[illegible]		KG	
Nominal Concentration mg/L %	REP	Observ.	No. Alive	Observ.	No. Alive	Observ.	No. Alive	Observ.	No. Alive	Observ.	No. Alive	Observ.	No. Alive
E-PH03-SC 50%	A	Loaded	20	N	20	N	20	20	N	N	20	1d	19
	B	↓	↓	N	20	N	20	20	N	N	20	N	20
	C			N	20	N	20	20	N	N	20	1d	19
	D			N	20	N	20	20	N	N	20	2d	18
	E			N	20	N	20	20	N	N	20	1d	19
E-PH03-SC 100%	A			N	20	N	20	N	20	1d	19	N	19
	B			N	20	4d	16	N	16	N	16	N	16
	C			N	20	N	20	N	20	N	20	1d	19
	D			N	20	2d	18	N	18	1d	17	N	17
	E	↓	↓	N	20	4d	16	N	16	N	16	N	16
	A												
	B												
	C												
	D												
	E												
	A												
	B												
	C												
	D												
	E												
	A												
	B												
	C												
	D												
	E												

Observation Key

AS = Organisms At Surface of Water
BS = Brine Shrimp
ERR = Erratic
FOS = Film on Surface of Water
G = Gulping
GY = Gyrating
HYP = Hyperactive
LE = Loss of Equilibrium
LETH = Lethargic
LT = Light Pigmentation
N (NONE) = Normal Conditions
NF = Not Found
NO. ALIVE = Number of Organisms Alive (survival)
OBS = Observations
PRE = Precipitate
RAR = Rapid Respiration
REP = Replicate
UC = Undissolved Chemical
YTC/ALG = YTC and green algae (daphnids)

jmr

MACTEC Engineering and Consulting
TOXICOLOGY LABORATORY
GAINESVILLE, FLORIDA

PAGE:
FORM NO: 097A REFTOX
REVISED: MARCH 2001

REFERENCE TOXICANT TEST DATA SHEET

Sponsor: Misc.	Project Number: Ref Tox
Test Material	**Test Conditions**
Amount of SDS : 0.100 (g) Volume of Milli-Q Water: 100 (mL) Date Prepared: 3/21/03	[x] Definitive [ ] Static [ ] Screening Duration: 96 Hours
Test Organism History	Dilution Water: natural filtered seawater
Species: M. bahia Batch Number: 2003-17 Age/Life Stage: 1 days Date of Acclimation/Maintenance: 3/21/03 See Page 107 of Org. receipt Log Mortality 48 hours prior to test: <5 (%) Replicates/Concentration: 1 No. Of Organisms/Replicate: 10	Lighting: [x] Fluorescent [ ] Incandescent Photoperiod: 16 Hr Light 8 Hr Dark Test Vessel Dimensions: ___ L X ___ W ___ H Test Solution Height: ___ (cm) Test Vessel Volume: 0.34 (L) Test Solution Volume: 0.20 (L) Test Temperature: 20 ± 1 °C Test Salinity: 30 ± 2 ppt
Test Area Used: table area above waterbath	Test Vessel Composition: [x] Glass [ ] Plastic
Test Concentrations Based on: [x] Whole Material [ ] Active Ingredient	Test Protocol/Guideline Followed:

Test Concentrations: (Units: mg/L)	Control	2.5	5.0	10.0	20.0	40.0	
Amount Dilution Water Added (mL)	200	199.5	199	198	196	192	
Amount of Stock Added (mL)	0	0.5	1.0	2.0	4.0	8.0	

Additional Observations: _____

Data By: HEB Date: 3/21/03

REFERENCE TOXICANT TEST DATA: DAY 0 – 2

Client: Misc.	Project Number: Ref Tox	Species: M. bahia

DAY 0 — REF TOXICANT: SDS

Nominal Concentration (mg/L)	Date: 3/21/03		Time: 1530		Analyst: HEB	
	# Alive	Observ.	Temp. (°C)	Sal (ppt)	DO (mg/L)	pH
Control	10	loaded	20.9	30	7.3	8.3
2.5	↓	↓	21.2	30	7.4	8.3
5			21.5	30	7.4	8.3
10			21.5	30	7.3	8.3
20			21.6	30	7.6	8.3
40			21.6	30	7.4	8.3

DAY 1	Date: 3/22/03		Time: 1730		Analyst: HEB	
Nominal Concentration	# Alive	Observ.	Temp. (°C)	Sal (ppt)	DO (mg/L)	pH
Control	10	N	19.2	30	7.4	8.2
2.5	10	N	19.2	30	7.3	8.2
5	10	N	19.2	30	7.1	8.2
10	8	2d	19.2	30	7.0	8.2
20	0	10d	19.2	30	7.1	8.2
40	0	10d	19.3	30	7.1	8.1

DAY 2	Date: 3/23/03		Time: 1930		Analyst: HEB	
Nominal Concentration	# Alive	Observ.	Temp. (°C)	Sal (ppt)	DO (mg/L)	pH
Control	10	N	19.6	30	7.2	8.2
2.5	9	1d	19.6	30	7.3	8.2
5	9	1d	19.5	31	7.2	8.2
10	5	3d ~~5d~~	19.7	30	6.9	8.1

CG

MACTEC Engineering and Consulting, Inc.
TOXICOLOGY LABORATORY
GAINESVILLE, FLORIDA

QA FORM NO: 097 REFTOX
Revised: March 2001

REFERENCE TOXICANT TEST DATA: DAY 3 AND 4 SDS

Client: Mrsc	Project Number: Ref Tox	Species: M. bahia

DAY 3

Nominal Concentration	Date: 03/24/03		Time: 1400		Analyst: [illegible]	
	# Alive	Observ.	Temp. (°C)	Sal (ppt)	DO (mg/L)	pH
Control	10 ~~N~~	N	19.8	31	7.3	8.2
2.5	9	N	19.8	30	6.7	8.1
5	9	N	19.8	31	7.0	8.1
10	5	N	19.8	31	6.8	8.1
~~20~~						
~~40~~						

DAY 4	Date: 3/25/03		Time: 1600		Analyst: KG	
Nominal Concentration	# Alive	Observ.	Temp. (°C)	Sal (ppt)	DO (mg/L)	pH
Control	9	ld	19.3	30	7.5	8.1
2.5	9	N	19.3	30	7.6	8.1
5	8	ld	19.4	30	7.5	8.1
10	4	ld	19.4	30	7.0	~~8.1~~ 8.4 3/26/03
20						
40						

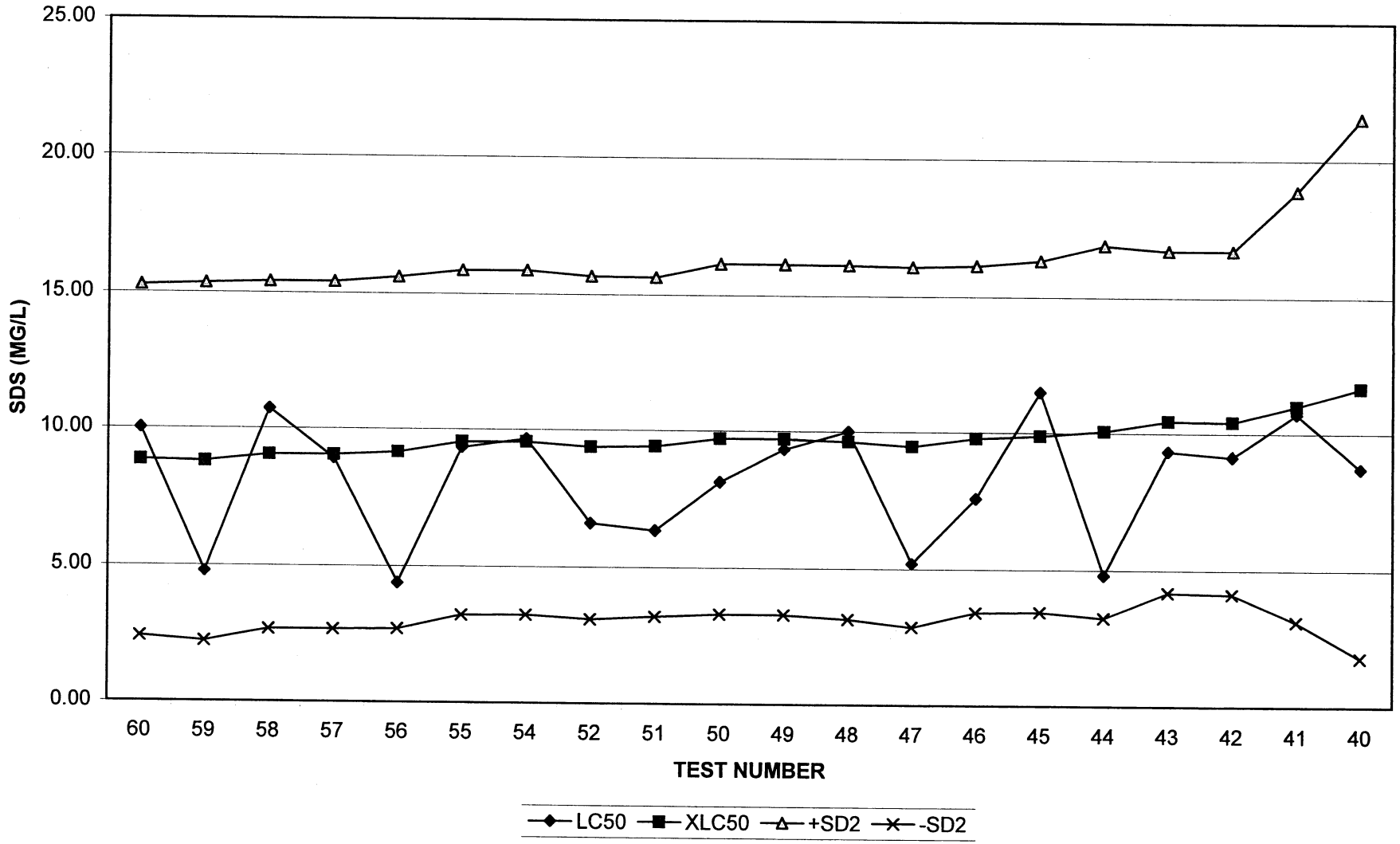
SUMMARY

Nominal Concentration	Number Dead
Control	1
2.5	1
5	2
10	6
20	10
40	10

COMMENTS:

[illegible]

MYSIDOPSIS BAHIA
ACUTE REFERENCE TOXICITY TEST:SODIUM LAURYL SULFATE



8/28/2003

TRIMMED SPEARMAN-KARBER METHOD. VERSION 1.5

DATE: 03/21/03 TEST NUMBER: 1 DURATION: 96 h
[illegible]ICANT : SDS
SPECIES: M. bahia

RAW DATA: --- ----	Concentration (mg/L)	Number Exposed	Mortalities
	.00	10	1
	2.50	10	1
	5.00	10	2
	10.00	10	6
	20.00	10	10
	40.00	10	10

SPEARMAN-KARBER TRIM: .00%

SPEARMAN-KARBER ESTIMATES: LC50: 8.91
95% LOWER CONFIDENCE: 6.88
95% UPPER CONFIDENCE: 11.53

NOTE: MORTALITY PROPORTIONS WERE NOT MONOTONICALLY INCREASING.
ADJUSTMENTS WERE MADE PRIOR TO SPEARMAN-KARBER ESTIMATION.

Aquatic Research Organisms

DATA SHEET

I. Organism History

Species: AMERICAMYSIS bahia (Mysidopsis bahia)

Source: Lab reared ✓ Hatchery reared ____ Field collected ____

Hatch date 3-19-03 Receipt date ____

Lot number 031903MS Strain ____

Brood Origination FLORIDA

II. Water Quality

Temperature 25 °C Salinity ≈27 ppt DO —

pH 7.8 Hardness — ppm

III. Culture Conditions

System: RECIRC

Diet: Flake Food ✓ Phytoplankton ____ Trout Chow ✓

Brine Shrimp ✓ Rotifers ____ Other ENCAP. SHRIMP DIET

Prophylactic Treatments: ____

Comments: ____

IV. Shipping Information

Client: MACTEC # of Organisms: 320+

Carrier: FEDEX Date Shipped: 3-20-03

Biologist: Mark [illegible]

1 - 800 - 927 - 1650

PO Box 1271 • One Lafayette Road • Hampton, NH 03842 • (603) 926-1650

rec'd 3/21/03 1100

INV # 120205

PO # 5604

Environmental Science & Engineering, Inc.
Aquatic Toxicology Laboratory
Gainesville, Florida

ESE QA Form No.: 008
Effective: Mar 1993

SUBJECT: ORGANISM RECEIPT/COLLECTION LOG

Date Recieved	Species	Population Size	Population Condition	Collector/ Supplier	Salinity/ Hardness	Temperature (C)	Initials
3/18/03	M. nasuta	2003-16 ~500	normal	ARO	—	8.5	~~JMM~~ adult
3/21/03	Americamysis bahia	320+ 2003-17	normal	ARO	28	23.1	HEB 2-days
3/21/03	M. beryllina	320+ 2003-18	normal	ARO	27	23.1	HEB 10-days
3/23/03	M. bahia	375 2003-19	normal	Aquatic Indic.	20 ppm	25.0	JA 2 days
3/23/03	M. beryllina	85 2003-20	normal	Aquatic Indic.	20 ppm	25.0	JA 10 day
3/26/03	M. edulis	~48 2003-21	normal	ARO	—	11.3	JMM adult
3/28/03	M. bahia	450+ 2003-22	normal	ARO	28 ppt	22.8	HEB 3days
3/31/03	Brine Shrimp Cysts	15 oz can	normal	Aquatic Eco-Systems	—	—	CG
4/2/03	M. nasuta	2003-23 1500	normal	ARO	—	12.7	CG adult
4/3/03	N. Virens	2003-24 500	normal	ARO	—	10.5	CG adult
4/4/03	L. plumulosus	2003-25 ~~+200~~ +500	normal	ARO	23 ppt	17.4	CG

Harding ESE
Toxicology Laboratory
Gainesville, Florida

Page: 58
QA Form No. 009
Revised: March 2001

SUBJECT: DAILY RECORD OF INVERTEBRATE HOLDING CONDITIONS

Species: Americamysis bahia Lot Number: 2003-17 Date collected/received: 3/21/03 Date of hatch: _____ Collection location/supplier: Aquatic Research Organisms Page 107 of animal receipt and collection log.
Data continued from Page _____ of this log book.

Date	3/21/03				
Time	1130				
Technician	MEB				
Number alive	320				
Number dead	<5%				
Hardness/Salinity	28				
Method/Meter	Ref 10				
Temperature (EC)	23.1				
Meter number	FS-6				
D.O. (mg/L)	>10				
Meter number	DO3				
pH (s.u.)	7.8				
Meter number	SA290				
Water	Clear				
Food	BS				
Clean	✓				
Number removed					
Project No. / Task	312253				

_____ Data continued on Page _____ of this log book.
_____ Population was terminated. Date 3/2 How _____

Comments: test initiated

CR

MACTEC Engineering & Consulting, Inc.
Toxicology Laboratory
Gainesville, Florida

Page: 1 of 1

QA Form No. 018
Revised: October 2002

Project No: 312253.0300 Client: Ponce Harbor/ PPB Test: Elutriate, M. beryllina

DAILY LOG

3/23/03. M. beryllina received. Fed brine shrimp (BS) while in holding.

3/24/03. Elutriate prepared by mixing 1 part sediment with 4 parts sitewater, for 30 minutes. Mixture allowed to settle for one hour and the overlying water then siphoned off. Dilutions prepared and loaded into test vessels. Airlines setup. Water quality monitored. Organisms fed BS while in holding. Organisms loaded into test vessels. 4 hours after elutriate test started, organisms were observed and counted.

3/25/03. Water quality monitored. Organisms observed. Airlines checked.

3/26/03. Water quality monitored. Organisms observed. Airlines checked.

3/27/03. Water quality monitored. Organisms observed. Airlines checked.

3/28/03. Water quality monitored. Organisms observed and counted. Test terminated.

MACTEC Engineering and Consulting, Inc.
Gainesville, Florida

QA Form No.: 097
Revised: March 2001

SUBJECT: TOXICITY TEST SYSTEM DESCRIPTION SHEET

Sponsor: Ponce Harbor

Project No.: 312253.0300

TEST MATERIAL

Test Material: Sediment

See Page 652-657 of Effluent Log for Test Material Information.

PROTOCOL/ GUIDELINES FOLLOWED:

Test Concentrations Based on: ( ) Active Ingredient
(x) Whole Material

TEST ORGANISM HISTORY

Test Species: Menidia beryllina

Lot Number: 2003 - 20

Age/ Life Stage: 10 days

Date Acclimation/ Maintenance Began: 3/23/03

See Page 107 of Organisms Receipt Log for Data.

Mortality (%) During 48 Hours Prior to Test Initiation: < 1%

TEST CONDITIONS

( ) Range Finder
( ) Screening
(X) Definitive

(X) Static
( ) Flow-Through

Lighting: (x) Fluorescent ( ) Incandescent

Photoperiod: 16 Hours Light
8 Hours Dark

Test Duration: 96 hrs

Test Area Used: Water bath

Test Temperature (°C): 20 +/- 2

Test Salinity (ppt): 30 +/- 2

Replicates Per Concentration: 5

Organisms Per Replicate: 20

Dilution Water: Natural filtered seawater

TEST VESSELS

Dimensions (cm): Length x Width x Height

Test Solution Height (cm) :

Test Vessels: ~~(x) Open~~ (X) Covered

Test Container Volume: 250 mL

Test Solution Volume: 200 mL

TEST SOLUTION PREPARATION

Test Concentration %	Control	0	10	50	100				
Volume of Dilution Water Added (mL)	1500	1500	1350	750	-				
Volume of Effluent/ Stock Added (mL)	-	-	150	750	1500				

ADDITIONAL OBSERVATIONS:

DATA BY: JMW DATE: 3/24/03

JMW

MACTEC
Gainesville, Florida

QA Form Number: 016
Revised: March 2001

ACUTE TOXICITY DATA SHEET - WATER QUALITY DATA

SPONSOR: Ponce Harbor/ PPB	PROJECT NUMBER: 312253
TEST SUBSTANCE: Elutriate	TEST SPECIES: M. beryllina

DATE		03/24/03				3/25/03				3/26/03				3/27/03				3/28/03			
TIME		1600				1645				0950				0920				1600			
INIT.		DD				KG				DD				KG				HEB			
NOMIN CONCN % mg/L µg/L	REP	TEMP	PH	DO	SL or CN	TEMP	pH	DO	SL or CN	TEMP	pH	DO	SL or CN	TEMP	pH	DO	SL or CN	TEMP	pH	DO	SL or CN
Seawater Control 100%	A	18.4	8.3	8.0	30																
	B					18.9	8.1	7.8	29				2								
	C									19.0	7.5	7.4	29								
	D													19.0	8.2	7.9	30				
	E																	20.5	8.0	7.2	30
Control Sediment 10%	A	19.5	8.3	8.0	30																
	B					18.8	8.1	7.9	29	~~18.8~~											
	C									18.8	7.7	7.5	29								
	D													19.0	8.1	7.7	30				
	E																	20.4	8.0	7.0	30
Control Sediment 50%	A	19.2	8.2	7.9	30																
	B					18.7	8.3	7.8	29												
	C									18.8	7.8	7.6	29								
	D													18.9	8.3	7.9	30				
	E																		7.		~~30~~
																		20.3	7.7	4.3	30
INST/METH		FS6	SA290A	DO3	Ref9	FS6	SA290A	DO3	Ref9	FS6	SA290A	DO3	Ref9	FS6	SA290A	DO3	Ref9	FS6	SA290B	DO3	Ref9

KEY

TEMP - Temperature
SL - Salinity (ppt)
pH - standard units

DO - Dissolved oxygen (mg/L)
CN - Conductivity (µmhos/cm)
INST - Instrument
NOMIN CONCN - Nominal concentration

INIT. - Initials
REP - Replicate
METH - Method

MACTEC
Gainesville, Florida

QA Form Number: 016
Revised: March 2001

ACUTE TOXICITY DATA SHEET - WATER QUALITY DATA

SPONSOR: Ponce Harbor/ PPB

PROJECT NUMBER: 312253

TEST SUBSTANCE: Elutriate

TEST SPECIES: M. beryllina

DATE		03/24/03				3/25/03				3/26/03				3/27/03				3/28/03			
TIME		1600				1645				0950				0920				1600			
INIT.		DD				KG				DD				KG				HEB			
NOMIN CONCN % mg/L µg/L	REP	TEMP	PH	DO	SL or CN	TEMP	pH	DO	SL or CN	TEMP	pH	DO	SL or CN	TEMP	pH	DO	SL or CN	TEMP	pH	DO	SL or CN
Control Sediment 100%	A	18.4	8.1	7.7	30																
	B				30	18.7	8.3	7.6	29				29								
	C				30					18.8	8.0	7.5	29								
	D				30									19.0	8.3	7.7	30				
	E				30													20.5	8.0	5.3	30
E-PH03-SC 10%	A	19.5	8.3	8.0	30			~~7.9~~ KG													
	B					18.6	8.1	7.4	29												
	C									18.6	7.9	7.6	30								
	D													18.9	8.3	7.8	30				
	E																	20.3	8.1	7.3	30
E-PH03-SC 50%	A	19.1	8.3	8.0	30																
	B					18.5	8.3	7.2	29												
	C									18.6	8.1	7.8	30								
	D													18.9	8.3	8.0	30				
	E																	20.4	8.2	~~5.3~~ 7.3	30 HEB
																					~~30~~ 30
INST/METH		FS6	SA290A	DO3	Ref9	FS6	SA290A	DO3	Ref9	FS6	SA290A	DO3	Ref9	FS6	SA290B	DO3	Ref9	FS6	SA290A	DO3	Ref9

KEY

TEMP - Temperature
SL - Salinity (ppt)
pH - standard units

DO - Dissolved oxygen (mg/L)
CN - Conductivity (µmhos/cm)
INST - Instrument
NOMIN CONCN - Nominal concentration

INIT. - Initials
REP - Replicate
METH - Method

MACTEC
Gainesville, Florida

QA Form Number: 016
Revised: March 2001

ACUTE TOXICITY DATA SHEET - WATER QUALITY DATA

SPONSOR: Ponce Harbor/ PPB

PROJECT NUMBER: 312253

TEST SUBSTANCE: Elutriate

TEST SPECIES: M. beryllina

DATE		03/24/03				3/24/03				3/26/03				3/27/03				3/28/03			
TIME		1600				1645				0950				0920				1600			
INIT.		DD				KG				DD				KG				HEB			
NOMIN CONCN % mg/L µg/L	REP	TEMP	PH	DO	SL or CN	TEMP	pH	DO	SL or CN	TEMP	pH	DO	SL or CN	TEMP	pH	DO	SL or CN	TEMP	pH	DO	SL or CN
E-PH03-SC 100%	A	19.0	8.3	7.9	30																
	B					18.6	8.2	7.2	29												
	C									18.8	8.1	7.3	30								
	D													18.9	8.4	7.9	30				
	E																	20.3	8.2	7.0	30
	A																				
	B																				
	C																				
	D																				
	E																				
	A																				
	B																				
	C																				
	D																				
	E																				
INST/METH		FS6	SA290A	DO3	Ref9	FS6	SA290A	DO3	Ref9	FS6	SA290A	DO3	Ref9	FS6	SA290B	DO3	Ref9	FS6 ~~SA290B~~	SA290B ~~DO3~~	DO3	Ref9

KEY

TEMP - Temperature
SL - Salinity (ppt)
pH - standard units

DO - Dissolved oxygen (mg/L)
CN - Conductivity (µmhos/cm)
INST - Instrument
NOMIN CONCN - Nominal concentration

INIT. - Initials
REP - Replicate
METH - Method

MACTEC Engineering & Consulting, Inc. (formerly Harding ESE)
Gainesville, Florida

QA Form: 015d Region II
Revised: November 2002

Subject: Acute Toxicity Data Sheet – Survival

Sponsor: PPB/Ponce Harbor **Project No.:** 312253

Test Substance: elutriate **Test Species:** M. beryllina

Test Hour		0-Hour		4-Hours		24-Hours		48-Hours		72-Hours		96-Hours	
Date		3/24/03		3/24/03		3/25/03		3/26/03		3/27/03		3/28/03	
Feeding		—		—		—		—		—		—	
Time		1700		2030		1650		1010		0945		1600	
Technician		DD		JM		KG		DD		KG		HBB	
Nominal Concentration mg/L %	REP	Observ.	No. Alive	Observ.	No. Alive	Observ.	No. Alive	Observ.	No. Alive	Observ.	No. Alive	Observ.	No. Alive
Seawater Control 100%	A	loaded	20	N	20	N	20	N	20	N	20	N	20
	B			N	20	N	20	N	20	N	20	N	20
	C			N	20	N	20	N	20	N	20	N	20
	D			N	20	N	20	N	20	N	20	N	20
	E			N	20	N	20	N	20	N	20	ld	19
Control Sediment 10%	A			N	20	N	20	ld	19	N	19	N	19
	B			N	20	N	20	N	20	N	20	N	20
	C			N	20	N	20	N	20	N	20	ld	19
	D			N	20	N	20	N	20	N	20	N	20
	E			N	20	N	20	ld	19	N	19	N	19
Control Sediment 50%	A			N	20	N	20	N	20	N	20	N	20
	B			N	20	N	20	N	20	N	20	N	20
	C			N	20	N	20	N	20	N	20	N	20
	D			N	20	N	20	N	20	N	20	ld	19
	E			N	20	N	20	ld	19	ld	18	ld	17
Control Sediment 100%	A			N	20	N	20	N	20	N	20	ld	19
	B			N	20	N	20	N	20	N	20	N	20
	C			N	20	ld	19	ld	18	N	18	N	18
	D			N	20	N	20	N	20	N	20	ld	19
	E			N	20	N	20	N	20	N	20	ld	19
E-PH03-SC 10%	A			N	20	N	20	ld	19	N	19	N	19
	B			N	20	N	20	N	20	N	20	ld	19
	C			N	20	N	20	ld	19	N	19	ld	18
	D			N	20	N	20	N	20	N	20	ld	19
	E			N	20	N	20	N	20	N	20	ld	19

Observation Key

AS = Organisms At Surface of Water
BS = Brine Shrimp
ERR = Erratic
FOS = Film on Surface of Water
G = Gulping
GY = Gyrating
HYP = Hyperactive
LE = Loss of Equilibrium
LETH = Lethargic
LT = Light Pigmentation
N (NONE) = Normal Conditions
NF = Not Found
NO. ALIVE = Number of Organisms Alive (survival)
OBS = Observations
PRE = Precipitate
RAR = Rapid Respiration
REP = Replicate
UC = Undissolved Chemical
YTC/ALG = YTC and green algae (daphnids)

JM

MACTEC Engineering & Consulting, Inc. (formerly Harding ESE)
Gainesville, Florida

QA Form: 015d Region II
Revised: November 2002

Subject: Acute Toxicity Data Sheet – Survival

Sponsor: PPB/Ponce Harbor **Project No.:** 312253

Test Substance: elutriate **Test Species:** M. beryllina

Test Hour		0-Hour		4-Hours		24-Hours		48-Hours		72-Hours		96-Hours	
Date		03/24/03		3/24/03		3/25/03		3/25/03		3/27/03		3/28/03	
Feeding				—		—		—		—		—	
Time		1700		2030		1650		1030		0940		~~1400~~ 1600	
Technician		DD		JM		KG		DD		KC		HEB	
Nominal Concentration mg/L %	REP	Observ.	No. Alive	Observ.	No. Alive	Observ.	No. Alive	Observ.	No. Alive	Observ.	No. Alive	Observ.	No. Alive
E-PH03-SC 50%	A	Loaded	20	N	20	2d	18	N	18	N	18	6d	12
	B			N	20	N	20	N	20	N	20	1d	19
	C			N	20	N	20	N	20	N	20	4d	16
	D			N	20	N	20	N	20	N	20	2d	18
	E			N	20	N	20	N	20	N	20	N	20
E-PH03-SC 100%	A			N	20	1d	19	N	19	3d	16	2d	14
	B			N	20	N	20	1d	19	1d	18	2d	16
	C			N	20	N	20	2d	18	N	18	N	18
	D			N	20	1d	19	2d	17	N	17	3d	14
	E			N	20	2d	18	5d	13	1d	12	2d	10
	A							N					
	B							N					
	C							N					
	D							DD 3/25/03					
	E							N					
	A												
	B												
	C												
	D												
	E												
	A												
	B												
	C												
	D												
	E												

Observation Key

AS =	Organisms At Surface of Water	LE =	Loss of Equilibrium	OBS =	Observations
BS =	Brine Shrimp	LETH =	Lethargic	PRE =	Precipitate
ERR =	Erratic	LT =	Light Pigmentation	RAR =	Rapid Respiration
FOS =	Film on Surface of Water	N (NONE) =	Normal Conditions	REP =	Replicate
G =	Gulping	NF =	Not Found	UC =	Undissolved Chemical
GY =	Gyrating	NO. ALIVE =	Number of Organisms Alive (survival)	YTC/ALG =	YTC and green algae (daphnids)
HYP =	Hyperactive				

MACTEC Engineering and Consulting
TOXICOLOGY LABORATORY
GAINESVILLE, FLORIDA

PAGE:
FORM NO: 097A REFTOX
REVISED: MARCH 2001

REFERENCE TOXICANT TEST DATA SHEET

Sponsor: Misc.	Project Number: Ref Tox
Test Material	**Test Conditions**
Amount of SDS: 0.100 (g) Volume of Milli-Q Water: 100 (mL) Date Prepared: 3/~~25~~ 24/03	[x] Definitive [x] Static [ ] Screening Duration: 96 Hours
Test Organism History	Dilution Water: natural filt. seawater
Species: M. beryllina Batch Number: 2003-20 Age/Life Stage: 10 days Date of Acclimation/Maintenance: 3/24/03 See Page 107 of Organisms Receipt Log Mortality 48 hours prior to test: <1 (%) Replicates/Concentration: 1 No. Of Organisms/Replicate: 10	Lighting: [ ] Fluorescent [ ] Incandescent Photoperiod: 16 Hr Light 8 Hr Dark Test Vessel Dimensions: ____L X ____W ____H Test Solution Height: _____(cm) Test Vessel Volume: 0.60 (L) Test Solution Volume: 0.20 (L) Test Temperature: 20 ± 1 °C Test Salinity: 20 ± 2 ppt
Test Area Used: table above water bath	Test Vessel Composition: [x] Glass [ ] Plastic
Test Concentrations Based on: [x] Whole Material [ ] Active Ingredient	Test Protocol/Guideline Followed:

Test Concentrations: (Units: mg/L)	Control	1.25	2.5	5.0	10.0	20.0	
Amount Dilution Water Added (mL)	200	199.75	199.5	199	198	196	
Amount of Stock Added (mL)	0	0.25	0.5	1.0	2.0	4.0	

Additional Observations: _____

Data By: DD Date: 3/~~25~~ 24/03

MACTEC Engineering and Consulting
TOXICOLOGY LABORATORY
GAINESVILLE, FLORIDA

Test with 20ppt

PAGE:
QA FORM NO: 097RT REFTOX
Revised: March 2001

REFERENCE TOXICANT TEST DATA: DAY 0 – 2

Client: Misc.	Project Number: Ref Tox	Species: M. beryllina

DAY 0 — REF TOXICANT: SDS

Nominal Concentration (mg/L)	Date: 3/24/03		Time: 1100		Analyst: CG	
	# Alive	Observ.	Temp. (°C)	Sal (ppt)	DO (mg/L)	pH
Control	10	Loaded	19.0	19	7.8	7.6
1.25	↓	↓	19.1	19	7.7	7.6
2.5			19.0	19	7.7	7.6
5.0			19.0	19	7.7	7.6
10.0			19.0	19	7.7	7.6
20.0			19.1	19	7.7	7.6

DAY 1 — Date: 3/~~28~~ 25/03 — Time: 1100 — Analyst: HEB

Nominal Concentration	# Alive	Observ.	Temp. (°C)	Sal (ppt)	DO (mg/L)	pH
Control	10	N	19.7	19	8.0	8.1
1.25	10	N	20.0	19	7.2	8.0
2.5	4	bd	20.0	19	7.0	8.0
5.0	0	10d	19.9	19	4.0	8.0
10.0	0	10d	19.8	19	4.0	8.0
20.0	0	10d	19.9	19	4.0	7.8

DAY 2 — Date: 3/~~29~~ 26/03 — Time: 1100 — Analyst: HEB

Nominal Concentration	# Alive	Observ.	Temp. (°C)	Sal (ppt)	DO (mg/L)	pH
Control	10	N	20.0	19	7.20	8.2
1.25	10	N	20.0	20	6.80	8.2
2.5	3	1d	20.0	20	6.80	8.2
5.0						
10.0						
20.0						

PAGE:

Harding ESE
TOXICOLOGY LABORATORY
GAINESVILLE, FLORIDA

QA FORM NO: 097 REFTOX
Revised: March 2001

Test with 20ppt

REFERENCE TOXICANT TEST DATA: DAY 3 AND 4

Client: Misc.	Project Number: Ref Tox	Species: M. beryllina

DAY 3

Nominal Concentration	Date: 3/~~28~~ 27 Jm/03		Time: 1130		Analyst: KG	
	# Alive	Observ.	Temp. (°C)	Sal (ppt)	DO (mg/L)	pH
Control	10	N	19.8		7.1	8.3
1.25	10	N	19.9		7.2	8.2
2.5	3	N	20.1		7.4	8.3
5.0						
10.0						
20.0						

DAY 4

Nominal Concentration	Date: 3/~~29~~ 28 Jm/03		Time: 1130		Analyst: DD	
	# Alive	Observ.	Temp. (°C)	Sal (ppt)	DO (mg/L)	pH
Control	10	N	19.1	19	6.6	8.3
1.25	10	N	19.5	19	6.9	8.3
2.5	3	N	19.6	19	7.0	8.3
5.0						
10.0						
20.0						

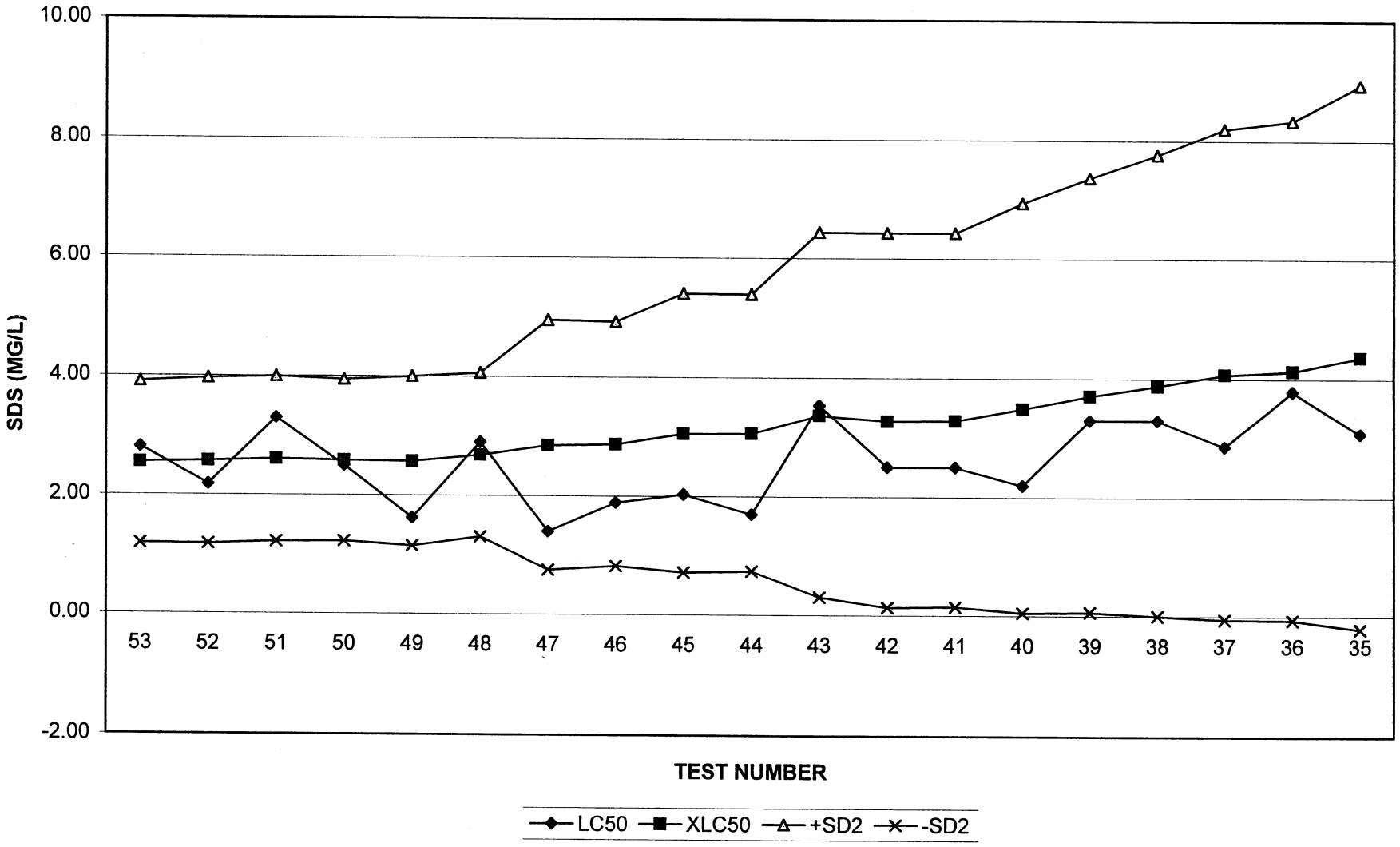
SUMMARY

Nominal Concentration	Number Dead
Control	~~10~~ DD 0
1.25	~~10~~ DD 0
2.5	7
CG ~~10.0~~ 5.0	10
3/27/03 10.0	10
20.0	10

COMMENTS:

Jm

MENIDIA BERYLLINA
ACUTE REFERENCE TOXICITY TEST:SODIUM LAURYL SULFATE



8/29/2003

TRIMMED SPEARMAN-KARBER METHOD. VERSION 1.5

DATE: 03/27/03 TEST NUMBER: 1 DURATION: 96 h
TOXICANT : SDS
SPECIES: M. beryllina

RAW DATA:

Concentration (mg/L)	Number Exposed	Mortalities
.00	10	0
1.25	10	0
2.50	10	7
5.00	10	10
10.00	10	10
20.00	10	10

SPEARMAN-KARBER TRIM: .00%

SPEARMAN-KARBER ESTIMATES: LC50: 2.18
95% LOWER CONFIDENCE: 1.78
95% UPPER CONFIDENCE: 2.66

3/23/03

Received ~~375 M. bahia (elutr)~~

875 M. beyllina (elutriate test)

from Agnetic Indicators

J. Ry

312253. 0500

add'l orgs for elutr. test

Environmental Science & Engineering, Inc.
Aquatic Toxicology Laboratory
Gainesville, Florida

ESE QA Form No.: 008
Effective: Mar 1993

SUBJECT: ORGANISM RECEIPT/COLLECTION LOG

Date Recieved	Species	Population Size	Population Condition	Collector/ Supplier	Salinity/ Hardness	Temperature (C)	Initials
3/18/03	M. nasuta	2003-16 ~500	normal	ARO	—	8.5	~~JMM~~ adult
3/21/03	Americamysis bahia	320+ 2003-17	normal	ARO	28	23.1	HEB 2-days
3/21/03	M. beryllina	320+ 2003-18	normal	ARO	27	23.1	HEB 10-days
3/23/03	M. bahia	375 2003-19	normal	Aquatic Indic.	20 ppm	25.0	JA 2 days
3/23/03	M. beryllina	885 2003-20	normal	Aquatic Indic	20 ppm	25.0	JA 10 day
3/26/03	M. edulis	~48 2003-21	normal	ARO	—	11.3	JMM adult
3/28/03	M. bahia	450+ 2003-22	normal	ARO	28 ppt	22.8	HEB 3days
3/31/03	Brine Shrimp Cysts	15 oz can	normal	Aquatic Eco-Systems	—	—	CG
4/2/03	M. nasuta	2003-23 ~500	normal	ARO	—	12.7	CG adult
4/3/03	N. Virens	2003-24 500	normal	ARO	—	10.5	CG adult
4/4/03	L. plumulosus	2003-25 ~~±200~~ +500	normal	ARO	23 ppt	17.4	CG

ENVIRONMENTAL SCIENCE AND ENGINEERING
Toxicology Laboratory
Gainesville, Florida

Page: 99
ESE QA Form No. 010
Effective: AUG 1997

SUBJECT: DAILY RECORD OF FISH HOLDING CONDITIONS

Species: M. ~~Beryllina~~ beryllina
Lot Number: 2003-20
Date collected/received: 3/23/03
Date of hatch: 3/13/03
Collection location/supplier: Aquatic Indicators Inc
Page 107 of animal receipt and collection log.
Data continued from Page _____ of this log book.

Date	3/23/03				
Time	18:30				
Technician	JA				
Number alive	875				
Number dead	0				
Hardness/Salinity	—				
Method/Meter	—				
Temperature (°C)	25.0				
Meter number	FS-7				
D.O. (mg/L)	>~~8~~10				
Meter number	DO-3				
pH (s.u.)	8.1				
Meter number	SA-290A				
Water	—				
Food	BS				
Clean	Yes				
Number removed	0				
Project No. / Task	312253				

_____ Data continued on Page _____ of this log book.
_____ Population was terminated. Date_____ How_____

Comments: _____

CR

MACTEC Engineering & Consulting, Inc.
Toxicology Laboratory
Gainesville, Florida

Page: _____
QA Form No. 018
Revised: October 2002

Project No: 312253 Client: Ponce Harbor/ PPB Test: Elutriate-M. edulis

DAILY LOG

5/23/03 - organisms removed from packing box (from 8°C ± cold room) and placed in 20°C saltwater vessels. After ~30 minutes, saltwater was replaced with new 20°C 30 ppt saltwater (organisms chilled cold the water). (Organisms were received 5/20/03 and held in coldroom). Thermal shock method was used to induce spawning (from 20°C for ~45 min to 1 hour, to ~10°C for ~30 min). Organisms were constantly monitored and placed into separate vessels when release of gametes began. Elutriate prepared. WQ monitored. Test vessels and Ref Tox vessels were loaded with 1 ml each, of embryo suspension. Suspension was not allowed to settle during loading to ensure uniform concentration of embryos/vessel.

5/24/03. pH, DO, temp, & salinity were determined from "dummy chemistry" replicates for test & Ref Tox vessels. Vessels covered.

5/25/03 Water quality monitored in test and Ref Tox test vessels. Vessels remain covered. Volume of each vessel was reduced and subsamples placed in Sedgwick-Rafter cell for enumeration.

C6

MACTEC Engineering and Consulting, Inc.
Gainesville, Florida

QA Form No.: 097
Revised: March 2001

SUBJECT: TOXICITY TEST SYSTEM DESCRIPTION SHEET

Sponsor: Ponce Harbor

Project No.: 312253

TEST MATERIAL

Test Material: ~~Effluent~~ Elutriate

See Page 652-657 of Effluent Log for Test Material Information.

PROTOCOL/ GUIDELINES FOLLOWED:

Test Concentrations Based on: ( ) Active Ingredient
(x) Whole Material

TEST ORGANISM HISTORY

Test Species: mytilus edulis

Lot Number: 2003-42

Age/ Life Stage: adult

Date Acclimation/ Maintenance Began: 5/20/03

See Page 108 of Organisms Log for Data.

Mortality (%) During 48 Hours Prior to Test Initiation: <1%

TEST CONDITIONS

( ) Range Finder
(x) Screening
() Definitive

(X) Static
( ) Flow-Through

Lighting: (x) Fluorescent ( ) Incandescent

Photoperiod: 16 Hours Light
8 Hours Dark

Test Duration: 48-72 hrs

Test Area Used: Water bath, chilled water

Test Temperature (°C): 16 +/- 1

Test Salinity (ppt): 30 +/- 3

Replicates Per Concentration: 5

Organisms Per Replicate: 20-30 embryos/mL

Dilution Water: Natural filtered seawater

TEST VESSELS

Dimensions (cm): 8 Length x 8 Width x 10 Height

Test Solution Height (cm) : 9

Test Vessels: ( ) Open (x) Covered -if salinity increases

Test Container Volume: 250 mL

Test Solution Volume: 200 mL

TEST SOLUTION PREPARATION

Test Concentration %	Control	0	10	50	100				
Volume of Dilution Water Added (mL)	1500	1500	1350	750	-				
Volume of Effluent/ Stock Added (mL)	-	-	150	750	1500				

ADDITIONAL OBSERVATIONS:

DATA BY: JMM DATE: 5/23/03

MACTEC
Gainesville, Florida

QA Form Number: 016
Revised: March 2001

ACUTE TOXICITY DATA SHEET - WATER QUALITY DATA

SPONSOR: Ponce Harbor/ PPB

PROJECT NUMBER: 312253

TEST SUBSTANCE: Elutriate

TEST SPECIES: M. edulis

DATE		5/23/03				5/24/03				5/25/03							
TIME		1815				1100				1715							
INIT.		CG				JMW				JMW							
NOMIN CONCN % mg/L µg/L	REP	TEMP	PH	DO	SL	TEMP	pH	DO	SL	TEMP	pH	DO	SL	TEMP	pH	DO	SL
Seawater Control	A	17.5	8.0	8.3	30	~~16~~ JMW											
	B					16.5	7.8	7.9	30		JMW 7.9						
	C									16.8	~~8.2~~	8.2	29				
	D																
	E																
Ctrol-Sed 10%	A	17.0	8.0	8.0	28												
	B					16.1	8.1	7.8	30								
	C									16.8	8.1	8.1	28				
	D																
	E																
Ctrol-Sed 50%	A	17.0	7.9	8.6	28												
	B					16.1	8.1	7.7	30								
	C									16.8	8.1	8.0	30				
	D																
	E																
INST/METH		FS-6	SA2900	DO3	Ref 10	FS-6	SA2900	DO3	Ref 10	FS-6	SA2900	DO3	Ref 10				

Water quality recorded from "chem" replicate for ea conc.
JMW

KEY

TEMP - Temperature
SL - Salinity (ppt)
pH - standard units

DO - Dissolved oxygen (mg/L)
CN - Conductivity (µmhos/cm)
INST - Instrument
NOMIN CONCN - Nominal concentration

INIT. - Initials
REP - Replicate
METH - Method

CG

MACTEC
Gainesville, Florida

QA Form Number: 016
Revised: March 2001

ACUTE TOXICITY DATA SHEET - WATER QUALITY DATA

SPONSOR: Ponce Harbor/ PPB

PROJECT NUMBER: 312253

TEST SUBSTANCE: Elutriate

TEST SPECIES: M. edulis

DATE		5/23/03				5/24/03				5/25/03							
TIME		1815				1100				1715							
INIT.		CG				[illegible]				[illegible]							
NOMIN CONCN % mg/L µg/L	REP	TEMP	PH	DO	SL	TEMP	pH	DO	SL	TEMP	pH	DO	SL	TEMP	pH	DO	SL
Ctrol-Sed 100%	A	17.0	7.8	7.6	27												
	B	~~16.1~~ [illegible]	~~8.0~~	~~7.4~~	~~29~~	16.1	8.0	7.4	29								
	C									16.8	8.1	8.0	28				
	D																
	E																
E-PH03-SC 10%	A	17.0	8.0	8.6	28												
	B					16.1	8.1	7.6	30								
	C									16.9	8.1	8.0	29				
	D																
	E																
E-PH03-SC 50%	A	16.8	8.0	8.4	28												
	B					16.1	8.1	7.7	30								
	C									16.9	8.1	7.9	29				
	D																
	E																
INST/METH		FS-6	SR2000	DO3	Ref 10	FS-6	SR2000	DO3	Ref 10	FS-6	SR2000	DO3	Ref 10				

Water quality from monitored "chem" replicate [illegible]

KEY

TEMP - Temperature
SL - Salinity (ppt)
pH - standard units

DO - Dissolved oxygen (mg/L)
CN - Conductivity (µmhos/cm)
INST - Instrument
NOMIN CONCN - Nominal concentration

INIT. - Initials
REP - Replicate
METH - Method

CC

MACTEC
Gainesville, Florida

QA Form Number: 016
Revised: March 2001

ACUTE TOXICITY DATA SHEET - WATER QUALITY DATA

SPONSOR: Ponce Harbor/ PPB

PROJECT NUMBER: 312253

TEST SUBSTANCE: Elutriate

TEST SPECIES: M. edulis

DATE		5/23/03				5/24/03				5/25/03							
TIME		1815				1100				1715							
INIT.		CC				JM				JMR							
NOMIN CONCN % mg/L µg/L	REP	TEMP	PH	DO	SL	TEMP	pH	DO	SL	TEMP	pH	DO	SL	TEMP	pH	DO	SL
E-CH03-SC 100%	A	17.0	8.0	7.9	27												
	B					16.1	8.1	7.5	30								
	C									16.8	8.2	7.9	28				
	D																
	E																
	A																
	B																
	C																
	D																
	E																
	A																
	B																
	C																
	D																
	E																
INST/METH		FS-6	SA2900	DO3	Ref 10	FS-6	SA2900	DO3	Ref 10	FS-6	SA2900	DO3	Ref 10				

KEY

TEMP - Temperature
SL - Salinity (ppt)
pH - standard units

DO - Dissolved oxygen (mg/L)
CN - Conductivity (µmhos/cm)
INST - Instrument

INIT. - Initials
REP - Replicate
METH - Method
NOMIN CONCN - Nominal concentration

CG

MACTEC Engineering & Consulting, Inc. (formerly Harding ESE)
Gainesville, Florida

QA Form: 015d
Revised: November 2002

SUBJECT: ACUTE TOXICITY DATA SHEET – SURVIVAL

Sponsor: Ponce Harbor/PPB

Project No.: 312253

Test Substance: Sediment

Test Species: M. edulis

TEST HOUR		0-HOUR		24-HOURS		48-HOURS		72-HOURS	
DATE		5/23/03		5/24/03					
TIME		1815		1100					
TECHNICIAN		CG JMR		CG JMR					
NOMINAL CONCENTRATION mg/L %	REP	OBSERV.	Embryo per vessel (ml)	OBSERV.	Embryo per vessel (ml)	OBSERV.	Embryo per vessel (ml)	OBSERV.	Embryo per vessel (ml)
Seawater Ctrl	A	N	1ml	N					
	B								
	C								
	D								
	E								
Sed Control 10%	A								
	B								
	C								
	D								
	E								
Sed Control 50%	A								
	B								
	C								
	D								
	E								
Sed Control 100%	A								
	B								
	C								
	D								
	E	N	N	N					

MACTEC Engineering & Consulting, Inc. (formerly Harding ESE)
Gainesville, Florida

QA Form: 015d
Revised: November 2002

SUBJECT: ACUTE TOXICITY DATA SHEET – SURVIVAL

Sponsor: Ponce Harbor/PPB

Project No.: 312253

Test Substance: Sediment

Test Species: M. edulis

TEST HOUR		0-HOUR		24-HOURS		48-HOURS		72-HOURS	
DATE		5/23/03		5/24/03					
TIME		1815		1100					
TECHNICIAN		[illegible]		[illegible]					
NOMINAL CONCENTRATION mg/L %	REP	OBSERV.	Embryo per vessel (ml)	OBSERV.	Embryo per vessel (ml)	OBSERV.	Embryo per vessel (ml)	OBSERV.	Embryo per vessel (ml)
E-PH03-SC 10%	A	N	1ml	N	[illegible]				
	B								
	C								
	D								
	E								
E-PH03-SC 50%	A								
	B								
	C								
	D								
	E								
E-CH03-SC 100%	A								
	B								
	C								
	D								
	E	N	1ml	N	[illegible]				
	A								
	B								
	C								
	D								
	E								

Bivalve Embryo Test Final Counts

Date 5/25/03 Analyst Shankle
Sample # PONCE Reference Toxicant

PG 2 of 2

Initial Counts				Average
Begin	See page 1			
Middle				
End				
Final				

Final Counts

Rep #	Volume mLs	Complete Shell	Malformed Shell	Total Live	Deformed or trochophore	Unfert. eggs	Empty	
E-PH03-x 10% 5E	1	76	7	101	12	6	2	
50% 1A	1	65	10	90	11	4	10	
2B	1	80	1	95	14	0	12	
3C	1	60	4	88	19	5	11	
4D	1	62	7	93	18	6	9	
5E	1	60	9	81	10	2	9	
100% 1A	1	58	2	92	27	5	12	
2B	1	60	3	89	20	6	8	
3C	1	50	8	83	18	7	8	
4D	1	61	7	90	15	7	2	
5E	1	50	4	83	20	9	4	

Bivalve Embryo Test Final Counts

Date 5/25/03 Analyst [illegible]
Sample # PONCE Reference Toxicant
E-PH03-SC/SEAWATER CONTROL/E-PH03-CONTROL

	① Initial Counts	②	③	Average	
Begin	26/30	31/25	30/27	28.2/mL	169
Middle	30/34	28/20	31/24	27.8	167
End	24/19	24/22	19/27	22.8	137
Final				26.3	473

		live and complete		Final Counts	other live		dead	
Rep #	Volume mLs	Complete Shell	Malformed Shell	Total Live	Deformed or trochophore	Unfert. eggs	Empty	
Seawater Control A	1	68	6	101	20	7	4	
B	1	80	3	96	10	3	8	
C	1	79	3	107	19	6	0	
D	1	69	4	97	16	8	3	
E	1	71	6	95	18	0	5	
EPH03 CONT 10% A	1	70	2	103	26	5	0	
B	1	60	7	93	22	4	5	
C	1	78	5	98	14	1	5	
D	1	70	4	90	12	4	7	
E	1	73	0	97	19	5	9	
50% A	1	71	6	100	21	2	8	
B	1	79	5	106	19	3	4	
C	1	61	3	91	20	7	10	
D	1	69	3	93	19	2	6	
E	1	65	5	96	18	8	10	
100% A	1	60	9	101	30	2	7	
B	1	59	4	90	26	1	10	
C	1	72	7	92	9	4	3	
D	1	56	12	89	15	6	8	
E	1	66	6	94	18	4	2	
E-PH03-SC 100% A	1	69	9	89	10	1	8	
B	1	63	0	103	29	11	8	
C	1	71	6	98	17	4	4	
D	1	62	7	92	19	4	9	

MACTEC
Toxicology Laboratory
Gainesville, Florida

QA Form No: 1130a
Effective Date: April 2002

Sponsor: Ponce Harbor Project # 312253 96-hr Elutriate test w/ *M. edulis*

Sample ID	CTRL-PH03 10%				E-PH03-SC 10%				ctrL-PH03-saltwater			
Test Day	NH_3-N (ppm)	pH	Salinity (ppt)	Temp (°C)	NH_3-N (ppm)	pH	Salinity (ppt)	Temp (°C)	NH_3-N (ppm)	pH	Salinity (ppt)	Temp (°C)
Overlying Water Day, Date												
0 5/23/03	0.086 ~~0.228~~	7.7	30	20.0	0.228	7.6	30	20	0.036	7.8	30	20
1												
2												
3												
4 5/24/03	0.032	7.7	30	20.	0.209	7.6	30	20	0.183	7.8	30	20
5												
6												
7												
8												
9												
10												
Post Test												

Data By: [illegible] 5/2/03

MACTEC
Toxicology Laboratory
Gainesville, Florida

QA Form No: 1130a
Effective Date: April 2002

Sponsor: Ponce Harbor Project # 312253 96-hr Elutriate test w/ *M. edulis*

Sample ID	CTRL-PH03 50%				E-PH03-SC 50%							
Test Day	NH_3-N (ppm)	pH	Salinity (ppt)	Temp (°C)	NH_3-N (ppm)	pH	Salinity (ppt)	Temp (°C)	NH_3-N (ppm)	pH	Salinity (ppt)	Temp (°C)
Overlying Water Day, Date												
0 5/23/03	.480	7.8	30	20	2.02	7.7	30	20				
1												
2												
3												
4 5/27/03	0.500	7.8	30	20	1.60	7.7	30	20				
5												
6												
7												
8												
9												
10												
Post Test												

Data By: James H. [illegible] 5/2/03

MACTEC
Toxicology Laboratory
Gainesville, Florida

QA Form No: 1130a
Effective Date: April 2002

Sponsor: Ponce Harbor Project # 312253 96-hr Elutriate test w/ *M. edulis*

Sample ID	CTRL-PH03 100%				E-PH03-SC 100%							
Test Day	NH_3-N (ppm)	pH	Salinity (ppt)	Temp (°C)	NH_3-N (ppm)	pH	Salinity (ppt)	Temp (°C)	NH_3-N (ppm)	pH	Salinity (ppt)	Temp (°C)
Overlying Water Day, Date												
0 5/23/03	1.73	7.8	30	20	5.43	7.7	30	20				
1												
2												
3												
4 5/27/03	1.04	7.8	30	20	4.94	7.7	30	20				
5												
6												
7												
8												
9												
10												
Post Test												

Data By: [illegible] 6/02/03

MACTEC Engineering and Consulting
TOXICOLOGY LABORATORY
GAINESVILLE, FLORIDA

PAGE:
FORM NO: 097A REFTOX
REVISED: MARCH 2001

REFERENCE TOXICANT TEST DATA SHEET

Sponsor: Misc.	Project Number: Ref Tox
Test Material	**Test Conditions**
Amount of CuSO4 : 0.1 (g) Volume of Milli-Q Water: 1000 (mL) Date Prepared: 5/23/03	[ ] Definitive [x] Static [X] Screening Duration: 48-72 Hours
Test Organism History	Dilution Water: natural filtered seawater
Species: M. edulis Batch Number: 2003-42 Age/Life Stage: Adult Date of Acclimation/Maintenance: 5/20/03 See Page 108 of Organism Receipt Log Mortality 48 hours prior to test: <1 (%) Replicates/Concentration: 5 No. Of Organisms/Replicate: a formula	Lighting: [x Fluorescent [ ] Incandescent Photoperiod: 16 Hr Light 8 Hr Dark Test Vessel Dimensions: 8 L X 8 W 10 H Test Solution Height: 9 (cm) Test Vessel Volume: 0.25 (L) Test Solution Volume: 0.20 (L) Test Temperature: 16 ± 2 °C Test Salinity: 30 ± 3 ppt
Test Area Used: waterbath with chilled water	Test Vessel Composition: [x] Glass [ ] Plastic
Test Concentrations Based on: [x] Whole Material [ ] Active Ingredient	Test Protocol/Guideline Followed: ASTM: E 724-94

Test Concentrations: (Units: µg/L)	Control	10	20	40	80	160
Amount Dilution Water Added (mL)	2000	1999.8	1999.6	1999.2	1998.4	1996.8
Amount of Stock Added (mL)	0	0.2	0.4	0.8	1.6	3.2

Additional Observations:

Data By: CG Date: 5/23/03

CG

REFERENCE TOXICANT TEST DATA: DAY 0 – 2

Client: Misc.	Project Number: Ref Tox	Species: *Mytilus edulis*

DAY 0 — REF TOXICANT: CuSO4

Nominal Concentration (mg/L)	Date: 5/23/03		Time: 1830		Analyst: [illegible]	
	Embryo per vessel (ml)	Observ.	Temp. (°C)	Sal (ppt)	DO (mg/L)	pH
Seawater Control	1 ml	N	17.5	30	8.3	8.0
10	↓	↓	17.5	30	8.2	8.1
20			16.6	30	8.2	8.1
40			16.7	30	8.2	8.1
80			16.7	30	8.2	8.1
160			16.7	30	8.1	8.2

DAY 1	Date: 5/24/03		Time: 1030		Analyst: [illegible]	
Nominal Concentration	Embryo per vessel (ml)	Observ.	Temp. (°C)	Sal (ppt)	DO (mg/L)	pH
Seawater Control		N	16.5	30	7.9	7.8
10		N	16.4	30	7.7	7.9
20		N	16.3	30	7.7	8.0
40		N	16.1	30	7.7	8.0
80		N	16.1	30	7.7	8.0
160		N	16.0	30	7.7	8.0

DAY 2	Date: 5/25/03		Time: 1700		Analyst: [illegible]	
Nominal Concentration	Embryo per vessel (ml)	Observ.	Temp. (°C)	Sal (ppt)	DO (mg/L)	pH
Seawater Control		N	16.9	29	~~7.9~~ 8.2	7.9
10		N	16.9	30	8.0	8.0
20		N	16.8	30	8.0	8.0
40		N	16.8	30	8.0	8.1
80		N	16.8	30	8.0	8.1
160		N	16.9	30	7.9	8.1

Bivalve Embryo Test Final Counts

Date 5/25/03 Analyst Shintelle
Sample # _____ Reference Toxicant $CuSO_4$

Initial Counts				Average
Begin				
Middle	see test	run -	same date	
End				
Final				

Final Counts

Rep #	Volume mLs	Complete Shell	Malformed Shell	Total Live	Deformed or trochophore	Unfert. eggs	Empty	
Control A	1	68	6	101	20	7	4	
B	1	80	3	96	10	3	8	
C	1	79	3	107	19	6	0	
10 A	1	70	8	95	17	0	4	
B	1	81	4	100	10	5	5	
C	1	72	6	96	12	6	2	
20 A	1	63	9	90	12	6	0	
B	1	60	4	86	20	2	10	
C	1	52	2	95	33	8	6	
40 A	1	18	8	36	7	3	6	
B	1	11	5	34	12	6	10	
C	1	15	5	33	10	3	10	
80 A	1	2	4	10	4	0	1	
B	1	10	1	19	8	0	0	
C	1	0	0	2	1	1	0	
160 A	1	0	0	0	0	0	0	
B	1	0	0	0	0	0	0	
C	1	0	0	0	0	0	0	

Aquatic Research Organisms

DATA SHEET

I. Organism History

Species: Mytilus edulis

Source: Lab reared_____ Hatchery reared_____ Field collected ✓

Hatch date_____ Receipt date 5/19/03

Lot number 051903ME Strain WICD

Brood Origination HAMPTON NH

II. Water Quality

Temperature 12 °C Salinity 30 ppt DO SAT

pH 8.2 Hardness — ppm

III. Culture Conditions

System: FLOW THROUGH

Diet: Flake Food_____ Phytoplankton ✓ Trout Chow_____

Brine Shrimp_____ Rotifers_____ Other_____

Prophylactic Treatments:_____

Comments:_____

received 5/20/03 J. Ryan

IV. Shipping Information

Client: ESE/MATRC # of Organisms: 50+

Carrier: FED EX Date Shipped: 5/19/03

Biologist: [signature]

Force elutriate (gamete test)

Inv# 120362
PO# 5634

1 - 800 - 927 - 1650

PO Box 1271 • One Lafayette Road • Hampton, NH 03842 • (603) 926-1650

Environmental Science & Engineering, Inc.
Aquatic Toxicology Laboratory
Gainesville, Florida

ESE QA Form No.: 008
Effective: Mar 1993

SUBJECT: ORGANISM RECEIPT/COLLECTION LOG

Date Recieved	Species	Population Size	Population Condition	Collector/ Supplier	Salinity/ Hardness	Temperature (C)	Initials
5/1/03	N. Ulfens	2003-32 375	normal	ARO	—	13.0	KG
5/2/03	C. Leedsi	2003-33 200+	normal (dry condition)	MBL	—	24.1	AEB
5/3/03	C. Virginica	2003-34 50-60	normal	NW Seafood	—	8.3	JMR
5/6/03	M. Nasuta	2003-35 ~1000	Dry-DEAD (Hot)	ARO	—	19.2	JA
~~5/6~~ 5/7/03	M. bahia	2003-36 ~2000	normal	Aquatic Indicators	26.	23.6	CG
5/9/03	L. Plumalosa	2003-37 +2000	normal	ARO	20	18.6	JA
5/14/03	M. bahia	2003-38 2500+	normal	~~AI~~ Aquatic Indicators	21	21.9	JA
5/15/03	M. bahia	2003-39	normal	~~AI~~ Aquatic Indicators	20	22.1	DD
5/16/03	M. nasuta	2003-40 ~1000	normal	ARO	—	5.2	CG
5/16/03	M. benplina	2003-41 ~3000	normal	Aquatic Indicators	20	22.5	CG
5/20/03	M. edulis	2003-42 ~50-60	normal	ARO	—	12.1	JMR adult

Harding ESE
Toxicology Laboratory
Gainesville, Florida

Page: 76
QA Form No. 009
Revised: March 2001

SUBJECT: DAILY RECORD OF INVERTEBRATE HOLDING CONDITIONS

Species: M. edulis
Lot Number: 2003-42
Date collected/received: 5/20/03
Date of hatch: adult
Collection location/supplier: ARO
Page 108 of animal receipt and collection log.
Data continued from Page — of this log book.

Date	5/20/03				
Time	1400				
Technician	JMM				
Number alive	~50 to 60				
Number dead	<1%				
Hardness/Salinity	—				
Method/Meter	—				
Temperature (EC)	12.1				
Meter number	F56				
D.O. (mg/L)	—				
Meter number	—				
pH (s.u.)	—				
Meter number	—				
Water	no				
Food	no				
Clean	—				
Number removed	Ø				
Project No. / Task	312253				

_____ Data continued on Page _____ of this log book.
_____ Population was terminated. Date_____ How_____

Comments: Mytilus held in cold room ±4°C until used for test on 5/23/03.

CR

Appendix C

MACTEC Engineering & Consulting, Inc.
Toxicology Laboratory
Gainesville, Florida

QA Form No. 018
Revised: October 2002

Project No: 312253 Client: Ponce Harbor/ PPB Test: whole sediment M. bahia

DAILY LOG

3/27/03 Test and control sediment loaded into test vessels. Airlines set up and aeration started. Vessels contents allowed to settle overnight.

~~Organisms fed while in holding.~~

3/28/03 old water qualities monitored water renewed and new water qualities taken. Organisms loaded and fed. Airlines checked. Pore water + overlying water samples collected from each concentration.

3/29/03 old water qualities monitored. Water renewed in the am, overlying water for ammonia samples taken. New water qualities taken after water was renewed. airlines checked. Organisms fed. Organisms monitored

3/30/03 Old water qualities monitored. water renewed. Overlying water sample for ~~Am~~ ammonia ~~samples~~ taken. Organisms fed. pm water renewal and new water qualities taken. Airlines checked

3/31/03 old water qualities taken and water renewed. Overlying water samples for ammonia ~~samples~~ taken. New water qualities taken and all water renewed. Organisms monitored and fed. Airlines checked

4/1/03 old water qualities taken and water renewed. overlying water collected for ammonia. New water qualities taken. Organisms observed + fed. Air lines checked.

JMC

MACTEC Engineering & Consulting, Inc.
Toxicology Laboratory
Gainesville, Florida

QA Form No. 018
Revised: October 2002

Project No: 312253 Client: Ponce Harbor/ PPB Test: Whole Sediment M. bahia

DAILY LOG

4/2/03 Old water quality monitored. Water renewed. New water quality monitored. Organisms observed. Overlying water sample collected for ammonia determination. Organisms fed. Airlines checked.

4/3/03 Old water qualities taken. Overlying water for Ammonia samples taken. Organisms observed. New water qualities taken. Organisms fed. Airlines checked

4/4/03 Old water qualities monitored. Overlying water samples for Ammonia ~~samples~~ taken and water renewed in the AM. New water qualities taken after water renewed in the pm. Air lines checked. Organisms fed and monitored

4/5/03 Old water qualities monitored. Overlying water samples for Ammonia ~~samples~~ taken and water renewed in the AM. Water renewed again in the pm and new water qualities taken. Air lines checked Organisms fed and observed

4/6/03 Old water qualities taken. Overlying water samples for Ammonia ~~samples~~ taken. Organisms where observed New water qualities taken first renewed air line checked. organisms fed.

4/7/03. Water quality monitored. Overlying water sample collected from each concentration for ammonia determination. Organisms fed. Organisms observed and counted. Test terminated.

NOTE: All pore water samples were collected ~~from dummy~~ ~~replicates~~ at time of test renewal

MACTEC Engineering and Consulting, Inc.
Gainesville, Florida

QA Form No.: 097
Revised: March 2001

SUBJECT: TOXICITY TEST SYSTEM DESCRIPTION SHEET

Sponsor: Ponce Harbor/ PPB

Project No.:

TEST MATERIAL

Test Material: Sediment

See Page 652-657 of Effluent Log for Test Material Information.

PROTOCOL/ GUIDELINES FOLLOWED:

Test Concentrations Based on: ( ) Active Ingredient
(x) Whole Material

TEST ORGANISM HISTORY

Test Species: Mysiopsis bahia

Lot Number: 2003-22

Age/ Life Stage: 3 days

Date Acclimation/ Maintenance Began: 3/28/03

See Page 107 of org. receipt Log for Data.

Mortality (%) During 48 Hours Prior to Test Initiation: <1

TEST CONDITIONS

( ) Range Finder
(X) Screening
( ) Definitive

(X) Static
( ) Flow-Through

Lighting: (x) Fluorescent ( ) Incandescent

Photoperiod: 16 Hours Light
8 Hours Dark

Test Duration: 10 days

Test Area Used: Water bath

Test Temperature (°C): 20 +/- 2

Test Salinity (ppt): 30 +/- 2

Replicates Per Concentration: 5

Organisms Per Replicate: 20

Dilution Water: natural filtered seawater

TEST VESSELS

Dimensions (cm): Length x Width x Height

Test Solution Height (cm) :

Test Vessels: (x) ~~Open~~ (X) Covered

Test Container Volume: 1 L

Test Solution Volume: 725 mL

TEST SOLUTION PREPARATION

Test Concentration % mg/L									
Volume of Dilution Water Added (mL)									
Volume of Effluent/ Stock Added (mL)									

ADDITIONAL OBSERVATIONS:

200 mL of sediment

DATA BY: HOB

DATE: 3/28/03

MACTEC Engineering and Consulting, Inc.
Gainesville, Florida

QA Form No. 106
Effective: July 2002

DAILY FEEDING LOG

Sponsor: Ponce Harbor/ PPB

Project No.: 312253

Test Substance: whole sediment

Test Species: M. bahia

TEST DAY	DATE	TIME	INITIALS	FOOD TYPE	QUANTITY	COMMENTS
0	3/28/03	1600	HEB	Artemia nauplii (BS)	.2mL	
1	3/29/03	1700	HEB	BS	.2mL	
2	3/30/05	1115	KG	BS	.2mL	
3	3/31/03	1700	DD	BS	.2ml	
4	4/1/03	1620	KG	BS	.2mL	
5	4/02/03	1600	DD	BS	.2ml	
6	4/3/03	1600	KG	BS	.2ml	
7	4/4/03	1600	HEB	BS	.2mL	
8	4/5/03	1630	HEB	BS	.2mL	
9	4/6/03	1630	JA	BS	.2mL	
10	4/7/03	1000	DD	BS	.2ml	

BS= Brine Shrimp nauplii YTC= Yeast/Trout Chow/Cereal Leaves Algae= Green Algae (*Selenastrum capricornutum*)

MACTEC
Toxicology Laboratory
Gainesville, Florida

QA Form No: 1130a
Effective Date: April 2002

Sponsor: Ponce Harbor Project # 312253 Whole Sediment M. bahia

Sample ID	CTRL-PH03				RFS – PH03				E-PH03-SC			
Test Day	NH_3-N (ppm)	pH	Salinity (ppt)	Temp (°C)	NH_3-N (ppm)	pH	Salinity (ppt)	Temp (°C)	NH_3-N (ppm)	pH	Salinity (ppt)	Temp (°C)
Pore Water Date: 3/28/03	10.8	8.2		19.5	7.2	8.4		19.1	28.8	8.3	20.1	
Overlying Water Day, Date	NH_3-N union. amm.				NH_3-N union. amm.				NH_3-N union. amm.			
0 3/28/03	0.996 / 0.059	8.2	30	19.5	1.17 / 0.060 to 0.070	8.2	31	19.1	7.3 / 0.431	8.2	20.1	31
1 3/29/03	0.624 / 0.037	8.2	31	19.5	0.794 / 0.041 to 0.047	8.2	31	19.1	0.962 / 0.049 to 0.057 to 0.087	8.3	19.4	32
2 3/30/03	0.708 / 0.042	8.2	31	19.5	0.834 / 0.049 to 0.066 to 0.075	8.3	31	19.4	6.18 / 0.365 / 0.559	8.3	19.5	31
3 3/31/03	0.67 / 0.040	8.2	32	19.7	0.708 / 0.042	8.3	32	19.7	5.81 / 0.525	8.4	19.7	32
4 4/1/03	—	—	32	—	—	CG ~~8.3~~ 4/3/03	32	CG ~~19.4~~ 4/3/03	4.64 / 0.320	8.3	19.4	32
5 4/2/03	—	—	31	—	—	-	32	—	3.86 / 0.266	8.3	19.3	31
6 4/3/03	—	—	30	—	-	-	30	—	2.32 / 0.161	8.3	19.3	30
7 4/4/03	-			—	—	—	—	—	.765 / 0.062	8.2	25.0	31
8 4/5/03	—	—	—	—	—	—	—	—	.730 / 0.060	8.2	25.0	31
9 4/6/03	—	—	—	—	—	—	—	—	.534 / 0.044	8.2	25.0	31
10 4/7/03	—	—	-	—	—	—	—	—	.406 / 0.033	8.2	25.0	31
Post Test	—	—	—	—	—	—	—	—				

CG 4/1/03 CG 4/1/03

jm

MACTEC Engineering & Consulting, Inc.
TOXICOLOGY LAB: GAINESVILLE, FL

QA FORM NO: 108
Revised: Jan 2003

SUBJECT: SEDIMENT TOXICITY DATA SHEET

SPONSOR: Ponce Harbor/ PPB
SAMPLE ID: PH03-CTRL

PROJECT NUMBER: 312253
TEST SPECIES: m. bahia

DATE-DAY	REP	TEMP (°C) old	TEMP (°C) new	Hard (mg/L)	NH_3 (ppm)	pH (s.u.) old	pH (s.u.) new	DO (mg/L) old	DO (mg/L) new	Salinity (ppt) old	Salinity (ppt) new	INITIAL/ TIME old	INITIAL/ TIME new
3/28/03 0	A	18.8	19.3			8.0	7.8	7.2	7.4	30	30	HEB 1115	DD 1500
3/29/03 1	B	18.7	19.5			8.2	8.1	7.9	7.8	31	31	HEB 1130	KG 1700
3/30/03 2	C	18.9	19.2			8.0	8.3	8.2	8.1	31	31	1100 KG	HEB 1430
3/31/03 3	D	18.7	18.6			8.2	8.2	7.3	7.3	32	32	0920 CG	1600 CG
4/1/03 4	E	17.9	18.6			8.2	8.2	7.6	7.6	32	32	CG 0850	KG 1615
4/2/03 5	A	18.4	18.9			8.2	7.9	7.3	7.6	31	30	DD 0850	DD 1600
4/3/03 6	B	18.7	19.5			8.2	8.3	7.8	7.5	30	30	KG 0920	KG 1530
4/4/03 7	C	19.2	20.4			8.2	8.3	7.6	7.3	30	30	DD 0850	HEB 1600
4/5/03 8	D	19.3	19.2			8.2	8.4	7.7	7.4	31	30	DD 910	HEB 1630
4/6/03 9	E	19.1	18.6			8.3	8.2	7.5	7.4	30	30	JA 930	JA 16:00
4/7/03 10	A	19.7				8.2		7.0		30		DD 930	

Observ Date-Day	Replicate A	B	C	D	E	Int.
3/28/03 0	20 loaded					HEB
3/29/03 1	N	N	N	N	N	HEB
3/30/03 2	N	N	N	N	N	KG
3/31/03 3	N	N	N	N	N	CG
4/1/03 4	N	N	N	N	N	CG
4/2/03 5	N	N	N	N	N	DD
4/3/03 6	N	N	N	N	N	KG
4/4/03 7	N	N	N	N	N	DD
4/5/03 8	N	N	N	N	N	DD
4/6/03 9	N	N	N	N	N	JA
4/7/03 10	19A, 1NF	18 A, 2NF	20	20	19, 1NF	JM

19A

KEY: AS = AT SURFACE EMR = EMERGENCE A = ALIVE D = DEAD NF = NOT FOUND
N=NONE REP = REPLICATE COND = CONDUCTIVITY ALK = ALKALINITY AMP = AMPLEXUS
HARD = HARDNESS TEMP = TEMPERATURE YTC = YEAST/ TROUT CHOW/CEROPHYLL

JM

MACTEC Engineering & Consulting, Inc.
TOXICOLOGY LAB: GAINESVILLE, FL

QA FORM NO: 108
Revised: Jan 2003

SUBJECT: SEDIMENT TOXICITY DATA SHEET

SPONSOR: Ponce Harbor/ PPB
SAMPLE ID: PH03-RFS

PROJECT NUMBER: 312253
TEST SPECIES: m. bahia

DATE-DAY	REP	TEMP (°C) old	TEMP (°C) new	Hard (mg/L)	NH_3 (ppm)	pH (s.u.) old	pH (s.u.) new	DO (mg/L) old	DO (mg/L) new	Salinity (ppt) old	Salinity (ppt) new	INITIAL/ TIME old	INITIAL/ TIME new
3/28/03 0	A	18.7	19.1			8.2	8.1	6.8	6.9	31	30	HEB 1115	DD 1500
3/29/03 1	B	18.7	19.4			8.2	8.2	7.5	7.9	32	31	HEB 1130	KG 1700
3/30/03 2	C	18.9	18.9			8.2	8.3	8.1	8.0	31	31	KG 1100	HEB 1430
3/31/03 3	D	18.0	18.4			8.3	8.2	7.6	7.4	32	32	CG 0920	1600 CG
4/1/03 4	E	17.6	18.6			8.2	8.1	7.6	7.6	32	32	CG 0850	KG 1615
4/2/03 5	A	18.6	19.2			8.0	7.8	6.3	6.6	32	30	DD 0905	DD 1600
4/3/03 6	B	18.7	19.6			8.1	8.2	7.6	7.5	30	30	KG 0920	KG 1530
4/4/03 7	C	19.2	19.9			7.9	8.1	7.1	6.8	30	30	DD 0850	HEB 1600
4/5/03 8	D	19.2	19.4			8.1	8.3	7.2	6.9	31	30	DD 0910	HEB 1630
4/6/03 9	E	19.2	19.5			8.0	8.2	7.3	6.5	30	30	JA 930	JA 16:00
4/7/03 10	A	19.9				8.2		7.1		30		DD 930	

Observ Date-Day	Replicate A	B	C	D	E	Int.
3/28/03 0	20 loaded					HEB
3/29/03 1	N	N	N	N	N	HEB
3/30/03 2	N	N	N	N	N	KG
3/31/03 3	N	N	N	N	N	CG
4/1/03 4	N	N	N	N	N	CG
4/2/03 5	N	N	N	N	N	DD
4/3/03 6	N	N	N	N	N	KG
4/4/03 7	N	N	N	N	N	DD
4/5/03 8	N	N	N	N	N	DD
4/6/03 9	N	N	N	N	N	JA
4/7/03 10	~~3A~~ 3NF	19A 1NF	1NF, 19A	17A, 3NF	20A	JM

17 Alive

KEY: AS = AT SURFACE EMR = EMERGENCE A = ALIVE D = DEAD NF = NOT FOUND
N=NONE REP = REPLICATE COND = CONDUCTIVITY ALK = ALKALINITY AMP = AMPLEXUS
HARD = HARDNESS TEMP = TEMPERATURE YTC = YEAST/ TROUT CHOW/CEROPHYLL

JM

MACTEC Engineering & Consulting, Inc.
TOXICOLOGY LAB: GAINESVILLE, FL

QA FORM NO: 108
Revised: Jan 2003

SUBJECT: SEDIMENT TOXICITY DATA SHEET

SPONSOR: Ponce Harbor/ PPB
SAMPLE ID: E-PH03-SC

PROJECT NUMBER: 312253
TEST SPECIES: m. bahia

DATE-DAY	REP	TEMP (°C) old	TEMP (°C) new	Hard (mg/L)	NH_3 (ppm)	pH (s.u.) old	pH (s.u.) new	DO (mg/L) old	DO (mg/L) new	Salinity (ppt) old	Salinity (ppt) new	INITIAL/ TIME old	INITIAL/ TIME new
3/28/03 0	A	18.6	19.2			8.2	8.1	7.1	6.7	31	30	HEB 1115	~~DD~~ 1500
3/29/03 1	B	18.6	19.3			8.2	8.2	7.4	7.8	32	31	HEB 1130	KG 1700
3/30/03 2	C	18.6	18.7			8.3	8.4	8.1	7.9	31	31	KG 1100	HEB 1430
3/31/03 3	D	18.7	18.4			8.3	8.2	7.7	7.7	32	32	CG 0920	1600 CG
4/1/03 4	E	17.8	18.5			8.2	8.2	7.5	7.5	32	32	CG 0850	KG 1615
4/2/03 5	A	18.3	19.3			8.1	8.0	7.5	5.9	31	30	~~DD~~ 0900	~~DD~~ 1600
4/3/03 6	B	18.6	19.6			8.2	8.2	7.8	7.3	30	30	KG 0920	KG 1530
4/4/03 7	C	19.2	19.7			8.1	8.2	~~7.6~~ 7.2 ~~DD~~	7.2	30	30	~~DD~~ 0850	HEB 1600
4/5/03 8	D	19.3	19.4			8.2	8.3	7.5	7.2	31	30	~~DD~~ 0910	HEB 1630
4/6/03 9	E	19.2	19.6			8.1	8.3	7.4	6.8	30	30	JA 0930	JA 1600
4/7/03 10	A	19.8	19			7.8		~~7.8~~ ~~DD~~ 4.9			30	~~DD~~ 930	

Observ Date-Day	Replicate A	B	C	D	E	Int.
3/28/03 0	20 loaded					HEB
3/29/03 1	N	N	N	N	N	HEB
3/30/03 2	N	N	N	N	N	KG
3/31/03 3	N	N	N	N	N	CG
4/1/03 4	N	N	N	N	N	CG
4/2/03 5	N	N	N	N	N	~~DD~~
4/3/03 6	N	N	N	N	N	KG
4/4/03 7	N	N	N	N	N	~~DD~~
4/5/03 8	N	N	N	N	N	~~DD~~
4/6/03 9	N	N	N	N	N	JA
4/7/03 10	20A	20A	20A	17A, 3NF	19A, 1NF	JM

KEY: AS = AT SURFACE EMR = EMERGENCE A = ALIVE D = DEAD NF = NOT FOUND
N=NONE REP = REPLICATE COND = CONDUCTIVITY ALK = ALKALINITY AMP = AMPLEXUS
HARD = HARDNESS TEMP = TEMPERATURE YTC = YEAST/ TROUT CHOW/CEROPHYLL

JM

MACTEC Engineering and Consulting
TOXICOLOGY LABORATORY
GAINESVILLE, FLORIDA

PAGE:
FORM NO: 097A REFTOX
REVISED: MARCH 2001

REFERENCE TOXICANT TEST DATA SHEET

Sponsor: Misc.	Project Number: Ref Tox
Test Material	**Test Conditions**
Amount of SDS : 0.1 (g) Volume of Milli-Q Water: 100 (mL) Date Prepared: 3/28/03	[X] Definitive [X] Static [ ] Screening Duration: 96 Hours
Test Organism History	Dilution Water: Natural Filtered Seawater
Species: M. bahia Batch Number: 2003-22 Age/Life Stage: 3 days Date of Acclimation/Maintenance: 3/28/03 See Page 107 of Org. receipt Log Mortality 48 hours prior to test: <1 (%) Replicates/Concentration: 1 No. Of Organisms/Replicate: 10	Lighting: [X] Fluorescent [ ] Incandescent Photoperiod: 16 Hr Light 8 Hr Dark Test Vessel Dimensions: ___ L X ___ W ___ H Test Solution Height: ___ (cm) Test Vessel Volume: 0.340 (L) Test Solution Volume: 0.200 (L) Test Temperature: 20 ± 1 °C Test Salinity: 20 ± 2 ppt
Test Area Used: Waterbath	Test Vessel Composition: [X] Glass [ ] Plastic
Test Concentrations Based on: [X] Whole Material [ ] Active Ingredient	Test Protocol/Guideline Followed:

Test Concentrations: (Units: mg/L)	Control	2.5	5.0	10.0	20.0	40.0	
Amount Dilution Water Added (mL)	200	199.5	199	198	196	192	
Amount of Stock Added (mL)	0	0.5	1	2	4	8	

Additional Observations: ___

Data By: HEB Date: 3/28/03

REFERENCE TOXICANT TEST DATA: DAY 0 – 2

Client: Misc.	Project Number: Ref Tox	Species: M. bahia

DAY 0 — REF TOXICANT: SDS

Nominal Concentration (mg/L)	Date: 03/28/03		Time: 1500		Analyst: [illegible]	
	# Alive	Observ.	Temp. (°C)	Sal (ppt)	DO (mg/L)	pH
Control	10	loaded	20.1	19	6.5	7.4
2.5	↓	↓	20.0	19	6.4	7.3
5			20.0	19	6.5	7.3
10			20.0	19	6.5	7.3
20			20.0	19	6.5	7.3
40			20.0	19	6.5	7.3

DAY 1	Date: 3/29/03		Time: 1100		Analyst: HEB	
Nominal Concentration	# Alive	Observ.	Temp. (°C)	Sal (ppt)	DO (mg/L)	pH
Control	10	N	19.7	20	7.7	8.2
2.5	9	1d	19.8	20	7.7	8.2
5	9	1d	19.8	20	7.8	8.2
10	6	4d	19.8	20	7.8	8.2
20	0	10d	19.9	20	7.8	8.3
40	0	10d	20.1	19	7.9	8.2

DAY 2	Date: 3/30/03		Time: 1400		Analyst: HEB	
Nominal Concentration	# Alive	Observ.	Temp. (°C)	Sal (ppt)	DO (mg/L)	pH
Control	10	N	20.0	20	7.0	8.4
2.5	9	N	20.2	20	7.5	8.4
5	7	2d	20.1	20	7.5	8.4
10	3	3d	20.2	20	6.9	8.3
20						
40						

MACTEC Engineering and Consulting, Inc.
TOXICOLOGY LABORATORY
GAINESVILLE, FLORIDA

QA FORM NO: 097 REFTOX
Revised: March 2001

REFERENCE TOXICANT TEST DATA: DAY 3 AND 4

Client: Misc.	Project Number: Ref Tox	Species: M. bahia

DAY 3 SDS

Nominal Concentration	Date: 03/31/03		Time: 0940		Analyst: ~~DD~~	
	# Alive	Observ.	Temp. (°C)	Sal (ppt)	DO (mg/L)	pH
Control	~~9~~ 10	N	19.4	20	7.9	8.4
2.5	9	N	19.3	20	7.9	8.3
5	7	N	19.2	20	8.0	8.4
10	1 ~~2d~~ ~~DD~~	2d	19.4	20	7.7	8.3
			~~19.4~~ ~~DD~~			

DAY 4	Date: 4/1/03		Time: 1000 *		Analyst: CG	
Nominal Concentration	# Alive	Observ.	Temp. (°C)	Sal (ppt)	DO (mg/L)	pH
Control	10	N	19.0	20	8.2	8.4
2.5	6 ~~t~~ CG 4/1/03	~~N~~ CG 4/1/03 3d	19.1	20	7.9	8.4
5	5	2d	19.0	20	8.2	8.4
10	1	N	19.0	20	8.2	8.4
20	—	—	—	—	—	—
40	—	—	—	—	—	—

SUMMARY

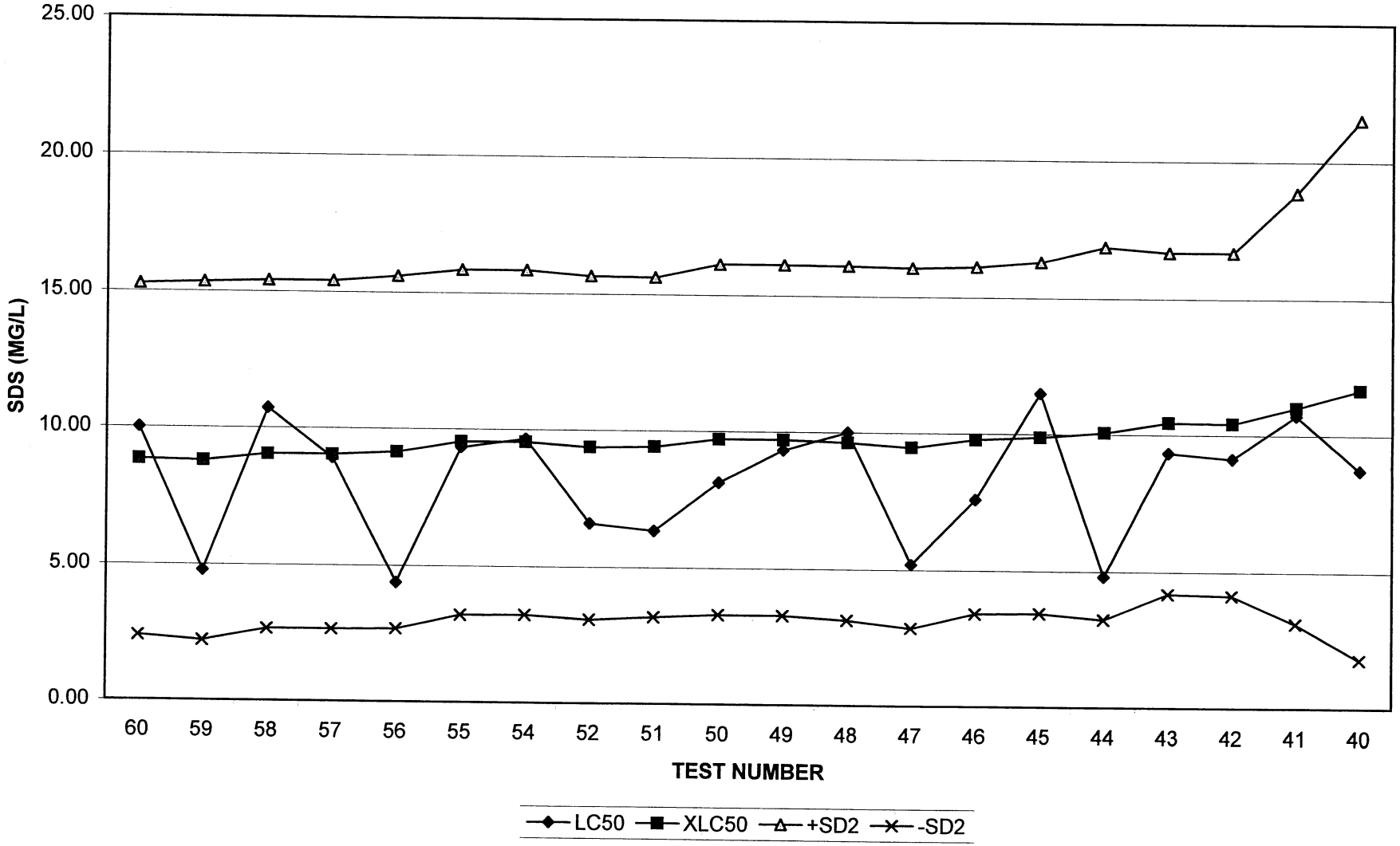
Nominal Concentration	Number Dead
Control	0
2.5	4
5.0	5
10.0	9
20.0	10
40.0	10

COMMENTS:

* WQ monitored in morning. ~~and~~ Organisms counted and test terminated @ 1605

Jm

MYSIDOPSIS BAHIA
ACUTE REFERENCE TOXICITY TEST:SODIUM LAURYL SULFATE



8/28/2003

TRIMMED SPEARMAN-KARBER METHOD. VERSION 1.5

DATE: 04/01/03 TEST NUMBER: Ponce-Wh DURATION: 96 h
TOXICANT : SDS
SPECIES: M. bahia

RAW DATA:

Concentration (mg/L)	Number Exposed	Mortalities
.00	10	0
2.50	10	4
5.00	10	5
10.00	10	9
20.00	10	10
40.00	10	10

SPEARMAN-KARBER TRIM: 40.00%

SPEARMAN-KARBER ESTIMATES: LC50: 4.39
95% LOWER CONFIDENCE: 1.69
95% UPPER CONFIDENCE: 11.39

Aquatic Research Organisms

DATA SHEET

I. Organism History

Species: AMERICAMYSIS BAHIA (mysiopsis bahia)

Source: Lab reared ✓ Hatchery reared ____ Field collected ____

Hatch date 3-25-03 Receipt date ____

Lot number 032503MS Strain ____

Brood Origination FLORIDA

II. Water Quality

Temperature 25 °C Salinity ≈28 ppt DO —

pH 7.8 Hardness — ppm

III. Culture Conditions

System: RECIRC

Diet: Flake Food ✓ Phytoplankton ____ Trout Chow ✓

Brine Shrimp ✓ Rotifers ____ Other Encap. Shrimp Diet

Prophylactic Treatments: ____

Comments: ____

IV. Shipping Information

Client: MACTEC # of Organisms: 450+

Carrier: FED X Date Shipped: 3-27-03

Biologist: Mark Rosengrist

APO Inv # 120225
PO # 5604

Ponce ~~Canaveral~~ Col
Wh. sed

1 - 800 - 927 - 1650

PO Box 1271 • One Lafayette Road • Hampton, NH 03842 • (603) 926-1650

[signature] 1400 3/28/03 lot (2003-22)

Environmental Science & Engineering, Inc.
Aquatic Toxicology Laboratory
Gainesville, Florida

ESE QA Form No.: 008
Effective: Mar 1993

SUBJECT: ORGANISM RECEIPT/COLLECTION LOG

Date Recieved	Species	Population Size	Population Condition	Collector/ Supplier	Salinity/ Hardness	Temperature (C)	Initials
3/18/03	M. nasuta	2003-16 ~500	normal	ARO	—	8.5	~~JMM~~ adult
3/21/03	Americamysis bahia	320+ 2003-17	normal	ARO	28	23.1	HEB 2-days
3/21/03	M. beryllina	320+ 2003-18	normal	ARO	27	23.1	HEB 10-days
3/23/03	M. bahia	375 2003-19	normal	Aquatic Indic.	20 ppm	25.0	JA ~~2 days~~
3/23/03	M. beryllina	885 2003-20	normal	Aquatic Indic.	20 ppm	25.0	JA 10 day
3/26/03	M. edulis	~48 2003-21	normal	ARO	—	11.3	~~JMM~~ adult
3/28/03	M. bahia	450+ 2003-22	normal	ARO	28 ppt	22.8	HEB 3days
3/31/03	Brine Shrimp Cysts	15 oz can	normal	Aquatic Eco-Systems	—	—	CG
4/2/03	M. nasuta	2003-23 1000	normal	ARO	—	12.7	CG adult
4/3/03	N. Virens	2003-24 500	normal	ARO	—	10.5	CG adult
4/4/03	L. plumulosus	2003-25 ~~±200~~ +300	normal	ARO	23 ppt	17.4	CG

Harding ESE
Toxicology Laboratory
Gainesville, Florida

Page: 61
QA Form No. 009
Revised: March 2001

SUBJECT: DAILY RECORD OF INVERTEBRATE HOLDING CONDITIONS

Species: M. bahia
Lot Number: 2003-22 Date collected/received: 3/28/03
Date of hatch: 3/25/03 Collection location/supplier: ARO
Page 107 of animal receipt and collection log.
Data continued from Page _____ of this log book.

Date	3/28/03				
Time	1400				
Technician	HEB				
Number alive	450				
Number dead	< 1%				
Hardness/Salinity	28				
Method/Meter	Ref 9				
Temperature (EC)	23.0				
Meter number	FS-6				
D.O. (mg/L)	>10				
Meter number	D03				
pH (s.u.)	7.7				
Meter number	SA290B				
Water	clear				
Food	BS				
Clean	✓				
Number removed	0				
Project No. / Task	312253				

_____ Data continued on Page _____ of this log book.
_____ Population was terminated. Date_____ How_____

Comments: test initiated; population healthy

CR

MACTEC Engineering & Consulting, Inc.
Toxicology Laboratory
Gainesville, Florida

QA Form No. 018
Revised: October 2002

Project No: 312253 Client: Ponce Harbor/ PPB Test: L. plumulosus, wh. Sed. Test

DAILY LOG

4/10/03. Sediments loaded, covered w/ 28 ppt saltwater. Aeration started. Reliminary: ~~pore~~ NH_3- let alone. Dummy replicates setup for pore water sample collection.

04/11/03 pay 0 water renewed. organisms loaded 20 per rep. airline checked water qualities taken. Pore water samples collected from dummy replicates.

4/12/03 pore ~~NH3~~ water samples taken. Old water qualities monitored Renewed, monitored Ref tox New water qualities monitored, air lines checked

4/13/03 water qualities taken. Pore ~~NH3~~ water samples taken from SC concentrations. water renewed. New water qualities monitored Airlines checked and organisms observed.

4/14/03 water qualities taken, ~~Pow NH3 taken~~ Pore water sample collected. water renewed. New water qualities monitored, airlines checked + Organisms observed.

4/15/03 water qualities taken. Pore, ~~NH3~~ Water sample collected, water renewed. New water qualities monitored, airlines checked and organisms observed.

4/16/03 water qualities taken Pore, ~~NH3~~ Water sample taken water renewed. Old water qualities monitored, airlines checked and organisms observed.

Jm

MACTEC Engineering & Consulting, Inc.
Toxicology Laboratory
Gainesville, Florida

QA Form No. 018
Revised: October 2002

Project No: 312253 Client: Ponce Harbor/ PPB Test: L. plumulosus, wh. Sed. Test

DAILY LOG

4/17/03 Old water qualities taken. ~~NH4~~ Pore water samples taken. Organisms observed. Water renewed. New water qualities taken. Airlines checked.

4/18/03 Old water qualities taken ~~NH4~~ Pore water Samples taken. Organisms observed. Water renewed. new water qualities taken. Airlines checked.

4/19/03 Old water qualities taken. ~~NH4~~ Pore water Samples taken. Organisms observed. water renewed. new water qualities taken. Airlines Checked.

4/20/03 Old water qualities taken. ~~NH4~~ Pore water Samples taken Organisms observed. water renewed. new water qualities taken. Airlines checked.

4/21/03 Old water qualities taken. ~~NH4~~ Pore water samples taken. Organisms Observed Airlines Checked.
Organisms counted, + test terminated.

NOTE: Pore water samples were collected from "dummy" replicates.

MACTEC Engineering and Consulting, Inc.
Gainesville, Florida

QA Form No.: 097
Revised: March 2001

SUBJECT: TOXICITY TEST SYSTEM DESCRIPTION SHEET

Sponsor: Ponce Harbor/ PPB

Project No.: 312253

TEST MATERIAL

Test Material: Sediment

See Page 652-657 of Effluent Log for Test Material Information.

PROTOCOL/ GUIDELINES FOLLOWED:

Test Concentrations Based on: ( ) Active Ingredient
(x) Whole Material

TEST ORGANISM HISTORY

Test Species: Leptochirus plumulosus

Lot Number: 2003 - 25

Age/ Life Stage: adult

Date Acclimation/ Maintenance Began: 4/9/03

See Page 107 of Organisms Receipt Log for Data.

Mortality (%) During 48 Hours Prior to Test Initiation: <1%

TEST CONDITIONS

( ) Range Finder
(X) Screening
( ) Definitive

(X) Static
( ) Flow-Through

Lighting: (x) Fluorescent ( ) Incandescent

Photoperiod: 16 hours Light
8 Hours Dark

Test Duration: 10 days

Test Area Used: Water bath

Test Temperature (°C): 20 +/- 2

Test Salinity (ppt): 28 +/- 2

Replicates Per Concentration: 5

Organisms Per Replicate: 20

Dilution Water: natural filtered seawater

TEST VESSELS

Dimensions (cm): Length x Width x Height

Test Solution Height (cm) :

Test Vessels: ~~(x) Open~~ (X) Covered

Test Container Volume: 1 L

Test Solution Volume: 725 mL

TEST SOLUTION PREPARATION

Test Concentration % mg/L									
Volume of Dilution Water Added (mL)									
Volume of Effluent/ Stock Added (mL)									

ADDITIONAL OBSERVATIONS:

DATA BY: CG DATE: 4/9/03

MACTEC

MACTEC Engineering and Consulting, Inc.
626 120th Avenue N.E.
Building B100
Bellevue, WA 98005

JOB NO. _____ SHEET _____ OF _____

PHASE _____ TASK 0300

JOB NAME Pine Harbor (whole sed)

BY [illegible] DATE _____

CHECKED BY _____ DATE _____

Spoke w/ Jim Ferretti of USEPA Region II
Explained that we had exhausted all possible avenues for producing Ampelisca for the whole sediment exposures.
He agreed that field availability was poor, and agreed to the species switch to Leptocheirus.

MACTEC
Toxicology Laboratory
Gainesville, Florida

QA Form No: 1130a
Effective Date: April 2002

Sponsor: Ponce Harbor Project # 312253 Whole Sediment, L. plumulosus

Sample ID	CTRL-PH03				RFS – PH03				union-ized / amm Total	E-PH03-SC		
Test Day	NH_3-N (ppm)	pH	Salinity (ppt)	Temp (°C)	NH_3-N (ppm)	pH	Salinity (ppt)	Temp (°C)	NH_3-N (ppm)	pH	Salinity (ppt)	Temp (°C)
Pore Water Date: 4/9/03 CG	union. .0493 to .0715 / 7.3	8.0	27	21.3	union. .286 to .443 / 7.5	8.1	32	19.9	union. 2.24 / 38.0	8.2	34	20.3
Overlying Water Day, Date	–	–	–	–	–	–	–	–	1.37 / 23.2	8.2	28	19.8 ← P-test: 4/10/03 CG
0 4/11/03	–	–	–	–	–	–	–	–	/ 15.36	8.2	28	25.0
1 4/12/03	–	–	–	–	–	–	–	–	0.296 to 0.338 / 3.96	8.3	29	21.1
2 4/13/03	–	–	–	–	–	–	–	–	0.206 / 3.25	8.2	30	20.7
3 4/14/03	–	–	–	–	–	–	–	–	0.229 / 4.76	8.3	31	20.2
4 4/15/03	–	–	–	–	–	–	–	–	0.436 / 9.53	8.1	30	20.2
5 4/16/03	–	–	–	–	–	–	–	–	0.210 / 5.99	8.0	30	19.0
6 4/17/03	–	–	–	–	–	–	–	–	0.142 / 4.26	8.0	32	18.5
7 4/18/03	–	–	–	–	–	–	–	–	0.147 / 5.03	7.9	30	19.6
8 4/19/03	–	–	–	–	–	–	–	–	0.345 / 11.8	7.9	30	19.6
9 4/20/03	–	–	–	–	–	–	–	–	0.111 / 3.82	7.9	31	19.6
10 4/21/03	–	–	–	–	–	–	–	–	0.083 / 2.92	7.9	33	19.5
Post Test	–	–	–	–	–	–	–	–	–	–	–	–

Instruments SA290C SA290B Ref 9 FS7

JM

MACTEC Engineering & Consulting, Inc.
TOXICOLOGY LAB: GAINESVILLE, FL

QA FORM NO: 108
Revised: Jan 2003

SUBJECT: SEDIMENT TOXICITY DATA SHEET

SPONSOR: Ponce Harbor/ PPB
SAMPLE ID: PH03-CTRL

PROJECT NUMBER: 312253
TEST SPECIES: L. plumulosus

DATE-DAY	REP	TEMP (°C) old	TEMP (°C) new	Hard (mg/L)	NH_3 (ppm)	pH (s.u.) old	pH (s.u.) new	DO (mg/L) old	DO (mg/L) new	Salinity (ppt) old	Salinity (ppt) new	INITIAL/ TIME Old	INITIAL/ TIME new
04/11/03 0	A		19.2				8.3		7.6		28		JA 16:15
4/12/03 1	B	21.1	18.8			8.2	8.3	7.0	7.8	29	30	HEB 0900	DD 1730
4/13/03 2	C	21.6	22.0			8.1	8.3	7.7	7.2	28	29	KG 0830	HEB 1945
4/14/03 3	D	18.8	19.3			8.2	8.4	8.0	7.8	30	30	DD 0820	DD 1615
4/15/03 4	E	21.6	20.8			8.3	8.3 7.5	7.3	7.5	28	28	CG 1000	CG 1510
4/16/03 5	A	22.2	20.3			8.3	8.3	7.3	7.5	28	28	DD 0915	HEB 1230
4/17/03 6	B	19.8	19.9			8.2 7.8	8.3	7.8	7.5	28	28	KG 0900	KG 1500
4/18/03 7	C	19.8	20.2			8.2	8.1	7.7	7.6	29	30	DD 0905	DD 1800
4/19/03 8	D	21.3	20.1			8.3	8.3	7.6	7.8	29	29	DD 0905	DD 1730
4/20/03 9	E	20.1	19.9			8.4	8.2	8.0	7.9	30	29	DD 0530	JA 1630
4/21/03 10	A	20.6				8.4		7.5		~~30~~ 30		DD 1330	

Observ Date-Day	Replicate A	B	C	D	E	Int.
04/11/03 0	20	20	20	20	20	JA 16:15
4/12/03 1	N	N	N	N	N	HEB
4/13/03 2	N	N	N	N	N	HEB
4/14/03 3	N	N	N	N	N	CG
4/15/03 4	N	N	N	N	N	CG
4/16/03 5	N	N	N	N	N	HEB
4/17/03 6	N	N	N	N	N	KG
4/18/03 7	N	N	N	N	N	DD
4/19/03 8	N	N	N	N	N	DD
4/20/03 9	N	N	N	N	N	JA
4/21/03 10	19A 1NF	17A 3NF	dropped sample	20A	20A	DD

KEY: AS = AT SURFACE EMR = EMERGENCE A = ALIVE D = DEAD NF = NOT FOUND N=NONE
REP = REPLICATE COND = CONDUCTIVITY ALK = ALKALINITY AMP = AMPLEXUS
HARD = HARDNESS TEMP = TEMPERATURE YTC = YEAST/ TROUT CHOW/CEROPHYLL

MACTEC Engineering & Consulting, Inc.
TOXICOLOGY LAB: GAINESVILLE, FL

QA FORM NO: 108
Revised: Jan 2003

SUBJECT: SEDIMENT TOXICITY DATA SHEET

SPONSOR: Ponce Harbor/ PPB
SAMPLE ID: RFS-PH03

PROJECT NUMBER: 312253
TEST SPECIES: L. plumulosus

DATE-DAY	REP	TEMP (°C) old	TEMP (°C) new	Hard (mg/L)	NH_3 (ppm)	pH (s.u.) old	pH (s.u.) new	DO (mg/L) old	DO (mg/L) new	Salinity (ppt)		INITIAL/ TIME Old	INITIAL/ TIME new
04/11/03 0	A		19.2				8.2		6.7		28		JA 16:15
4/12/03 1	B	21.1	18.8			8.2	8.3	7.0	7.8	29	30	HEB 0900	[illegible] 1730
4/13/03 2	C	21.7	21.8			8.2	8.3	7.4	7.2	30	29	KG 0830	HEB 1945
4/14/03 3	D	19.1	19.1			8.2	8.3	7.9	7.6	30	29	[illegible] 0820	[illegible] 1615
4/15/03 4	E	21.6	20.8			8.2	8.3	7.2	7.4	29	29	CG 1000	CG 1510
4/16/03 5	A	22.5	20.4			8.3	8.3	7.0	7.4	28	28	[illegible] 0930	HEB 1230
4/17/03 6	B	20.2	19.8			8.2	8.3	7.7	7.6	28	28	KG 0900	KG 1500
4/18/03 7	C	19.8	20.0			8.1	8.2	7.5	7.7	28	30	[illegible] 0905	[illegible] 1800
4/19/03 8	D	21.1	20.1			8.2	8.2	7.5	7.7	28	29	[illegible] 0905	[illegible] 1730
4/20/03 9	E	20.2	20.1			8.4	8.2	8.0	7.8	30	29	[illegible] 0530	JA 1700
4/21/03 10	A	20.7				8.3		7.5		28		[illegible] 1330	

Observ Date-Day	Replicate A	B	C	D	E	Int.
04/11/03 0	20	20	20	20	20	JA 16:15
4/12/03 1	N	N	N	N	N	HEB
4/13/03 2	N	N	N	N	N	HEB
4/14/03 3	N	N	N	N	N	CG
4/15/03 4	N	N	N	N	N	CG
4/16/03 5	N	N	N	N	N	HEB
4/17/03 6	N	N	N	N	N	KG
4/18/03 7	N	N	N	N	N	[illegible]
4/19/03 8	N	N	N	N	N	[illegible]
4/20/03 9	N	N	N	N	N	JA
4/21/03	19 2NF	19 1NF	20	19 1NF	19 1NF	JA

KEY: AS = AT SURFACE EMR = EMERGENCE A = ALIVE D = DEAD NF = NOT FOUND N=NONE
REP = REPLICATE COND = CONDUCTIVITY ALK = ALKALINITY AMP = AMPLEXUS
HARD = HARDNESS TEMP = TEMPERATURE YTC = YEAST/ TROUT CHOW/CEROPHYLL

MACTEC Engineering & Consulting, Inc.
TOXICOLOGY LAB: GAINESVILLE, FL

QA FORM NO: 108
Revised: Jan 2003

SUBJECT: SEDIMENT TOXICITY DATA SHEET

SPONSOR: Ponce Harbor/ PPB
SAMPLE ID: E-PH03-SC

PROJECT NUMBER: 312253
TEST SPECIES: L. plumulosus

DATE-DAY	REP	TEMP (°C) old	TEMP (°C) new	Hard (mg/L)	NH_3 (ppm)	pH (s.u.) old	pH (s.u.) new	DO (mg/L) old	DO (mg/L) new	Salinity (ppt)		INITIAL/ TIME Old	INITIAL/ TIME new
04/11/03 0	A		19.3				8.2		7.6		28		JA 16:17
4/12/03 1	B	21.2	19.3 ~~19.3~~			8.3	8.3	7.2	7.7	29	30	HEB 0900	~~DD~~ 1730
4/13/03 2	C	21.6	21.8			8.2	8.4	7.1	7.3	30	29	KG 0530	HEB 1945
4/14/03 3	D	19.5	19.1			8.2	8.4	7.0	7.7	30	29	~~DD~~ 0820	~~DD~~ 1615
4/15/03 4	E	21.7	20.8			8.3	8.3	7.3	7.5	28	28	CG 1000	CG 1510
4/16/03 5	A	22.6	20.5			8.2	8.3	7.2	7.3	28	28	~~DD~~ 0930	HEB 1230
4/17/03 6	B	20.3	19.8			8.2	8.3	7.8	7.6	28	28	KG 0900	KG 1500
4/18/03 7	C	20.0	20.1			8.1	8.2	7.6	7.6	28	30	~~DD~~ 0905	~~DD~~ 1800
4/19/03 8	D	21.3	20.1			8.1	~~8.3~~ 8.1	7.6	7.7	28	29	~~DD~~ 0905	~~DD~~ 1730
4/20/03 9	E	20.4	20.1			8.1	8.1	7.3	7.8	30	29	~~DD~~ 0530	JA 1700
4/21/03 10	A	20.9				8.3		7.5		30		~~DD~~ 1330	

Observ Date-Day	Replicate A	B	C	D	E	Int.
04/11/03 0	20	20	20	20	20	JA 16:15
4/12/03 1	N	N	N	N	N	HEB
4/13/03 2	N	N	N	N	N	HEB
4/14/03 3	N	N	N	N	N	CG
4/15/03 4	N	N	N	N	N	CG
4/16/03 5	N	N	N	N	N	HEB
4/17/03 6	N	N	N	N	N	KG
4/18/03 7	N	N	N	N	N	~~DD~~
4/19/03 8	N	N	N	N	N	~~DD~~
4/20/03 9	N	N	N	N	N	JA
4/21/03	16 4NF	16 4NF	17 3NF	17 3NF	18 2NF	AM.

KEY: AS = AT SURFACE EMR = EMERGENCE A = ALIVE D = DEAD NF = NOT FOUND N=NONE
REP = REPLICATE COND = CONDUCTIVITY ALK = ALKALINITY AMP = AMPLEXUS
HARD = HARDNESS TEMP = TEMPERATURE YTC = YEAST/ TROUT CHOW/CEROPHYLL

MACTEC Engineering and Consulting
TOXICOLOGY LABORATORY
GAINESVILLE, FLORIDA

PAGE:
FORM NO: 097A REFTOX
REVISED: MARCH 2001

REFERENCE TOXICANT TEST DATA SHEET

Sponsor: Misc.	Project Number: Ref Tox
Test Material	**Test Conditions**
Amount of CdCL2 : 0.0328 (g) Volume of Milli-Q Water: 1000 (mL) Date Prepared: 4/11/03	[x] Definitive [x] Static [] Screening Duration: 96 Hours
Test Organism History	Dilution Water: natural filtered seawater
Species: L. plumulosus Batch Number: 2003-25 Age/Life Stage: Adult Date of Acclimation/Maintenance: 4/9/03 See Page 107 of Organism Receipt Log Mortality 48 hours prior to test: <1 (%) Replicates/Concentration: 1 No. Of Organisms/Replicate: 10	Lighting: [x Fluorescent [ ] Incandescent Photoperiod: 16 Hr Light 8 Hr Dark Test Vessel Dimensions: 100 L X - W 50 H Test Solution Height: 4 (cm) Test Vessel Volume: .34 (L) Test Solution Volume: .20 (L) Test Temperature: 20 ± 1 °C Test Salinity: 28 ± 2 ppt
Test Area Used: Waterbath table top.	Test Vessel Composition: [x] Glass [ ] Plastic
Test Concentrations Based on: [x] Whole Material [ ] Active Ingredient	Test Protocol/Guideline Followed:

Test Concentrations: (Units: mg/L)	Control	0.256	0.512	1.02	2.05	4.1	
Amount Dilution Water Added (mL)	250	246.8	243.6	237.2	224.4	199.8	
Amount of Stock Added (mL)	0	3.2	6.4	12.8	25.6	51.2	

Additional Observations:

Data By: [signature] Date: 04/11/03

[signature]

REFERENCE TOXICANT TEST DATA: DAY 0 – 2

Client: Misc.	Project Number: Ref Tox	Species: *L.plumulosus*

DAY 0 — REF TOXICANT: CdCl2

Nominal Concentration (mg/L)	Date: 4/11/03		Time: 1415		Analyst: DJ	
	# Alive	Observ.	Temp. (°C) A	Sal (ppt) A	DO (mg/L) A	pH A
Control	20 loaded	N	19.0	~~28~~ ~~1415~~	7.8	8.3
0.256			18.7	28 ~~2025~~	7.8	8.3
0.512			19.1	28	7.8	8.3
1.02			19.0	28	7.8	8.3
2.05			18.9	28	7.8	8.3
4.10			19.0	28	7.6	8.3
				~~28~~		

DAY 1	Date: 4/12/03		Time: 1600		Analyst: DJ	
Nominal Concentration	A # Alive B	Observ.	B Temp. (°C)	B Sal (ppt)	B DO (mg/L)	B pH
Control	10 / 10	N / N	19.6	28	7.7	8.3
0.256	10 / 10	N / N	19.5	28	7.7	8.3
0.512	10 / 10	N / N	19.7	28	7.7	8.3
1.02	10 / 10	N / N	19.6	28	7.7	8.2
2.05	10 / 10	N / N	19.5	28	7.6	8.2
4.10	10 / 10	N / N	19.6	25	7.6	8.2
	~~16~~					

DAY 2	Date: 4/13/03		Time: 1900		Analyst: treB	
Nominal Concentration	A # Alive B	Observ.	A Temp. (°C)	A Sal (ppt)	A DO (mg/L)	A pH
Control	10 10	N N	20.3	29	6.0	8.3
0.256	10 10	N N	20.2	28	5.7	8.3
0.512	10 10	N N	20.2	28	5.8	8.4
1.02	9 10	1d N	20.2	28	5.7	8.4
2.05	8 8	2d 2d	20.2	27	5.7	8.4
4.10	3A 4A	7d 6d	20.3	26	5.7	8.3

MACTEC Engineering and Consulting, Inc.
TOXICOLOGY LABORATORY
GAINESVILLE, FLORIDA

QA FORM NO: 097RT REFTOX
Revised: March 2001

REFERENCE TOXICANT TEST DATA: DAY 3 AND 4

Client: Misc.	Project Number: Ref Tox, CdCl2	Species: *L. plumulosus*

DAY 3

Nominal Concentration	Date: 04/14/03				Time: 1330		Analyst: [illegible]	
	A # Alive	B	A Observ.	B	B Temp. (°C)	B Sal (ppt)	B DO (mg/L)	B pH
Control	10	10	N	N	19.2	28	7.8	8.4
0.256	10	10	N	N	19.2	29	7.8	8.4
0.512	10	10	N	N	19.3	28	7.8	8.4
1.02	9	9	N	1d	19.2	28	7.7	8.4
2.05	7	8	1d	N	19.1	26	7.7	8.4
4.10	2	0	1d	4d	19.4	26	7.8	8.4

DAY 4	Date: 04/15/03				Time: 0930 *		Analyst: CG	
Nominal Concentration	A # Alive	B	A Observ.	B	A ~~Temp. (°C)~~ DO CG 4/15/03	A Sal (ppt)	A ~~DO (mg/L)~~ Temp. (°C) CG 4/15/03	A pH
Control	9	10	1d	N	7.6	30	19.0	8.3
0.256	10	10	N	N	7.5	29	19.0	8.3
0.512	8	10	2d	N	7.5	29	19.1	8.3
1.02	7	8	2d	1d	7.5	28	19.0	8.3
2.05	6	8	1d	N	7.7	26	19.0	8.3
4.10	0	0	2d ~~[illegible]~~ CG 4/15/03	N	7.7	26	19.2	8.3

Water quality monitored in AM, CG
test terminated at 1400.

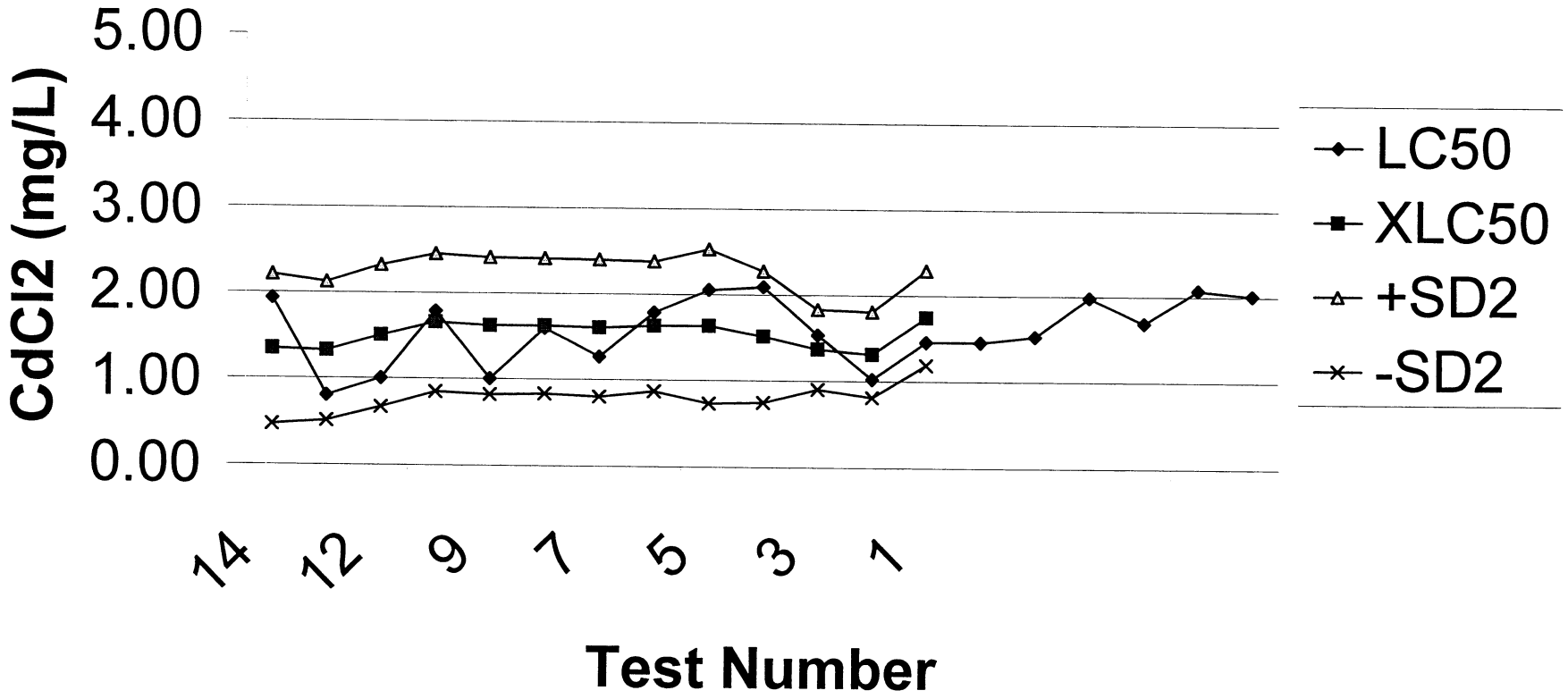
[illegible]

SUMMARY – Ref Tox, *L. plumulosus* (CdCl2)

COMMENTS:

Nominal Concentration	Number Dead
Control	1
0.256	0
0.512	2
1.02	5
2.05	6
4.10	20

L. plumulosus
Reference Toxicant Test:
Cadmium Chloride



TRIMMED SPEARMAN-KARBER METHOD. VERSION 1.5

DATE: 04/13/03 TEST NUMBER: PonceWhS DURATION: 96 h
TOXICANT : CdCl2
SPECIES: L. plumulosus

RAW DATA: --- ----	Concentration (mg/L)	Number Exposed	Mortalities
	.00	20	1
	.26	20	0
	.51	20	2
	1.02	20	5
	2.05	20	6
	4.10	20	20

SPEARMAN-KARBER TRIM: .00%

SPEARMAN-KARBER ESTIMATES: LC50: 1.93
95% LOWER CONFIDENCE: 1.56
95% UPPER CONFIDENCE: 2.37

NOTE: MORTALITY PROPORTIONS WERE NOT MONOTONICALLY INCREASING.
ADJUSTMENTS WERE MADE PRIOR TO SPEARMAN-KARBER ESTIMATION.

Aquatic Research Organisms

Rec'd 4/9/03 CG

DATA SHEET

I. **Organism History**

Species: Leptocheirus plumulosus

Source: Lab reared X Hatchery reared _____ Field collected _____

Hatch date 03/03 Receipt date _____

Lot number 040803LP Strain _____

Brood Origination Chesapeake Bay

II. **Water Quality**

Temperature 20 °C Salinity 20 ppt DO SAT

pH 8.1 Hardness NA ppm

III. **Culture Conditions**

System: SW static renewal

Diet: Flake Food _____ Phytoplankton _____ Trout Chow _____

Brine Shrimp _____ Rotifers _____ Other GORP

Prophylactic Treatments: _____

Comments: 3-5 mm, sediment cube enclosed

IV. **Shipping Information**

Client: ESE MAXTEC # of Organisms: 500+

Carrier: FED EX Date Shipped: 4/8/03

Biologist: Stan Leitzke

ARO inv # 120250
PO # 5604

1 - 800 - 927 - 1650

PO Box 1271 • One Lafayette Road • Hampton, NH 03842 • (603) 926-1650

Environmental Science & Engineering, Inc.
Aquatic Toxicology Laboratory
Gainesville, Florida

ESE QA Form No.: 008
Effective: Mar 1993

SUBJECT: ORGANISM RECEIPT/COLLECTION LOG

Date Recieved	Species	Population Size	Population Condition	Collector/ Supplier	Salinity/ Hardness	Temperature (C)	Initials
3/18/03	M. nasuta	2003-16 ~500	normal	ARO	—	8.5	~~JMM~~ adult
3/21/03	Americamysis bahia	320+ 2003-17	normal	ARO	28	23.1	HEB 2-days
3/21/03	M. beryllina	320+ 2003-18	normal	ARO	27	23.1	HEB 10-days
3/23/03	M. bahia	375 2003-19	normal	Aquatic Indic.	20 ppm	25.0	JA ~~2 days~~
3/23/03	M. beryllina	85 2003-20	normal	Aquatic Indic.	20 ppm	25.0	JA 10 day
3/26/03	M. edulis	~48 2003-21	normal	ARO	—	11.3	~~JMM~~ adult
3/28/03	M. bahia	450+ 2003-22	normal	ARO	28 ppt	22.8	HEB 3days
3/31/03	Brine Shrimp Cysts	15 oz can	normal	Aquatic Eco-Systems	—	—	CG
4/2/03	M. nasuta	2003-23 ~~1000~~	normal	ARO	—	12.7	CG adult
4/3/03	N. Virens	2003-24 500	normal	ARO	—	10.5	CG adult
4/4/03	L. plumulosus	2003-25 ~~±200~~ +500	normal	ARO	23 ppt	17.4	CG

Harding ESE
Toxicology Laboratory
Gainesville, Florida

Page: 65
QA Form No. 009
Revised: March 2001

SUBJECT: DAILY RECORD OF INVERTEBRATE HOLDING CONDITIONS

Species: Leptocheirus plumulosus
Lot Number: 2003-25
Date collected/received: 4/9/03
Date of hatch: 03/03
Collection location/supplier: ARO
Page 107 of animal receipt and collection log.
Data continued from Page _____ of this log book.

Date	4/9/03	4/10/03	4/11/03		
Time	1145	1545	1310		
Technician	CG	[illegible]	[illegible]		
Number alive	>99%	>99%	>99%		
Number dead	<1%	<1%	<1%		
Hardness/Salinity	23	24	25		
Method/Meter	Ref 9	Ref 9	Ref 9		
Temperature (EC)	17.4	19.0	18.5		
Meter number	FS7	FS7	FS7		
D.O. (mg/L)	>10	>10	5.8		
Meter number	D03	D03	D03		
pH (s.u.)	7.0	7.0	7.4		
Meter number	SA290B	SA290B	SA290B		
Water	clear	muddy	muddy		
Food	—	yes, in mud	Yes, in mud		
Clean	—	—	—		
Number removed	0	0	0		
Project No. / Task	312253	312253	312253		

_____ Data continued on Page _____ of this log book.
_____ Population was terminated. Date_____ How_____

Comments: _____

CR

Appendix D

MACTEC

MACTEC Engineering and Consulting, Inc.
626 120th Avenue N.E.
Building B100
Bellevue, WA 98005

JOB NO. _____ SHEET 1 OF 1
PHASE _____ TASK 0400
JOB NAME Ponce Harbor (Brookum)
BY [illegible] DATE _____
CHECKED BY _____ DATE _____

Spoke w/ Jim Fonetti of USEPA Region II concerning failure of both species (controls) for Ponce Harbor over the weekend.

He agreed to the need for a retest. I explained that we did not know (for sure) the precise cause but had that water tanks flow into a little used tank which then gravity fed to the test vessels. I have concluded that there may have been something in the ~~test~~ bottom of the tank which caused the failure.

Plumbing issues to be corrected and test restarted as soon as organisms available.

MACTEC Engineering & Consulting, Inc.
Toxicology Laboratory
Gainesville, Florida

Page: _____
QA Form No. 018
Revised: October 2002

Project No: 312253.0400 Client: Ponce Test: Bioaccumulation

DAILY LOG

4/10/03 - Organisms loaded. water qualities taken. Flow rate taken. airlines checked.

4/11/03 - WQ checked, flow rate measured, airlines checked organisms observed

4/12/03 - Water qualities monitored, flow rates measured, airlines checked and organisms observed.

4/13/03 - Water qualities monitered, flow rates measured, airlines checked and organisms observed.

4/14/03 - water qualities monitered, flow rates measured airlines checked + Organisms observed

4/15/03 - airlines checked and organisms observed. water qualities monitored. Flow rates checked.

4/16/03 - airlines checked + Organisms observed water qualites monitored. Flow rates checked

4/17/03 - water qualities taken. organisms observed. Flow rates monitored and airlines checked.

4/18/03 water qualities taken. Organisms observed. Flow rates monitored + airlines checked.

4/19/03 water qualities taken Organisms observed Flow rates monitored + airlines checked

4/20/03 water qualities taken Organism Observed Flow rates monitored + airlines checked

4/21/03 water qualities taken, organisms observed. Flow rates monitored + airlines checked.

MACTEC Engineering & Consulting, Inc.
Toxicology Laboratory
Gainesville, Florida

Page: _____
QA Form No. 018
Revised: October 2002

Project No: 312253.0400 Client: Ponce Test: Bioaccumulation

DAILY LOG

4/22/03 - Water qualites taken, Organisms observed, flow rates checked. Airlines checked

4/23/03 Water qualities taken, organisms observed Flow rates checked, airlines checked

4/24/03 Water qualities taken, organisms observed, flow rates measured and airlines checked (data reviewed, JMR. Will adjust water chiller/heater.)

4/25/03 - water qualites taken, organisms observed, flow rates taken and airlines checked.

4/26/03 - water qualities taken, flow rates taken airlines checked and organisms observed

4/27/03 water quality monitored. flow rates taken airlines checked and organisms observed

4/28/03 Water quality monitored, Flow rates taken, Airlines checked + organisms observed

4/29/03 Water quality monitored, Flow rates taken, airlines checked and organisms observed.

4/30/03 Water qualities taken, airlines checked, and organisms observed. Flow rates monitored.

5/1/03 water qualities taken, organisms observed, flow rates taken airlines checked

5/2/03 Water qualities monitored, airlines checked, Flow rates taken + organisms observed

MACTEC Engineering & Consulting, Inc.
Toxicology Laboratory
Gainesville, Florida

Page: _____
QA Form No. 018
Revised: October 2002

Project No: 312253.0400 Client: Ponce Test: Bioaccumulation

DAILY LOG

5/3/03 Water qualities monitored, air lines checked, flow rates taken + organisms observed

5/4/03 water qualities monitored, air lines checked, organisms observed + flow rates taken

5/5/03 Water qualities monitored, air lines checked, organisms observed + flow rates taken

5/6/03 water qualities taken. airline checked organisms observed + flow rate taken

5/7/03 water qualities monitored, airline checked organisms observed + flow rates taken

5/8/03 Water quality monitored. Organisms counted, + bagged in saltwater to depurate overnight. test terminated.

MACTEC Engineering and Consulting, Inc.
Gainesville, Florida

QA Form No.: 097
Revised: March 2001

SUBJECT: TOXICITY TEST SYSTEM DESCRIPTION SHEET

Sponsor: Ponce Harbor/ PPB

Project No.: 312253.0400

TEST MATERIAL

Test Material: Sediment

See Page 654 of Effluent Log for Test Material Information.

PROTOCOL/ GUIDELINES FOLLOWED:

Test Concentrations Based on: ( ) Active Ingredient
(x) Whole Material

TEST ORGANISM HISTORY

Test Species: nereis virens

Lot Number: 2003-26

Age/ Life Stage: adult

Date Acclimation/ Maintenance Began: 4/9/03

See Page 107 of Org. Receipt Log for Data.

Mortality (%) During 48 Hours Prior to Test Initiation: <1%

TEST CONDITIONS

( ) Range Finder
(X) Screening
( ) Definitive

( ) Static
(X) Flow-Through

Lighting: (x) Fluorescent ( ) Incandescent

Photoperiod: Hours Light
Hours Dark

Test Duration: 28 days

Test Area Used: Backroom

Test Temperature (°C): 20 +/- 2

Test Salinity (ppt): 30 +/- 2

Replicates Per Concentration: 5

Organisms Per Replicate: 20

Dilution Water: natural filtered seawater

TEST VESSELS

Dimensions (cm): Length x Width x Height

Test Solution Height (cm) :

Test Vessels: (x) Open ( ) Covered

Test Container Volume: 38 L (10 gallons)

Test Solution Volume: 30 L

TEST SOLUTION PREPARATION

Test Concentration % mg/L									
Volume of Dilution Water Added (mL)									
Volume of Effluent/ Stock Added (mL)									

ADDITIONAL OBSERVATIONS:

5 cm minimum sediment per tank

DATA BY: CG DATE: 4/10/03

MACTEC Engineering and Consulting (formerly Harding ESE)

Revised: Oct 2002

SUBJECT: SEDIMENT TOXICITY DATA SHEET - WATER QUALITY

QA FORM NO: 108

SPONSOR: Ponce Harbor/ PPB
SAMPLE ID: PH03-CTRL

PROJECT NUMBER:
TEST SPECIES: n. virens

DATE-DAY	REP	Temp (°C)	NH_3 (ppm)	pH (s.u.)	DO (mg/L)	Salinity (ppt)	INITIAL
4/10/03 0	A	18.7		8.3	7.8	30	KG
4/11/03 1	B	18.1		8.3	7.8	30	HEB
4/12/03 2	C	18.0		7.9	8.1	30	HEB
4/13/03 3	D	18.4		8.1	8.4	32	KG
4/14/03 4	E	17.9		8.2	7.9	32	DD
4/15/03 5	A	18.0		8.2	8.0	32	CG
4/16/03 6	B	17.8 ~~20.2~~		8.2	8.1	31	DD
4/17/03 7	C	20.3		8.2	7.6	32	KG
4/18/03 8	D	18.8		8.2	7.4	32	DD
4/19/03 9	E	19.2		8.1	7.8	31	DD
4/20/03 10	A	19.6		8.0	7.4	32	DD
4/21/03 11	B	19.2		8.2	7.5	32	CG
4/22/03 12	C	19.3		8.1	7.6	32	CG
4/23/03 13	D	19.5		8.0	7.5	32	[illegible]
4/24/03 14	E	17.0		8.0	7.8	31	RD
4/25/03 15	A	17.8		8.0	7.6	32	CG
4/26/03 16	B	19.0		8.0	7.7	31	KG
4/27/03 17	C	19.1		8.0	7.5	32	HEB
4/28/03 18	D	18.4		7.8	8.3	32	RD
4/29/03 19	E	19.3		8.0	8.0	29	CG
4/30/03 20	A	19.4		8.0	8.1	31	CG
5/1/03 21	B	18.2		8.0	7.1	31	KG
5/2/03 22	C	19.5		7.8	7.6	32	RD
5/3/03 23	D	18.9		7.8	7.2	31	RD
5/4/03 24	E	19.3		8.0	7.3	31	KG
5/5/03 25	A	18.4		8.0	8.0	32	RD
5/6/03 26	B	19.3		8.1	7.5	30	[illegible]
5/7/03 27	C	18.0		7.4	8.0	31	DD
5/8/03 28	D	17.8		7.8	8.0	30	DD

MACTEC Engineering and Consulting (formerly Harding ESE) Revised: Oct 2002

SUBJECT: SEDIMENT TOXICITY DATA SHEET - WATER QUALITY QA FORM NO: 108

SPONSOR: Ponce Harbor/ PPB
SAMPLE ID: RFS-PH03

PROJECT NUMBER: 312253.0400
TEST SPECIES: n. virens

DATE-DAY	REP	Temp (°C)	NH_3 (ppm)	pH (s.u.)	DO (mg/L)	Salinity (ppt)	INITIAL
4/10/03 0	A	19.0		8.3	7.7	30	KG
4/11/03 1	B	18.4		8.3	7.5	30	HEB
4/12/03 2	C	18.0		8.0	8.5	30	HEB
4/13/03 3	D	17.9		8.1	7.9	32	KG
4/14/03 4	E	17.6		8.2	7.8	32	DD
4/15/03 5	A	17.7		8.3	8.0	32	CG
4/16/03 6	B	17.3		8.2	8.2	31	DD
4/17/03 7	C	20.4		8.2	7.7	32	KG
4/18/03 8	D	18.3		8.2	7.4	32	DD
4/19/03 9	E	19.0		7.9	7.6	32	DD
4/20/03 10	A	19.8		8.0	7.4	32	DD
4/21/03 11	B	20.3		8.1	7.2	32	CG
4/22/03 12	C	19.8		8.2	8.3	32	CG
4/23/03 13	D	20.0		8.0	7.4	32	RD
4/24/03 14	E	17.6		7.9	7.3	32	RD
4/25/03 15	A	18.8		8.0	7.3	32	CG
4/26/03 16	B	19.4		8.0	7.5	31	KG
4/27/03 17	C	18.8		7.8	7.4	32	HEB
4/28/03 18	D	18.2		7.9	7.6	30	RD
4/29/03 19	E	18.6		8.0	8.1	30	CG
4/30/03 20	A	19.0		8.0	8.1	30	CG
5/1/03 21	B	19.0		8.0	6.9	30	KG
5/2/03 22	C	19.3		7.9	7.4	32	RD
5/3/03 23	D	19.2		7.9	7.8	31	RD
5/4/03 24	E	19.0		7.8	7.0	31	KG
5/5/03 25	A	18.7		8.0	7.5	31	RD
5/6/03 26	B	19.1		7.9	8.0	30	JA
5/7/03 27	C	17.9		7.9	8.1	30	DD
5/8/03 28	D	18.0		7.9	7.6	30	KG

MACTEC Engineering and Consulting (formerly Harding ESE) Revised: Oct 2002

SUBJECT: SEDIMENT TOXICITY DATA SHEET - WATER QUALITY QA FORM NO: 108

SPONSOR: Ponce Harbor/ PPB
SAMPLE ID: E-PH03-SC

PROJECT NUMBER: 312253.0400
TEST SPECIES: n. virens

DATE-DAY	REP	Temp (°C)	NH_3 (ppm)	pH (s.u.)	DO (mg/L)	Salinity (ppt)	INITIAL
4/10/03 0	A	19.2		8.2	7.7	30	KG
4/11/03 1	B	18.2		8.2	7.4	30	HEB
4/12/03 2	C	18.0		8.0	8.4	30	HEB
4/13/03 3	D	17.9		8.1	8.0	32	KG
4/14/03 4	E	17.7		8.2	7.7	32	DD
4/15/03 5	F	17.8		8.3	6.9	32	CG
4/16/03 6	G	17.6		8.1	7.9	31	DD
4/17/03 7	H	20.5		8.2	7.5	31	KG
4/18/03 8	I	18.5		8.2	7.4	32	DD
4/19/03	A	19.3		7.9	7.2	31	DD
4/20/03 10	B	19.9		7.6	5.0	32	DD
4/21/03 11	C	19.8		8.1	7.3	[illegible]	CG
4/22/03 12	D	19.9		8.1	7.7	32	CG
4/23/03 13	E	19.6		8.0	7.4	32	RD
4/24/03 14	F	17.2		8.0	7.4	31	RD
4/25/03 15	G	18.1		7.9	7.3	32	CG
4/26/03 16	H	19.3		7.9	7.6	31	KG
4/27/03 17	I	18.6		7.9	7.4	32	HEB
4/28/03 18	A	12.7		7.9	7.3	31	RD
4/29/03 19	B	18.3		7.9	8.1	30	CG
4/30/03 20	C	19.0		7.9	7.9	31	CG
5/1/03 21	D	18.8		8.0	7.6	31	KG
5/2/03 22	E	19.8		7.9	7.3	32	RD
5/3/03 23	F	19.3		8.0	7.7	31	RD
5/4/03 24	G	19.6		7.8	7.0	31	KC
5/5/03 25	H	18.8		7.9	7.8	31	RD
5/6/03 26	I	19.0		7.9	8.0	30	JA
5/7/03 27	A	18.2		7.8	7.8	30	DD
5/8/03 28	B	18.1		7.8	7.9	30	KG

mL/min

Harding ESE

Revised: March 2001

SUBJECT: SEDIMENT TOXICITY DATA SHEET - WATER FLOW

QA FORM NO: 108E

SPONSOR: PONCE
SAMPLE ID: E PH03 SC

PROJECT NUMBER: 312253.0400
TEST SPECIES: ~~M. nasuta~~ N. virens

DATE-DAY	REP A	REP B	REP C	REPD	REP E	INITIAL
4/10/03 0	100	100	95	80	98	KG
4/11/03 1	100	96	100	86	95	HEB
4/12/03 2	96	85	80	75	90	HEB
4/13/03 3	102	98	75	80	82	KG
4/14/03 4	78	82	76	90	105	[illegible]
4/15/03 5	100	84	78	77	82	CG
4/16/03 6	120	80	100	90	80	[illegible]
4/17/03 7	115	110	100	80	100	KG
4/18/03 8	100	80	100	80	120	[illegible]
4/19/03 9	80	90	100	140	76	[illegible]
4/20/03 10	78	78	86	90	100	[illegible]
4/21/03 11	83	77	76	95	88	CG
4/22/03 12	94	100	83	91	83	CG
4/23/03 13	120	96	100	80	80	RD
4/24/03 14	112	80	80	82	84	RD
4/25/03 15	78	81	93	98	108	CG
4/26/03 16	85	72	82	100	75	KG
4/27/03 17	84	76	78	80	90	HEB
4/28/03 18	76	100	76	96	76	RD
4/29/03 19	84	91	95	81	79	CG
4/30/03 20	76	79	101	87	89	CG
5/1/03 21	92	80	75	92	100	KG
5/2/03 22	78	88	84	100	96	RD
5/3/03 23	88	76	100	104	88	RD
5/4/03 24	102	95	98	92	100	KG
5/5/03 25	92	100	78	82	80	RD
5/6/03 26	104	96	120	84	90	JA
5/7/03 27	101	83 flow	99	92 terminated	87 off	KG
5/8/03 28						

mL/min

Harding ESE

Revised: March 2001

SUBJECT: SEDIMENT TOXICITY DATA SHEET - WATER FLOW

QA FORM NO: 108E

SPONSOR: PONCE
SAMPLE ID: CTRL-PH03

PROJECT NUMBER: 312253.0400
TEST SPECIES: ~~M. nasuta~~ N. virens

DATE-DAY	REP A	REP B	REP C	REPD	REP E	INITIAL
4/10/03 0	80	115	92	78	100	KG
4/11/03 1	76	75	85	77	96	HEB
4/12/03 2	75	84	72	90	86	HEB
4/13/03 3	80	100	75	92	78	KG
4/14/03 4	76	80	78	77	85	DS
4/15/03 5	82	81	75	85	90	CG
4/16/03 6	100	120	78	84	88	DS
4/17/03 7	105	105	85	90	110	KG
4/18/03 8	76	80	76	80	80	DS
4/19/03 9	76	80	80	84	100	DS
4/20/03 10	76	84	88	86	78	DS
4/21/03 11	79	98	82	91	65	CG
4/22/03 12	78	75	81	79	92	CG
4/23/03 13	84	76	116	86	76	RD
4/24/03 14	84	80	76	100	94	RD
4/25/03 15	78	77	90	110	101	CG
4/26/03 16	80	75	72	80	84	KG
4/27/03 17	76	80	74	84	90	HEB
4/28/03 18	76	84	78	80	88	RD
4/29/03 19	78	82	83	91	83	CG
4/30/03 20	81	78	77	86	85	CG
5/1/03 21	80	90	85	84	92	KG
5/2/03 22	78	100	76	84	88	RD
5/3/03 23	80	76	100	76	92	RD
5/4/03 24	75	82	90	102	78	KG
5/5/03 25	96	80	84	100	98	RD
5/6/03 26	120	100	90	84	120	JM
~~5/7/03 27~~	~~92~~	~~91~~	~~83~~	~~79~~	~~100~~	~~CG~~ CG
28		Flow turned off/terminated test				

mL/min

Harding ESE

Revised: March 2001

SUBJECT: SEDIMENT TOXICITY DATA SHEET - WATER FLOW

QA FORM NO: 108E

SPONSOR: PONCE
SAMPLE ID: RFS-PH03

PROJECT NUMBER: 312253.0400
TEST SPECIES: ~~M. nasuta~~ N. virens

DATE-DAY	REP A	REP B	REP C	REPD	REP E	INITIAL
4/10/03 0	80	75	100	92	115	KG
4/11/03 1	81	92	100	82	90	HEB
4/12/03 2	80	94	96	82	80	HEB
4/13/03 3	78	92	100	70	80	KG
4/14/03 4	79	80	90	76	80	DD
4/15/03 5	77	79	80	75	91	CG
4/16/03 6	110	80	84	76	120	DD
4/17/03 7	110	110	100	75	110	KG
4/18/03 8	80	100	120	90	120	DD
4/19/03 9	80	80	130	76	80	DD
4/20/03 10	82	130	90	78	84	DD
4/21/03 11	75	101	82	79	78	CG
4/22/03 12	89	95	92	75	89	CG
4/23/03 13	88	96	84	86	140	RP
4/24/03 14	80	76	76	78	80	RD
4/25/03 15	98	112	109	97	86	CG
4/26/03 16	72	70	85	86	90	KG
4/27/03 17	100	76	76	80	87	HEB
4/28/03 18	78	80	76	100	88	RD
4/29/03 19	77	77	89	86	107	CG
4/30/03 20	85	75	81	79	77	CG
5/1/03 21	100	82	92	95	102	KG
5/2/03 22	84	108	78	86	76	RD
5/3/03 23	80	88	76	96	100	RD
5/4/03 24	72	80	95	80	78	KG
5/5/03 25	78	102	80	88	80	RD
5/6/03 26	120	100	80	78	96	JH
5/7/03 27	100	80	92 ~~Flow~~	80 ~~turned~~	89 ~~off~~	KG
5/8/03 28		termination				

mL/min

Harding ESE

Revised: March 2001

SUBJECT: SEDIMENT TOXICITY DATA SHEET - WATER FLOW

QA FORM NO: 108E

SPONSOR: PONCE
SAMPLE ID: E PH03 SC

PROJECT NUMBER: 312253.0400
TEST SPECIES: ~~M nasuta~~ N. virens

DATE-DAY	REP F	REP G	REP H	REPI		INITIAL
4/10/03 0	80	90	85	84		KG
4/11/03 1	75	75	82	83		HEB
4/12/03 2	78	82	82	75		HEB
4/13/03 3	80	78	82	100		KG
4/14/03 4	120	78	80	86		[illegible]
4/15/03 5	94	87	91	102		CG
4/16/03 6	100	110	108	106		[illegible]
4/17/03 7	95	100	115	110		KG
4/18/03 8	100	78	76	100		[illegible]
4/19/03 9	76	100	96	120		[illegible]
4/20/03 10	80	79	80	110		[illegible]
4/21/03 11	78	77	85	89		CG
4/22/03 12	82	87	81	77		CG
4/23/03 13	76	80	82	76		RD
4/24/03 14	76	78	84	100		RD
4/25/03 15	82	76	83	79		CG
4/26/03 16	75	100	82	95		KG
4/27/03 17	76	96	80	79		HEB
4/28/03 18	76	92	78	80		RD
4/29/03 19	82	87	82	78		CG
4/30/03 20	85	79	86	83		CG
5/1/03 21	102	100	72	82		KG
5/2/03 22	92	80	76	84		RD
5/3/03 23	100	80	84	120		RD
5/4/03 24	98	88	92	75		KG
5/5/03 25	96	100	90	76		RD
5/6/03 26	94	120	86	~~96~~ 96		JH
5/7/03 27	89	101	86	95		KG
5/8/03 28		termination				

~~Flow turned off~~

MACTEC Engineering and Consulting (formerly Harding ESE) Revised: March 2001

SUBJECT: SEDIMENT TOXICITY DATA SHEET - SURVIVAL QA FORM NO: 108E

SPONSOR: Ponce Harbor/ PPB
SAMPLE ID: PH03-CTRL

PROJECT NUMBER: 312253.~~001~~0400
TEST SPECIES: N. virens

DATE-DAY	REP A	REP B	REP C	REPD	REP E	INITIAL
4/10/03 0	20 loaded	→				CG
4/11/03 1	N	N	N	N	N	HEB
4/12/03 2	N	1 d	N	N	N	HEB
4/13/03 3	N	N	1 d	N	N	KG
4/14/03 4	N	N	N	N	N	~~SD~~
4/15/03 5	N	N	N	1 d	N	CG
4/16/03 6	N	N	N	N	N	~~DD~~
4/17/03 7	N	N	N	N	N	KG
4/18/03 8	N	N	N	N	N	~~DD~~
4/19/03 9	N	N	N	N	N	~~DD~~
4/20/03 10	N	N	N	N	N	~~DD~~
4/21/03 11	N	N	N	N	N	CG
4/22/03 12	N	N	N	N	N	CG
4/23/03 13	N	N	N	N	N	JM
4/24/03 14	N	N	N	N	N	HEB
4/25/03 15	N	N	N	N	N	CG
4/26/03 16	N	N	N	N	N	HEB
4/27/03 17	N	N	N	1 d	N	HEB
4/28/03 18	N	N	N	N	N	JM
4/29/03 19	N	N	N	N	N	CG
4/30/03 20	N	N	N	N	N	CG
5/1/03 21	N	N	N	N	N	KG
5/2/03 22	N	N	N	N	N	RD
5/3/03 23	N	N	N	N	N	RD
5/4/03 24	N	N	N	N	N	KG
5/5/03 25	N	N	N	N	N	RD
5/6/03 26	N	N	N	N	N	JA
5/7/03 27	N	N	N	N	N	~~DD~~
5/8/03 28	20 A	19 A	18 A	18 A	20 A	JM

1 NF

MACTEC Engineering and Consulting (formerly Harding ESE) Revised: March 2001

SUBJECT: SEDIMENT TOXICITY DATA SHEET - SURVIVAL QA FORM NO: 108E

SPONSOR: Ponce Harbor/ PPB
SAMPLE ID: RFS-PH03

PROJECT NUMBER: 312253.0400
TEST SPECIES: N. virens

DATE-DAY	REP A	REP B	REP C	REPD	REP E	INITIAL
4/10/03 0	20 loaded	→				CG
4/11/03 1	N	1 d	N	N	N	HEB
4/12/03 2	N	N	N	N	N	HEB
4/13/03 3	N	N	N	N	N	KG
4/14/03 4	N	N	N	1 d	N	[illegible]
4/15/03 5	N	N	N	N	N	CG
4/16/03 6	N	N	N	N	N	[illegible]
4/17/03 7	N	N	N	N	1 d	KG
4/18/03 8	N	N	N	N	N	[illegible]
4/19/03 9	N	N	N	N	N	[illegible]
4/20/03 10	N	N	N	N	N	[illegible]
4/21/03 11	N	N	N	N	N	CG
4/22/03 12	N	N	N	N	N	CG
4/23/03 13	N	N	N	N	N	Jm
4/24/03 14	N	N	N	N	N	HEB
4/25/03 15	N	N	N	N	N	CG
4/26/03 16	N	N	N	N	N	HEB
4/27/03 17	N	N	N	N	N	HEB
4/28/03 18	N	N	N	N	N	Jm
4/29/03 19	N	N	N	N	N	CG
4/30/03 20	N	N	N	N	N	CG
5/1/03 21	N	N	N	N	N	KG
5/2/03 22	N	N	N	N	N	RD
5/3/03 23	N	N	N	N	N	RD
5/4/03 24	N	N	N	N	N	KG
5/5/03 25	N	N	N	N	N	RD
5/6/03 26	N	N	N	N	N	JH
5/7/03 27	N	N	N	N	N	[illegible]
5/8/03 28	20A	19A	20A	17A	19A	CG

2NF

MACTEC Engineering and Consulting (formerly Harding ESE)

Revised: March 2001

SUBJECT: SEDIMENT TOXICITY DATA SHEET - SURVIVAL

QA FORM NO: 108E

SPONSOR: Ponce Harbor/ PPB
SAMPLE ID: E-PH03-SC

PROJECT NUMBER: 312253.0400
TEST SPECIES: N. virens

DATE-DAY	REP A	REP B	REP C	REPD	REP E	INITIAL
4/10/03 0	20 loaded					CG
4/11/03 1	N	N	N	N	N	HEB
4/12/03 2	2 d	N	N	N	1 d	HEB
4/13/03 3	N	N	1 d	N	N	KG
4/14/03 4	N	N	N	N	N	DS
4/15/03 5	N	N	1 d	N	N	CG
4/16/03 6	1 d	N	N	N	N	DS
4/17/03 7	N	N	N	N	1 d	KG
4/18/03 8	N	N	N	N	N	DS
4/19/03 9	N	N	N	N	N	DS
4/20/03 10	N	N	N	N	N	DS
4/21/03 11	N	N	N	1 d	N	CG
4/22/03 12	N	N	N	N	N	CG
4/23/03 3	N	N	N	N	N	JM
4/24/03 4	N	N	N	N	N	HEB
4/25/03 5	N	N	N	N	N	CG
4/26/03 6	N	N	1 d	N	N	HEB
4/27/03 7	N	N	N	N	N	HEB
4/28/03 8	N	N	N	N	1 d	JM
4/29/03 9	N	N	N	N	N	CG
4/30/03 20	N	N	N	N	N	CG
5/1/03 21	N	N	N	N	N	KG
5/2/03 22	N	N	N	N	N	RD
5/3/03 23	N	N	N	1 d	N	RD
5/4/03 24	N	N	N	N	N	KG
5/5/03 25	N	N	N	N	N	RD
5/6/03 26	N	N	N	N	N	JA
5/7/03 27	N	N	N	N	N	DS
5/8/03 28	16 A	20 A	16 A	18 A	17 A	CG
	1 NF		1 NF			

MACTEC Engineering and Consulting (formerly Harding ESE) Revised: March 2001

SUBJECT: SEDIMENT TOXICITY DATA SHEET - SURVIVAL QA FORM NO: 108E

SPONSOR: Ponce Harbor/ PPB
SAMPLE ID: E-PH03-SC

PROJECT NUMBER: 312253.0400
TEST SPECIES: N. virens

DATE-DAY	REP F	REP G	REP H	REPI		INITIAL
4/10/03 0	20 / cooled →					CG
4/11/03 1	N	1 d	N	N		HEB
4/12/03 2	N	N	N	N		HEB
4/13/03 3	N	N	N	N		KG
4/14/03 4	N	N	N	1 d		DD
4/15/03 5	N	N	N	N		CG
4/16/03 6	N	N	N	N		DD
4/17/03 7	N	1 d	N	N		KG
4/18/03 8	N	N	N	N		DD
4/19/03 9	N	N	N	N		DD
4/20/03 10	N	1 d	N	N		DD
4/21/03 11	N	N	N	1 d		CG
4/22/03 12	N	N	N	N		CG
4/23/03 13	N	N	N	N		JM
4/24/03 14	N	N	N	N		HEB
4/25/03 15	N	N	N	N		CG
4/25/03 16	N	N	1 d	N		HEB
4/26/03 17	N	N	N	N		HEB
4/27/03 18	N	N	N	N		JM
4/28/03 19	N	N	N	N		CG
4/29/03 20	N	N	1 d	N		CG
4/30/03 21	N	N	N	N		KG
5/1/03 22	N	N	N	N		RD
5/2/03 23	N	N	N	N		RD
5/3/03 24	N	N	N	N		KG
5/4/03 25	N	N	N	N		RD
5/6/03 26	N	N	N	N		JA
5/7/03 27	N	N	N	N		DD
5/8/03 28	20 A	17 A	16 A	18 A		CG

2NF

Environmental Science & Engineering, Inc.
Aquatic Toxicology Laboratory
Gainesville, Florida

ESE QA Form No.: 008
Effective: Mar 1993

SUBJECT: ORGANISM RECEIPT/COLLECTION LOG

Date Recieved	Species	Population Size	Population Condition	Collector/ Supplier	Salinity/ Hardness	Temperature (C)	Initials
3/18/03	M. nasuta	2003-16 ~500	normal	ARO	—	8.5	~~JMM~~ adult
3/21/03	Americamysis bahia	320+ 2003-17	normal	ARO	28	23.1	HEB 2-days
3/21/03	M. beryllina	320+ 2003-18	normal	ARO	27	23.1	HEB 10-days
3/23/03	M. bahia	375 2003-19	normal	Aquatic Indic.	20 ppm	25.0	JA 2 days
3/23/03	M. beryllina	885 2003-20	normal	Aquatic Indic.	20 ppm	25.0	JA 10 day
3/26/03	M. edulis	~48 2003-21	normal	ARO	—	11.3	JMM adult
3/28/03	M. bahia	450+ 2003-22	normal	ARO	28 ppt	22.8	HEB 3days
3/31/03	Brine Shrimp Cysts	15 oz can	normal	Aquatic Eco-Systems	—	—	CG
4/2/03	M. nasuta	2003-23 1000	normal	ARO	—	12.7	CG adult
4/3/03	N. Virens	2003-24 500	normal	ARO	—	10.5	CG adult
4/9/03	L. plumulosus	2003-25 ~~+200~~ +500	normal	ARO	23 ppt	17.4	CG

N. virens

SYSTAT Rectangular file P:\EAT\COMMON\BIOASSAY\ppb\ponce\tissues\\Nvirens.SYD, created Mon Aug 25, 2003 at 14:15:38, contains variables:

SPECIES$ SAMPLE$ CHEM$ TISCONC

The following results are for:
CHEM$ = 1,4Dichlorov
SAMPLE$ = EPH03SC

	TISCONC
N of cases	5
Minimum	0.270
Maximum	0.450
Mean	0.354
95% CI Upper	0.437
95% CI Lower	0.271
Standard Dev	0.067

The following results are for:
CHEM$ = Naphthalene
SAMPLE$ = EPH03SC

	TISCONC
N of cases	5
Minimum	0.960
Maximum	2.320
Mean	1.618
95% CI Upper	2.301
95% CI Lower	0.935
Standard Dev	0.550

The following results are for:
CHEM$ = Biphenyl
SAMPLE$ = EPH03SC

N. virens

	TISCONC
N of cases	5
Minimum	0.160
Maximum	0.310
Mean	0.222
95% CI Upper	0.300
95% CI Lower	0.144
Standard Dev	0.063

The following results are for:
CHEM$ = Acenaphthyle
SAMPLE$ = EPH03SC

	TISCONC
N of cases	5
Minimum	0.040
Maximum	0.140
Mean	0.094
95% CI Upper	0.142
95% CI Lower	0.046
Standard Dev	0.038

The following results are for:
CHEM$ = Acenaphthen
SAMPLE$ = EPH03SC

	TISCONC
N of cases	5
Minimum	0.110
Maximum	0.410
Mean	0.236
95% CI Upper	0.404
95% CI Lower	0.068
Standard Dev	0.136

The following results are for:
CHEM$ = Fluorene
SAMPLE$ = EPH03SC

N. virens

	TISCONC
N of cases	5
Minimum	0.130
Maximum	2.520
Mean	0.648
95% CI Upper	1.949
95% CI Lower	-0.653
Standard Dev	1.048

The following results are for:
CHEM$ = Phenanthrene
SAMPLE$ = EPH03SC

	TISCONC
N of cases	5
Minimum	0.730
Maximum	8.460
Mean	2.420
95% CI Upper	6.619
95% CI Lower	-1.779
Standard Dev	3.382

The following results are for:
CHEM$ = Anthracene
SAMPLE$ = EPH03SC

	TISCONC
N of cases	5
Minimum	0.090
Maximum	7.350
Mean	1.566
95% CI Upper	5.581
95% CI Lower	-2.449
Standard Dev	3.234

The following results are for:
CHEM$ = Fluoranthene
SAMPLE$ = EPH03SC

N. virens

	TISCONC
N of cases	5
Minimum	1.800
Maximum	3.900
Mean	2.808
95% CI Upper	4.004
95% CI Lower	1.612
Standard Dev	0.963

The following results are for:
CHEM$ = Pyrene
SAMPLE$ = EPH03SC

	TISCONC
N of cases	5
Minimum	1.840
Maximum	3.330
Mean	2.472
95% CI Upper	3.213
95% CI Lower	1.731
Standard Dev	0.597

The following results are for:
CHEM$ = Benzo(a)anth
SAMPLE$ = EPH03SC

	TISCONC
N of cases	5
Minimum	0.420
Maximum	1.420
Mean	0.680
95% CI Upper	1.197
95% CI Lower	0.163
Standard Dev	0.416

The following results are for:
CHEM$ = Chrysene
SAMPLE$ = EPH03SC

N. virens

	TISCONC
N of cases	5
Minimum	1.170
Maximum	6.960
Mean	2.652
95% CI Upper	5.685
95% CI Lower	-0.381
Standard Dev	2.442

The following results are for:
CHEM$ = Benzo(b)fluo
SAMPLE$ = EPH03SC

	TISCONC
N of cases	5
Minimum	0.590
Maximum	0.770
Mean	0.656
95% CI Upper	0.744
95% CI Lower	0.568
Standard Dev	0.071

The following results are for:
CHEM$ = Benzo(k)fluo
SAMPLE$ = EPH03SC

	TISCONC
N of cases	5
Minimum	0.720
Maximum	1.100
Mean	0.902
95% CI Upper	1.092
95% CI Lower	0.712
Standard Dev	0.153

The following results are for:
CHEM$ = Benzo(a)pyre
SAMPLE$ = EPH03SC

N. virens

	TISCONC
N of cases	5
Minimum	0.540
Maximum	0.700
Mean	0.626
95% CI Upper	0.705
95% CI Lower	0.547
Standard Dev	0.063

The following results are for:
CHEM$ = Indeno(1,2,3
SAMPLE$ = EPH03SC

	TISCONC
N of cases	5
Minimum	0.380
Maximum	0.460
Mean	0.432
95% CI Upper	0.473
95% CI Lower	0.391
Standard Dev	0.033

The following results are for:
CHEM$ = Dibenz(a,h)a
SAMPLE$ = EPH03SC

	TISCONC
N of cases	5
Minimum	0.040
Maximum	0.070
Mean	0.058
95% CI Upper	0.072
95% CI Lower	0.044
Standard Dev	0.011

The following results are for:
CHEM$ = Benzo(g,h,i)
SAMPLE$ = EPH03SC

N. virens

	TISCONC
N of cases	5
Minimum	0.520
Maximum	0.680
Mean	0.596
95% CI Upper	0.681
95% CI Lower	0.511
Standard Dev	0.069

The following results are for:
CHEM$ = Ag
SAMPLE$ = EPH03SC

	TISCONC
N of cases	5
Minimum	0.020
Maximum	0.028
Mean	0.023
95% CI Upper	0.027
95% CI Lower	0.019
Standard Dev	0.003

The following results are for:
CHEM$ = As
SAMPLE$ = EPH03SC

	TISCONC
N of cases	5
Minimum	0.767
Maximum	1.076
Mean	0.921
95% CI Upper	1.082
95% CI Lower	0.759
Standard Dev	0.130

The following results are for:
CHEM$ = Cd
SAMPLE$ = EPH03SC

N. virens

	TISCONC
N of cases	5
Minimum	0.022
Maximum	0.032
Mean	0.026
95% CI Upper	0.031
95% CI Lower	0.021
Standard Dev	0.004

The following results are for:
CHEM$ = Cr
SAMPLE$ = EPH03SC

	TISCONC
N of cases	5
Minimum	0.343
Maximum	0.881
Mean	0.622
95% CI Upper	0.937
95% CI Lower	0.307
Standard Dev	0.254

The following results are for:
CHEM$ = Cu
SAMPLE$ = EPH03SC

	TISCONC
N of cases	5
Minimum	25.295
Maximum	67.181
Mean	55.547
95% CI Upper	77.926
95% CI Lower	33.167
Standard Dev	18.024

The following results are for:
CHEM$ = Hg
SAMPLE$ = EPH03SC

N. virens

	TISCONC
N of cases	5
Minimum	0.019
Maximum	0.028
Mean	0.022
95% CI Upper	0.027
95% CI Lower	0.018
Standard Dev	0.004

The following results are for:
CHEM$ = Ni
SAMPLE$ = EPH03SC

	TISCONC
N of cases	5
Minimum	0.322
Maximum	0.564
Mean	0.470
95% CI Upper	0.598
95% CI Lower	0.342
Standard Dev	0.103

The following results are for:
CHEM$ = Pb
SAMPLE$ = EPH03SC

	TISCONC
N of cases	5
Minimum	0.242
Maximum	0.345
Mean	0.283
95% CI Upper	0.348
95% CI Lower	0.218
Standard Dev	0.052

The following results are for:
CHEM$ = Se
SAMPLE$ = EPH03SC

N. virens

	TISCONC
N of cases	5
Minimum	0.329
Maximum	0.439
Mean	0.367
95% CI Upper	0.421
95% CI Lower	0.313
Standard Dev	0.044

The following results are for:
CHEM$ = Zn
SAMPLE$ = EPH03SC

	TISCONC
N of cases	5
Minimum	8.375
Maximum	21.119
Mean	12.987
95% CI Upper	19.251
95% CI Lower	6.722
Standard Dev	5.045

The following results are for:
CHEM$ = 1,4Dichlorov
SAMPLE$ = RSPH03

	TISCONC
N of cases	5
Minimum	1.360
Maximum	2.580
Mean	2.034
95% CI Upper	2.677
95% CI Lower	1.391
Standard Dev	0.518

The following results are for:
CHEM$ = Naphthalene
SAMPLE$ = RSPH03

N. virens

	TISCONC
N of cases	5
Minimum	1.650
Maximum	3.620
Mean	2.594
95% CI Upper	3.592
95% CI Lower	1.596
Standard Dev	0.804

The following results are for:
CHEM$ = Biphenyl
SAMPLE$ = RSPH03

	TISCONC
N of cases	5
Minimum	0.220
Maximum	0.370
Mean	0.262
95% CI Upper	0.338
95% CI Lower	0.186
Standard Dev	0.061

The following results are for:
CHEM$ = Acenaphthyle
SAMPLE$ = RSPH03

The following results are for:
CHEM$ = Acenaphthen
SAMPLE$ = RSPH03

	TISCONC
N of cases	5
Minimum	0.150
Maximum	0.330
Mean	0.218
95% CI Upper	0.309
95% CI Lower	0.127
Standard Dev	0.073

The following results are for:
CHEM$ = Fluorene

N. virens
SAMPLE$ = RSPH03

	TISCONC
N of cases	5
Minimum	0.180
Maximum	0.300
Mean	0.232
95% CI Upper	0.295
95% CI Lower	0.169
Standard Dev	0.051

The following results are for:
CHEM$ = Phenanthrene
SAMPLE$ = RSPH03

	TISCONC
N of cases	5
Minimum	1.050
Maximum	2.100
Mean	1.432
95% CI Upper	1.934
95% CI Lower	0.930
Standard Dev	0.404

The following results are for:
CHEM$ = Anthracene
SAMPLE$ = RSPH03

	TISCONC
N of cases	5
Minimum	0.130
Maximum	0.370
Mean	0.194
95% CI Upper	0.321
95% CI Lower	0.067
Standard Dev	0.103

The following results are for:
CHEM$ = Fluoranthene
SAMPLE$ = RSPH03

N. virens

	TISCONC
N of cases	5
Minimum	2.520
Maximum	4.800
Mean	3.778
95% CI Upper	4.997
95% CI Lower	2.559
Standard Dev	0.981

The following results are for:
CHEM$ = Pyrene
SAMPLE$ = RSPH03

	TISCONC
N of cases	5
Minimum	2.720
Maximum	4.980
Mean	4.054
95% CI Upper	5.114
95% CI Lower	2.994
Standard Dev	0.854

The following results are for:
CHEM$ = Benzo(a)anth
SAMPLE$ = RSPH03

	TISCONC
N of cases	5
Minimum	0.640
Maximum	1.960
Mean	0.956
95% CI Upper	1.656
95% CI Lower	0.256
Standard Dev	0.564

The following results are for:
CHEM$ = Chrysene
SAMPLE$ = RSPH03

N. virens

	TISCONC
N of cases	5
Minimum	1.730
Maximum	3.150
Mean	2.294
95% CI Upper	3.053
95% CI Lower	1.535
Standard Dev	0.611

The following results are for:
CHEM$ = Benzo(b)fluo
SAMPLE$ = RSPH03

	TISCONC
N of cases	5
Minimum	0.700
Maximum	1.680
Mean	1.050
95% CI Upper	1.515
95% CI Lower	0.585
Standard Dev	0.375

The following results are for:
CHEM$ = Benzo(k)fluo
SAMPLE$ = RSPH03

	TISCONC
N of cases	5
Minimum	0.960
Maximum	1.770
Mean	1.188
95% CI Upper	1.597
95% CI Lower	0.779
Standard Dev	0.329

The following results are for:
CHEM$ = Benzo(a)pyre
SAMPLE$ = RSPH03

N. virens

	TISCONC
N of cases	5
Minimum	0.930
Maximum	2.020
Mean	1.190
95% CI Upper	1.770
95% CI Lower	0.610
Standard Dev	0.467

The following results are for:
CHEM$ = Indeno(1,2,3
SAMPLE$ = RSPH03

	TISCONC
N of cases	5
Minimum	0.530
Maximum	1.240
Mean	0.730
95% CI Upper	1.088
95% CI Lower	0.372
Standard Dev	0.289

The following results are for:
CHEM$ = Dibenz(a,h)a
SAMPLE$ = RSPH03

	TISCONC
N of cases	5
Minimum	0.060
Maximum	0.160
Mean	0.084
95% CI Upper	0.138
95% CI Lower	0.030
Standard Dev	0.043

The following results are for:
CHEM$ = Benzo(g,h,i)
SAMPLE$ = RSPH03

N. virens

	TISCONC
N of cases	5
Minimum	0.700
Maximum	1.340
Mean	0.874
95% CI Upper	1.204
95% CI Lower	0.544
Standard Dev	0.266

The following results are for:
CHEM$ = Ag
SAMPLE$ = RSPH03

	TISCONC
N of cases	5
Minimum	0.030
Maximum	0.041
Mean	0.035
95% CI Upper	0.042
95% CI Lower	0.028
Standard Dev	0.006

The following results are for:
CHEM$ = As
SAMPLE$ = RSPH03

	TISCONC
N of cases	5
Minimum	1.452
Maximum	2.108
Mean	1.745
95% CI Upper	2.152
95% CI Lower	1.338
Standard Dev	0.328

The following results are for:
CHEM$ = Cd
SAMPLE$ = RSPH03

N. virens

	TISCONC
N of cases	5
Minimum	0.030
Maximum	0.043
Mean	0.036
95% CI Upper	0.044
95% CI Lower	0.028
Standard Dev	0.006

The following results are for:
CHEM$ = Cr
SAMPLE$ = RSPH03

	TISCONC
N of cases	5
Minimum	0.817
Maximum	1.477
Mean	1.128
95% CI Upper	1.489
95% CI Lower	0.767
Standard Dev	0.291

The following results are for:
CHEM$ = Cu
SAMPLE$ = RSPH03

	TISCONC
N of cases	5
Minimum	30.974
Maximum	90.028
Mean	66.556
95% CI Upper	98.157
95% CI Lower	34.956
Standard Dev	25.450

The following results are for:
CHEM$ = Hg
SAMPLE$ = RSPH03

N. virens

	TISCONC
N of cases	5
Minimum	0.033
Maximum	0.044
Mean	0.037
95% CI Upper	0.043
95% CI Lower	0.032
Standard Dev	0.005

The following results are for:
CHEM$ = Ni
SAMPLE$ = RSPH03

	TISCONC
N of cases	5
Minimum	0.566
Maximum	1.140
Mean	0.783
95% CI Upper	1.050
95% CI Lower	0.517
Standard Dev	0.215

The following results are for:
CHEM$ = Pb
SAMPLE$ = RSPH03

	TISCONC
N of cases	5
Minimum	0.335
Maximum	1.791
Mean	0.852
95% CI Upper	1.586
95% CI Lower	0.118
Standard Dev	0.591

The following results are for:
CHEM$ = Se
SAMPLE$ = RSPH03

N. virens

	TISCONC
N of cases	5
Minimum	0.431
Maximum	0.689
Mean	0.524
95% CI Upper	0.664
95% CI Lower	0.384
Standard Dev	0.113

The following results are for:
CHEM$ = Zn
SAMPLE$ = RSPH03

	TISCONC
N of cases	5
Minimum	10.953
Maximum	36.331
Mean	22.275
95% CI Upper	35.062
95% CI Lower	9.488
Standard Dev	10.298

SYSTAT Rectangular file P:\EAT\COMMON\BIOASSAY\ppb\ponce\tissues\Mnasuta.SYD,
created Mon Aug 25, 2003 at 11:49:07, contains variables:

SPECIES$ SAMPLE$ CHEM$ TISCONC

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = 1,4Dichlorov

	TISCONC
N of cases	5
Minimum	0.070
Maximum	0.090
Mean	0.082
95% CI Upper	0.092
95% CI Lower	0.072
Standard Dev	0.008

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = 1,4Dichlorov

	TISCONC
N of cases	5
Minimum	0.060
Maximum	0.080
Mean	0.068
95% CI Upper	0.078
95% CI Lower	0.058
Standard Dev	0.008

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Naphthalene

M.nasuta

	TISCONC
N of cases	5
Minimum	0.310
Maximum	0.510
Mean	0.350
95% CI Upper	0.461
95% CI Lower	0.239
Standard Dev	0.089

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Naphthalene

	TISCONC
N of cases	5
Minimum	0.240
Maximum	0.350
Mean	0.300
95% CI Upper	0.350
95% CI Lower	0.250
Standard Dev	0.040

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Biphenyl

	TISCONC
N of cases	5
Minimum	0.060
Maximum	0.130
Mean	0.082
95% CI Upper	0.116
95% CI Lower	0.048
Standard Dev	0.028

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Biphenyl

M.nasuta

	TISCONC
N of cases	5
Minimum	0.060
Maximum	0.100
Mean	0.078
95% CI Upper	0.096
95% CI Lower	0.060
Standard Dev	0.015

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Acenaphthyle

	TISCONC
N of cases	5
Minimum	0.015
Maximum	0.015
Mean	0.015
95% CI Upper	0.015
95% CI Lower	0.015
Standard Dev	0.000

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Acenaphthyle

	TISCONC
N of cases	5
Minimum	0.015
Maximum	0.015
Mean	0.015
95% CI Upper	0.015
95% CI Lower	0.015
Standard Dev	0.000

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Acenaphthen

M.nasuta

	TISCONC
N of cases	5
Minimum	0.025
Maximum	0.040
Mean	0.028
95% CI Upper	0.036
95% CI Lower	0.020
Standard Dev	0.007

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Acenaphthen

	TISCONC
N of cases	5
Minimum	0.025
Maximum	0.025
Mean	0.025
95% CI Upper	0.025
95% CI Lower	0.025
Standard Dev	0.000

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Fluorene

	TISCONC
N of cases	5
Minimum	0.060
Maximum	0.130
Mean	0.084
95% CI Upper	0.118
95% CI Lower	0.050
Standard Dev	0.027

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Fluorene

M.nasuta

	TISCONC
N of cases	5
Minimum	0.060
Maximum	0.080
Mean	0.066
95% CI Upper	0.077
95% CI Lower	0.055
Standard Dev	0.009

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Phenanthrene

	TISCONC
N of cases	5
Minimum	0.390
Maximum	0.720
Mean	0.502
95% CI Upper	0.663
95% CI Lower	0.341
Standard Dev	0.130

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Phenanthrene

	TISCONC
N of cases	5
Minimum	0.320
Maximum	0.400
Mean	0.372
95% CI Upper	0.411
95% CI Lower	0.333
Standard Dev	0.031

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Anthracene

M.nasuta

	TISCONC
N of cases	5
Minimum	0.020
Maximum	0.040
Mean	0.033
95% CI Upper	0.043
95% CI Lower	0.023
Standard Dev	0.008

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Anthracene

	TISCONC
N of cases	5
Minimum	0.020
Maximum	0.040
Mean	0.033
95% CI Upper	0.042
95% CI Lower	0.024
Standard Dev	0.008

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Fluoranthene

	TISCONC
N of cases	5
Minimum	0.940
Maximum	2.200
Mean	1.452
95% CI Upper	2.027
95% CI Lower	0.877
Standard Dev	0.463

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Fluoranthene

M.nasuta

	TISCONC
N of cases	5
Minimum	0.840
Maximum	1.300
Mean	1.070
95% CI Upper	1.291
95% CI Lower	0.849
Standard Dev	0.178

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Pyrene

	TISCONC
N of cases	5
Minimum	0.090
Maximum	1.550
Mean	0.902
95% CI Upper	1.565
95% CI Lower	0.239
Standard Dev	0.534

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Pyrene

	TISCONC
N of cases	5
Minimum	0.650
Maximum	0.790
Mean	0.732
95% CI Upper	0.799
95% CI Lower	0.665
Standard Dev	0.054

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Benzo(a)anth

M.nasuta

	TISCONC
N of cases	5
Minimum	0.090
Maximum	0.210
Mean	0.138
95% CI Upper	0.194
95% CI Lower	0.082
Standard Dev	0.045

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Benzo(a)anth

	TISCONC
N of cases	5
Minimum	0.070
Maximum	0.130
Mean	0.098
95% CI Upper	0.130
95% CI Lower	0.066
Standard Dev	0.026

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Chrysene

	TISCONC
N of cases	5
Minimum	0.460
Maximum	0.850
Mean	0.624
95% CI Upper	0.800
95% CI Lower	0.448
Standard Dev	0.142

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Chrysene

M.nasuta

	TISCONC
N of cases	5
Minimum	0.410
Maximum	0.590
Mean	0.520
95% CI Upper	0.612
95% CI Lower	0.428
Standard Dev	0.074

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Benzo(b)fluo

	TISCONC
N of cases	5
Minimum	0.150
Maximum	0.240
Mean	0.194
95% CI Upper	0.238
95% CI Lower	0.150
Standard Dev	0.036

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Benzo(b)fluo

	TISCONC
N of cases	5
Minimum	0.110
Maximum	0.160
Mean	0.140
95% CI Upper	0.163
95% CI Lower	0.117
Standard Dev	0.019

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Benzo(k)fluo

M.nasuta

	TISCONC
N of cases	5
Minimum	0.100
Maximum	0.250
Mean	0.150
95% CI Upper	0.222
95% CI Lower	0.078
Standard Dev	0.058

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Benzo(k)fluo

	TISCONC
N of cases	5
Minimum	0.110
Maximum	0.160
Mean	0.136
95% CI Upper	0.159
95% CI Lower	0.113
Standard Dev	0.018

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Benzo(a)pyre

	TISCONC
N of cases	5
Minimum	0.080
Maximum	0.150
Mean	0.106
95% CI Upper	0.138
95% CI Lower	0.074
Standard Dev	0.026

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Benzo(a)pyre

	TISCONC
N of cases	5
Minimum	0.090
Maximum	0.120
Mean	0.104
95% CI Upper	0.121
95% CI Lower	0.087
Standard Dev	0.013

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Indeno(1,2,3

	TISCONC
N of cases	5
Minimum	0.025
Maximum	0.030
Mean	0.027
95% CI Upper	0.030
95% CI Lower	0.024
Standard Dev	0.003

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Indeno(1,2,3

	TISCONC
N of cases	5
Minimum	0.025
Maximum	0.030
Mean	0.029
95% CI Upper	0.032
95% CI Lower	0.026
Standard Dev	0.002

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Dibenz(a,h)a

M.nasuta

	TISCONC
N of cases	5
Minimum	0.025
Maximum	0.025
Mean	0.025
95% CI Upper	0.025
95% CI Lower	0.025
Standard Dev	0.000

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Dibenz(a,h)a

	TISCONC
N of cases	5
Minimum	0.025
Maximum	0.025
Mean	0.025
95% CI Upper	0.025
95% CI Lower	0.025
Standard Dev	0.000

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Benzo(g,h,i)

	TISCONC
N of cases	5
Minimum	0.030
Maximum	0.050
Mean	0.038
95% CI Upper	0.048
95% CI Lower	0.028
Standard Dev	0.008

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Benzo(g,h,i)

M.nasuta

	TISCONC
N of cases	5
Minimum	0.020
Maximum	0.050
Mean	0.033
95% CI Upper	0.048
95% CI Lower	0.018
Standard Dev	0.012

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Ag

	TISCONC
N of cases	5
Minimum	0.011
Maximum	0.019
Mean	0.014
95% CI Upper	0.018
95% CI Lower	0.010
Standard Dev	0.003

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Ag

	TISCONC
N of cases	5
Minimum	0.010
Maximum	0.016
Mean	0.012
95% CI Upper	0.015
95% CI Lower	0.010
Standard Dev	0.002

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = As

M.nasuta

	TISCONC
N of cases	5
Minimum	1.260
Maximum	2.459
Mean	1.909
95% CI Upper	2.465
95% CI Lower	1.352
Standard Dev	0.448

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = As

	TISCONC
N of cases	5
Minimum	1.660
Maximum	2.097
Mean	1.814
95% CI Upper	2.040
95% CI Lower	1.588
Standard Dev	0.182

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Cd

	TISCONC
N of cases	5
Minimum	0.015
Maximum	0.031
Mean	0.019
95% CI Upper	0.027
95% CI Lower	0.011
Standard Dev	0.007

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Cd

M.nasuta

	TISCONC
N of cases	5
Minimum	0.015
Maximum	0.022
Mean	0.019
95% CI Upper	0.022
95% CI Lower	0.015
Standard Dev	0.003

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Cr

	TISCONC
N of cases	5
Minimum	0.110
Maximum	0.288
Mean	0.155
95% CI Upper	0.248
95% CI Lower	0.062
Standard Dev	0.075

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Cr

	TISCONC
N of cases	5
Minimum	0.120
Maximum	0.498
Mean	0.211
95% CI Upper	0.411
95% CI Lower	0.011
Standard Dev	0.161

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Cu

M.nasuta

	TISCONC
N of cases	5
Minimum	0.881
Maximum	1.728
Mean	1.111
95% CI Upper	1.546
95% CI Lower	0.677
Standard Dev	0.350

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Cu

	TISCONC
N of cases	5
Minimum	0.843
Maximum	1.854
Mean	1.153
95% CI Upper	1.657
95% CI Lower	0.650
Standard Dev	0.405

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Hg

	TISCONC
N of cases	5
Minimum	0.005
Maximum	0.014
Mean	0.007
95% CI Upper	0.012
95% CI Lower	0.003
Standard Dev	0.004

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Hg

	TISCONC
N of cases	5
Minimum	0.006
Maximum	0.008
Mean	0.007
95% CI Upper	0.008
95% CI Lower	0.006
Standard Dev	0.001

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Ni

	TISCONC
N of cases	5
Minimum	0.207
Maximum	0.368
Mean	0.256
95% CI Upper	0.336
95% CI Lower	0.177
Standard Dev	0.064

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Ni

	TISCONC
N of cases	5
Minimum	0.227
Maximum	0.440
Mean	0.287
95% CI Upper	0.397
95% CI Lower	0.177
Standard Dev	0.088

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Pb

M.nasuta

	TISCONC
N of cases	5
Minimum	0.071
Maximum	0.145
Mean	0.089
95% CI Upper	0.128
95% CI Lower	0.050
Standard Dev	0.031

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Pb

	TISCONC
N of cases	5
Minimum	0.069
Maximum	0.127
Mean	0.082
95% CI Upper	0.114
95% CI Lower	0.051
Standard Dev	0.025

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Se

	TISCONC
N of cases	5
Minimum	0.205
Maximum	0.393
Mean	0.281
95% CI Upper	0.367
95% CI Lower	0.195
Standard Dev	0.069

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Se

M.nasuta

	TISCONC
N of cases	5
Minimum	0.262
Maximum	0.296
Mean	0.285
95% CI Upper	0.301
95% CI Lower	0.268
Standard Dev	0.013

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Zn

	TISCONC
N of cases	5
Minimum	5.316
Maximum	10.313
Mean	7.130
95% CI Upper	9.460
95% CI Lower	4.799
Standard Dev	1.877

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Zn

	TISCONC
N of cases	5
Minimum	5.623
Maximum	7.057
Mean	6.248
95% CI Upper	6.990
95% CI Lower	5.506
Standard Dev	0.598

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = 1,4Dichlorov

	TISCONC
5% CI Upper	0.082
5% CI Lower	0.082

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = 1,4Dichlorov

	TISCONC
5% CI Upper	0.068
5% CI Lower	0.068

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Naphthalene

	TISCONC
5% CI Upper	0.353
5% CI Lower	0.347

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Naphthalene

	TISCONC
5% CI Upper	0.301
5% CI Lower	0.299

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Biphenyl

	TISCONC
5% CI Upper	0.083
5% CI Lower	0.081

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Biphenyl

	TISCONC
5% CI Upper	0.078
5% CI Lower	0.078

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Acenaphthyle

	TISCONC
	0.01
5% CI Upper	5
	0.01
5% CI Lower	5

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Acenaphthyle

	TISCONC
	0.01
5% CI Upper	5
	0.01
5% CI Lower	5

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Acenaphthen

	TISCONC
	0.02
5% CI Upper	8
	0.02
5% CI Lower	8

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Acenaphthen

	TISCONC
	0.02
5% CI Upper	5
	0.02
5% CI Lower	5

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Fluorene

	TISCONC
	0.08
5% CI Upper	5
	0.08
5% CI Lower	3

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Fluorene

M.nasuta

	TISCONC
5% CI Upper	0.06 6
5% CI Lower	0.06 6

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Phenanthrene

	TISCONC
5% CI Upper	0.50 6
5% CI Lower	0.49 8

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Phenanthrene

	TISCONC
5% CI Upper	0.37 3
5% CI Lower	0.37 1

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Anthracene

	TISCONC
5% CI Upper	0.03 3
5% CI Lower	0.03 3

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Anthracene

	TISCONC
5% CI Upper	0.03 3
5% CI Lower	0.03 3

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Fluoranthene

	TISCONC
5% CI Upper	1.46 6
5% CI Lower	1.43 8

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Fluoranthene

	TISCONC
5% CI Upper	1.075
5% CI Lower	1.065

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Pyrene

	TISCONC
5% CI Upper	0.918
5% CI Lower	0.886

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Pyrene

	TISCONC
5% CI Upper	0.734
5% CI Lower	0.730

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Benzo(a)anth

	TISCONC
5% CI Upper	0.139
5% CI Lower	0.137

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Benzo(a)anth

	TISCONC
5% CI Upper	0.099
5% CI Lower	0.097

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Chrysene

	TISCONC
5% CI Upper	0.628
5% CI Lower	0.620

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Chrysene

	TISCONC
5% CI Upper	0.522
5% CI Lower	0.518

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Benzo(b)fluo

	TISCONC
5% CI Upper	0.195
5% CI Lower	0.193

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Benzo(b)fluo

	TISCONC
5% CI Upper	0.141
5% CI Lower	0.139

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Benzo(k)fluo

	TISCONC
5% CI Upper	0.152
5% CI Lower	0.148

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Benzo(k)fluo

	TISCONC
5% CI Upper	0.137
5% CI Lower	0.135

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Benzo(a)pyre

	TISCONC
	0.10
5% CI Upper	7
	0.10
5% CI Lower	5

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Benzo(a)pyre

	TISCONC
	0.10
5% CI Upper	4
	0.10
5% CI Lower	4

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Indeno(1,2,3

	TISCONC
	0.02
5% CI Upper	7
	0.02
5% CI Lower	7

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Indeno(1,2,3

	TISCONC
	0.02
5% CI Upper	9
	0.02
5% CI Lower	9

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Dibenz(a,h)a

	TISCONC
	0.02
5% CI Upper	5
	0.02
5% CI Lower	5

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Dibenz(a,h)a

	TISCONC
5% CI Upper	0.025
5% CI Lower	0.025

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Benzo(g,h,i)

	TISCONC
5% CI Upper	0.038
5% CI Lower	0.038

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Benzo(g,h,i)

	TISCONC
5% CI Upper	0.033
5% CI Lower	0.033

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Ag

	TISCONC
5% CI Upper	0.014
5% CI Lower	0.014

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Ag

	TISCONC
5% CI Upper	0.012
5% CI Lower	0.012

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = As

	TISCONC
5% CI Upper	1.922
5% CI Lower	1.895

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = As

	TISCONC
5% CI Upper	1.820
5% CI Lower	1.809

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Cd

	TISCONC
5% CI Upper	0.020
5% CI Lower	0.019

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Cd

	TISCONC
5% CI Upper	0.019
5% CI Lower	0.019

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Cr

	TISCONC
5% CI Upper	0.157
5% CI Lower	0.153

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Cr

	TISCONC
5% CI Upper	0.216
5% CI Lower	0.206

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Cu

	TISCONC
5% CI Upper	1.12 2
5% CI Lower	1.10 1

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Cu

	TISCONC
5% CI Upper	1.16 6
5% CI Lower	1.14 1

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Hg

	TISCONC
5% CI Upper	0.00 7
5% CI Lower	0.00 7

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Hg

	TISCONC
5% CI Upper	0.00 7
5% CI Lower	0.00 7

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Ni

	TISCONC
5% CI Upper	0.25 8
5% CI Lower	0.25 5

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Ni

	TISCONC
5% CI Upper	0.29 0
5% CI Lower	0.28 4

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Pb

	TISCONC
5% CI Upper	0.090
5% CI Lower	0.088

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Pb

	TISCONC
5% CI Upper	0.083
5% CI Lower	0.082

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Se

	TISCONC
5% CI Upper	0.283
5% CI Lower	0.279

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Se

	TISCONC
5% CI Upper	0.285
5% CI Lower	0.284

The following results are for:
SAMPLE$ = RSPH03
CHEM$ = Zn

	TISCONC
5% CI Upper	7.186
5% CI Lower	7.074

The following results are for:
SAMPLE$ = EPH03SC
CHEM$ = Zn

M.nasuta

	TISCONC
5% CI Upper	6.266
5% CI Lower	6.230

Appendix G

Field Quality Assurance Audit Report

Field Quality Assurance Audit Report for Ponce Harbor, March 2003

The PPB Quality Assurance Coordinator performed a field quality assurance audit beginning March 3, 2003 and ending March 7, 2003. All aspects of on-site preparations, water and sediment sampling, field data collection, sample handling and shipping, and documentation were observed. This report summarizes the findings and the adequacy of the field effort with regard to meeting project goals.

Project Description

This project was designed to be an evaluation of Ponce Harbor sediment that was proposed for dredging under the oversight of the Puerto Rico Infrastructure Financing Authority (AFI). The evaluation was performed for 103 certification in accordance with the 1991 "Green Book" ("Evaluation of Dredged Material Proposed for Ocean Disposal - Testing Manual") with guidance from the US Environmental Protection Agency (USEPA) Region II draft document "Guidance for Performing Tests on Dredged Material Proposed for Ocean Disposal", dated 18 December 1992 with undated updates attached. The evaluation includes chemical analysis of sediments, waters, and elutriates; and water column and sediment bioassays; and bioaccumulation studies.

The current ocean disposal evaluation of Ponce Harbor considers potential dredge material (DM) from widening the channel and turning basin and deepening them to a depth of 53 feet plus 2 feet allowable overdraft. Approximately 1.4 million cubic yards of material would be dredged from the harbor.

Other concurrent work was performed to address the physical and chemical characterization of sediments from the Ponce Ocean Dredged Material Disposal Site (ODMDS) and reference stations. This concurrent characterization is not included in this report.

For this sediment evaluation, the harbor was divided into two vertical project segments. Previous sampling at Ponce Harbor had identified that generally a fine sand layer of sediment overlies a thicker layer of silty clay sediment. The upper fine sand layer was to be sampled as the first project segment. The deeper silty clay sediment was to be the second project segment.

Sediment samples were to be collected using a vibracore from nine harbor locations. Sediment cores were to be divided to isolate the two sediment layers. Sediment from each station and layer was to be characterized separately. A composite sample of each harbor sediment layer was to be comprehensively tested for physical, chemical, and toxicological parameters along with a reference sediment composite and a control sediment.

A Work and Quality Assurance Plan (W/QAP) was prepared for all aspects of the project. This plan was submitted to and approved by EPA Region II prior to the beginning of field work.

Organization

Work for this sediment evaluation project was performed for the Infrastructure Financing Authority of Puerto Rico with PPB Environmental Laboratories as the prime contractor. Ward Dickens was the PPB project director and Bryan Cotter was the PPB project manager. Field sampling and data collection activities was performed by Continental Shelf Associates with Frank Johnson the designated field team leader in charge of supervising all field activities. To supplement the field team provided by Continental Shelf, Water & Air Research provided two field technicians who worked under the direct supervision of Mr. Johnson.

Mr. Johnson is a degreed marine scientist with over 17 years of relevant experience. All field team members had over five years of experience with the tasks they were assigned to perform by Mr. Johnson.

Communication

Presampling coordination meetings were held to coordinate the logistical and teaming support needed to accomplish the field sampling and data collection effort. Before traveling to Puerto Rico, each individual was provided a copy of the W/QAP.

Mr. Johnson and the logistical support specialist from Continental Shelf Associates, Mark Schroeder, adequately communicated with the Ponce Harbor Director and his staff to coordinate storage of materials and equipment and proper access by the field team.

In the field, communication was maintained with cellular telephones among field team members, the PPB project director, AFI representatives, harbor staff, points of contacts for the analytical laboratories, and the Environmental Protection Agency project manager.

Execution of Field Data Collection

Field measurements were made with a Hydrolab® H2O Water Quality Multimeter for temperature, dissolved oxygen, depth, pH, conductivity, salinity, and turbidity. Instruments were calibrated at the beginning of each day according to the prescribed standard operating procedure (SOP). Similarly end-of-day calibration checks were performed each day. No calibration problems were experienced.

In situ measurements were made at mid-depth at each harbor station prior to beginning sediment collection. Data were recorded with indelible ink on a preprinted field sheet designed specifically for this project and reviewed before use by the PPB Project Quality Assurance Coordinator.

Execution of Sampling Procedures

Prior to arrival in Ponce Harbor, the vibracore working area at the bow of the barge was cleaned to remove materials that might potentially contaminate retrieved sediments. Upon inspection immediately prior to sampling, the area appeared free of petroleum residues, metal shavings and grid, and other debris that potentially could contaminate samples.

Prior to sampling a clean work area was created where sediment cores retrieved in the polyethylene sleeves could be processed. PVC pipes were cut in half length-wise and the inner surface of these were covered with Teflon®-sheeting. During the core processing procedure, sleeves filled with sediment were placed in the trough of these half pipes. The sleeves were then slit along the top and the sleeve material laid back upon the Teflon®-sheeting. Typically no contact was made between the sediment and the Teflon®-sheeting.

Sediment handling utensils, constructed of appropriate materials, and the Teflon®-sheeting on the half pipes were decontaminated between each station following the SOP prescribed by the relevant W/QAP.

A running tabulation was maintained to calculate how much sediment was collected at each station in order to generate a 50-gallon composite sample for the fine sand and silty clay project segments. The upper fine sand layer was not expected to be found at all stations and observations during sampling showed that it was not present at any of the nine stations.

Sediment sampling stations were located using a Coast Guard beacon differentially-corrected global positioning system (GPS) receiver. Location data was collected using the NAD 83 datum in decimal degrees. Each station was marked with a buoy attached to a weighted line. The sampling barge then approached the buoy from downwind and was positioned so that the bow area that had been prepared for vibracore sampling was secured in place over the buoy and upwind of the petroleum fueled equipment. The PPB Quality Assurance Coordinator verified the final sampling locations at several stations using a hand-held WAAS corrected GPS receiver.

The field team collected the water samples last to provide the greatest amount of time for shipment and laboratory processing of elutriate samples. Water was collected directly into the sample containers provided by the analytical laboratory and in the manner prescribed by the W/QAP.

General Sampling Observations

Observing sampling activities showed that the field team worked and communicated well together which indicates appropriate planning by the field team leader since a real potential existed for language communication problems with non-bilingual team members.

Field team members who worked with the sediment in a manner that could potentially compromise sample integrity wore clean nitrile gloves. Gloves were changed between

stations and were changed by team members when torn or otherwise believed to be compromised.

There was adequate equipment redundancy to ensure that failure of one piece of equipment did not stop field activities or cause a loss of data that might adversely impact the project.

The field team leader had formulated a sampling strategy that efficiently minimized field time. Sampling at the more exposed stations was prioritized so that during periods of strong wind (~30 knots) sampling activities could be moved to the more protected portion of the harbor.

Sample Handling, Storage, and Transport

Sediment samples were transferred from the slit-open polyethylene sleeves to 1-gallon glass jars with Teflon® lid liners. Sample containers were covered with plastic mesh to protect them from direct glass to glass contact between sample containers. Duct tape labels were placed in the shoulder of the containers and beneath the plastic mesh. Lids were labeled with indelible ink to facilitate container identification in the field. Water sample containers were treated in the same manner as sediment containers.

Both water and sediment containers were chilled with wet ice within 15 minutes of sample collection. For samples remaining on-site overnight, sample containers were re-iced after melted ice water was removed from the ice chest. Samples not shipped on the day of collection were stored in locked vehicles or secured in a locked equipment storage area.

Samples were shipped to the appropriate locations by Federal Express, which came to the sampling vessel docking area for pickup. The after-sampling follow-up with the laboratory revealed that no bottles were received broken and only a single lid was found to be cracked and the integrity of that sample was not believed to be compromised.

Documentation

Project-specific field sheets were prepared for various aspects of the sediment and water sampling and field data collection associated with this Section 103 evaluation. Preprinted field sheets were designed for water sampling, sediment sampling, Hydrolab® calibration, and in situ field measurement data collection. Data were entered onto this sheets with indelible ink. Corrected entries were made with a single strike-through line through the information to be changed and the initials of the data entry field team member which is consistent with the SOPs of the sampling contractor. A chronological listing of field activities also was maintained in a separate field logbook.

Information on field sheets included station depth, vibracore penetration length, volume of sediment collected from each project segment, date, times, sediment description, and other observations.

Each sample container was properly identified in one or more places. Sample identifiers were used that allow the station from which they were collected to be determined. Chain of custody forms were used to manifest the samples to the laboratory. Custody seals were used on each ice chest that was used as a shipping container for sample transport to the laboratory.

Adherence to Planned Schedule

Initial planning had allowed for two harbor stations to be sampled each day and sampling was planned to start on Tuesday and conclude on Saturday. Actual sampling began on Tuesday and concluded on Thursday with two stations, three stations, and four stations being sampled on consecutive days. Water sampling was put off until Friday since the last of the samples were shipped on that day.

Sampling and data collection were concluded approximately one and a half days ahead of schedule. However, it does not appear that an excessive amount of time was allotted for the field activities given the variety of problems that can arise and the impact that weather can have on the ability to accomplish field-related tasks.

Problems Encountered and Corrective Actions Taken

During routine water quality field measurements, an elevated salinity reading (40.1 ppt) was obtained. This reading appeared to be elevated because other measurements had yielded values of 35 and 36 ppt. This salinity value was verified using a refractometer (Model # RHS-10ATC). No corrective action was required.

During retrieval of sediment cores at the first station sampled, a potential for transferring paint from the cleaned deck to the vibracore nosecone was observed. As a corrective action a piece of plywood was used as a shield to prevent paint from being scraped from the deck and potentially contaminating the sediment or vibracore.

During the first two sampling days, afternoon winds from the east or southeast were blowing at approximately 30 knots and causing swells that precluded sampling at the outer stations. The only remedy was to sample the outer stations in the early morning before the wind became so strong. The "corrective action", or change in approach, was to start the work day an hour earlier.

Sampling was to occur at predetermined locations that were believed to have known water depths. At some stations there was a discrepancy between the anticipated and the observed depths. Guidance was requested from the EPA Region II Project Manager who directed the field team to collect sediment at the predetermined locations regardless of the water depths encountered.

Cables on the crane became twisted and the attached tackle became snagged on the crane due in part to the wind and waves. This potentially hazardous condition necessitated that the crane be lowered and several crewmembers and a small tugboat were required to free the

cables and tackle safely. For reference as to the sampling impact, this situation took approximately one hour to be rectified.

One 1-gallon sediment container was found to be missing its Teflon® lid liner. This container had a note attached to it and was returned to the laboratory by using it as a spacer to prevent other, full containers from moving during transport.

On the first day for sediment sample shipment, custody seals could not be located and cellular phone communications with the knowledgeable person on a second field team failed. As a corrective action, strapping tape was used to make signed and dated custody seals. Because this tape circumvented each ice chest several times, it was vulnerable to being snagged, rolled, and broken on the textured-metal floor of the Federal Express truck, which occurred in some incidents. Custody seals were located and used for subsequent shipments.

Adequacy of Field Effort for Project Needs

All aspects of the field effort were observed.

Appropriate cleaning and decontamination procedures were followed. Sampling and field measurements occurred as described in the approved W/QAP and followed the approved SOPs. Samples were handled, stored, and transported in the prescribed manner and were received by the laboratories in good condition. Data was legibly recorded on field sheets and in a project logbook. There was good communication among the field team, the laboratories, the EPA Region II project manager, and AFI.

Some problems were encountered and all were resolved successfully either technically, e.g., fixing broken equipment, or administratively, e.g., accepting that the anticipated station depth might be inaccurate and preceding to sample at the predetermined location as directed.

The finding of this audit is that all samples and data are adequate quality for their intended project uses.