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FINAL

THAMES RIVER
RAPID SEDIMENT CHARACTERIZATION PILOT STUDY

WORK PLAN/FIELD SAMPLING PLAN

NAVAL SUBMARINE BASE – NEW LONDON, GROTON, CT

Prepared for:

DEPARTMENT OF THE NAVY
NAVAL FACILITIES ENGINEERING COMMAND
NORTHERN DIVISION

10 Industrial Highway
Mail Stop, #82
Lester, PA 19113-2090

Prepared by:

Battelle
397 Washington St.
Duxbury, MA 02332

Neptune and Company
1505 15th Street, Suite B
Los Alamos, NM 87544

Contract No. N62472-00-D-1300

Task Order 0005

Project No. G486305

June 2003

DEPARTMENT OF THE NAVY
ENGINEERING FIELD ACTIVITY, NORTHEAST
NAVAL FACILITIES ENGINEERING COMMAND
10 INDUSTRIAL HIGHWAY
MAIL STOP, #82
LESTER, PA 19113-2090

IN REPLY REFER TO

5090
Code EV23\ME
June 12, 2003

Ms. Kymberlee Keckler
U.S. Environmental Protection Agency, Region I
1 Congress Street
Suite 1100 (HBT)
Boston, MA 02114-2023

Mr. Mark Lewis
Connecticut Department of Environmental Protection
Bureau of Water Management
79 Elm Street
Hartford, CT 06106

Dear Ms. Keckler and Mr. Lewis:

SUBJECT: FINAL WORK PLAN/FIELD SAMPLING PLAN FOR THE THAMES RIVER RAPID SEDIMENT CHARACTERIZATION PILOT STUDY, NAVAL SUBMARINE BASE NEW LONDON, GROTON, CT

Please find enclosed 2 copies of the Final Work Plan and Field Sampling Plan for the Thames River Rapid Sediment Characterization Pilot Study for your information. Please note that this fieldwork will begin June 16, 2003.

If you have any questions concerning this submittal please contact me at (610) 595-0567 extension 162.

Sincerely,

MARK EVANS
Remedial Project Manager
By direction of the
Commanding Officer

Copy to: Ms. Melissa Griffin, NSB-NLON (3 copies)
Ms. Jennifer Stump, Gannett Fleming
Mr. Ken Finkelstein, NOAA

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NAVAL SUBMARINE BASE - NEW LONDON
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DISTRIBUTION LIST

This document will be distributed to the following project participants once all approval signatures have been received:

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FINAL

THAMES RIVER
RAPID SEDIMENT CHARACTERIZATION PILOT STUDY
WORK PLAN/FIELD SAMPLING PLAN
NAVAL SUBMARINE BASE – NEW LONDON
GROTON, CONNECTICUT

June 12, 2003

REVIEW AND APPROVALS

Project Manager, Battelle Don Gunster	Donald Gunster Name	Date: 6/12/03
Technical Advisor, Battelle Patty White	Patty White Name	Date: 6/12/03
Project QA Manager, Battelle Rosanna Buhl	Rosanna Buhl Name	Date: 6-12-03
Field Leader, Battelle Alex Mansfield	Robert E. Helm for A.M. Name	Date: 6/12/03
Navy Project Manager Mark Evans	Mark Evans Name	Date: 6/11/03
Navy QA Officer Deb Felton	Deborah L. Felton Name	Date: 6/11/2003

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1.0 BACKGROUND

Naval Submarine Base New London (NSB-NLON) is located along the Thames River and lies within the towns of Ledyard and Groton, CT. The Lower Subase is a narrow strip of land that generally forms the western boundary of NSB-NLON and parallels the Thames River. As part of the Phase I and II Remedial Investigations (RI) completed for the Lower Subase, seven zones were identified by grouping Installation Restoration (IR) sites in close proximity to each other. In the RI, the areas adjacent to these seven zones in the Thames River were investigated for potential contamination from site-related activities. The seven offshore zones were assessed for potential risk using a weight-of-evidence approach. Two of these offshore zones (Zones 4 and 7) were identified in the Lower Subase RI as posing low to moderate potential ecological risk based on a small number of samples (Brown and Root, 1999). The other five zones were characterized as posing low potential ecological risk. Zone 7 subsequently was dredged in connection with the homeporting of a new class of submarines. This work included replacement of Pier 17 and dredging adjacent to Pier 15. However, no confirmation samples were taken after dredging to confirm the absence of residual sediment contamination. Additionally, dredging is planned in the northern part of Zone 4 in 2005 as part of the Pier 4/Pier 6 reconstruction project.

The Pier 1 Marine Railway sediment (not part of the Lower Subase RI) was sampled in October of 1999 after remnants of marine vessel overhaul activities were discovered during the draining of the railway for a building construction project. The purpose of the investigation was to evaluate whether chemicals from these activities had been released and transported to sediments in the Thames River. Results of this small-scale study indicated that concentrations of metals, polycyclic aromatic hydrocarbons (PAHs) and polychlorinated biphenyls (PCBs) in sediment exceed benchmark values, and that chemical concentrations in the Pier 1 area decreased from north to south away from the new building location (SAIC, 2000).

Additional investigation of the three offshore areas (Zone 7, Zone 4 and Pier 1) is being undertaken to determine whether these areas pose an unacceptable ecological risk and therefore require evaluation in a Feasibility Study (FS). Initially, a Rapid Sediment Characterization (RSC) Pilot Study will be completed to support development of data quality objectives (DQOs) for a detailed validation/investigation study, if required. For Zone 7 and Zone 4, the RSC Pilot Study will include a screening level ecological risk assessment (SLERA) and Step 3a refinement of the risk assessment process. As part of the SLERA analytical chemistry results will be compared to conservative ecological threshold values to determine which constituents are carried forward through the assessment. This process is described in Figure 1. In Step 3a of the Navy risk assessment process, the frequency of exceedances of ecological thresholds, comparison to background levels, and bioavailability are considered to refine the exposure assessment. This process is outlined in Figure 2. For Pier 1, it is assumed that a baseline ecological risk assessment (BERA) will be required.

This work plan/field sampling plan (WP/FSP) addresses the field sampling activities planned to support the RSC Pilot Study. Attachments 1 and 2 contain field documentation forms and standard operating procedures (SOPs), respectively. The Quality Assurance Project Plan (QAPP) for the Pilot Study is provided as Appendix A, and the response to comments on the draft WP/FSP is provided as Appendix B. The Health and Safety Plan (HASP) is provided as a companion document.

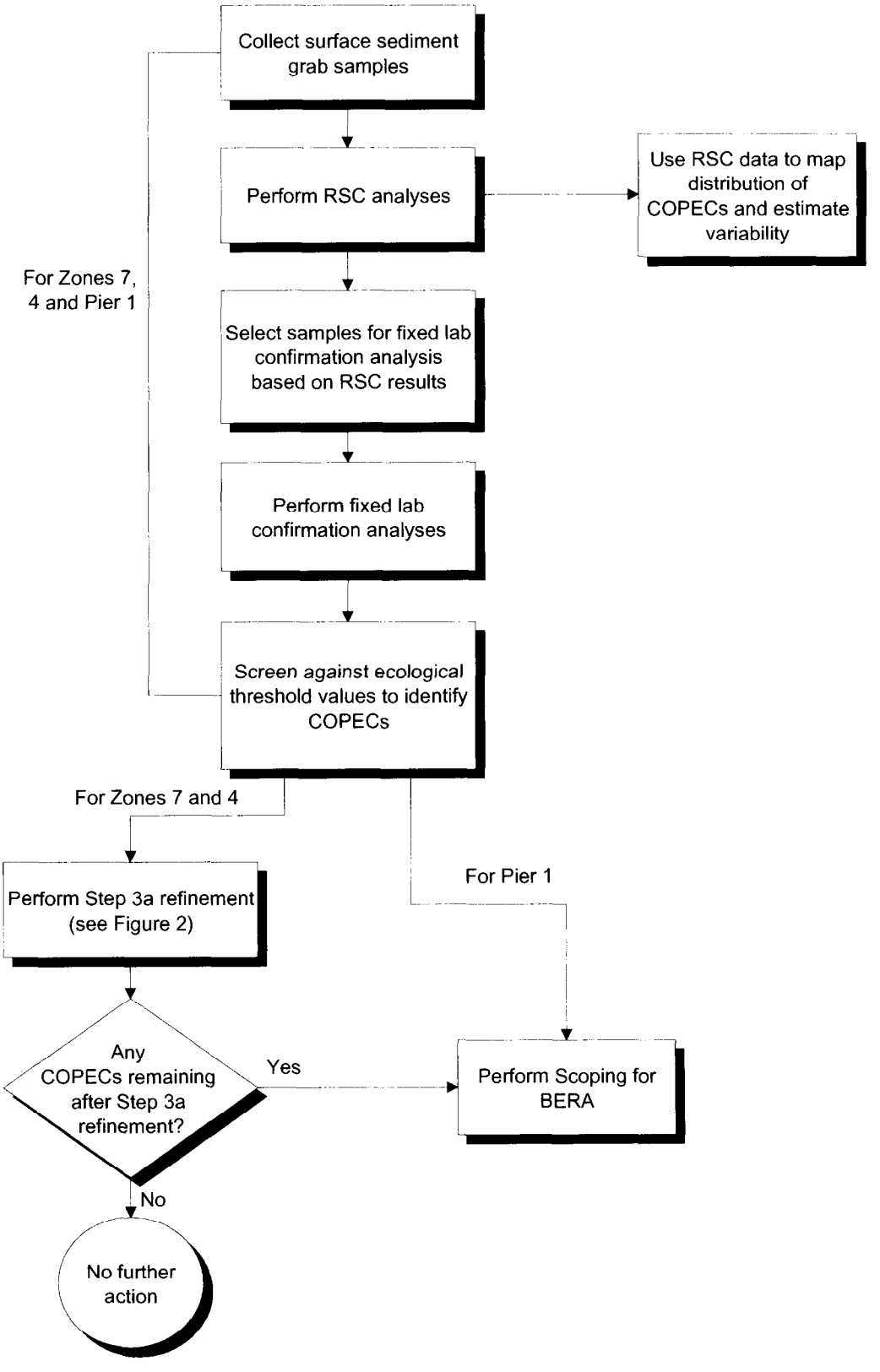

Figure 1. Overall Pilot Study Approach

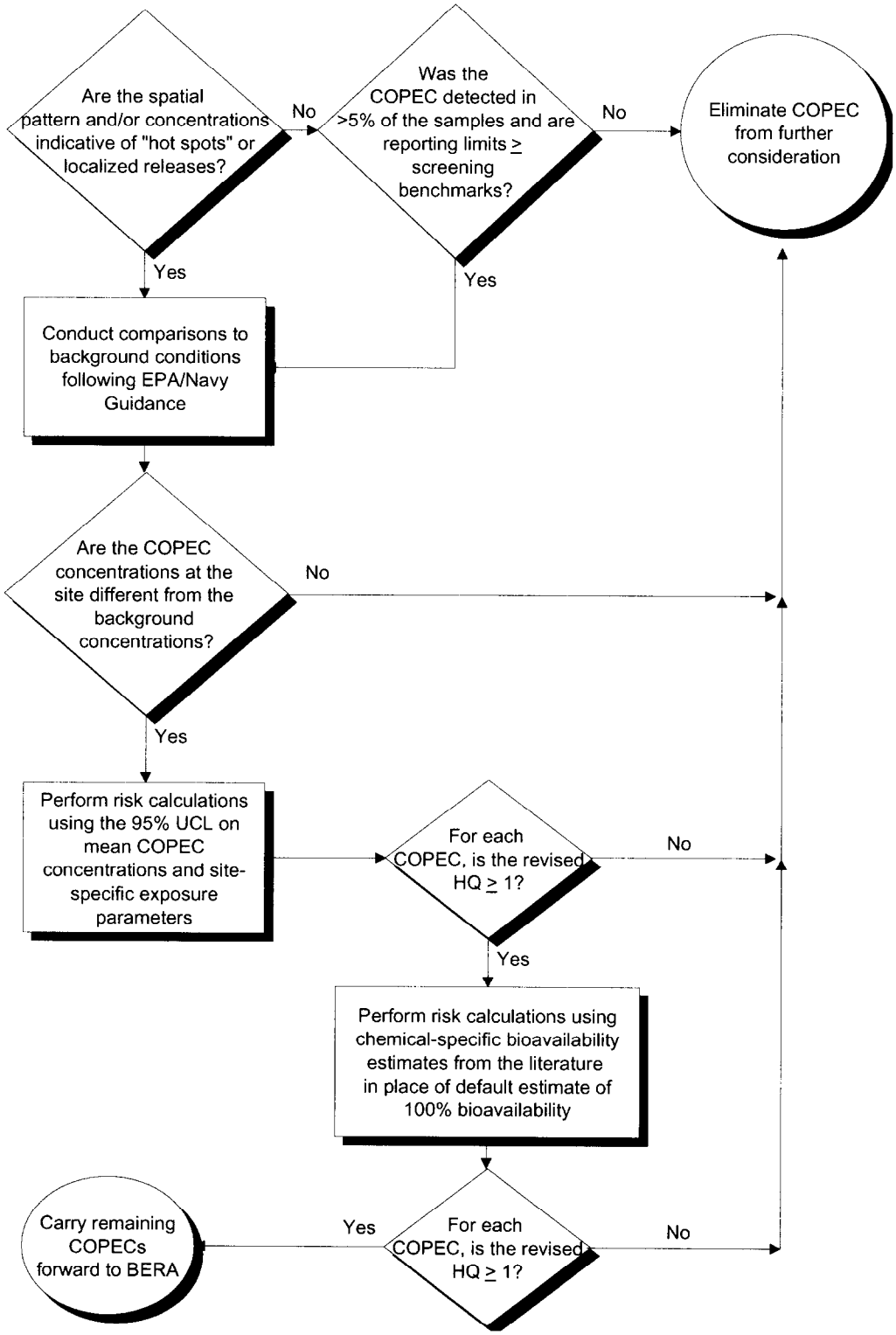

Figure 2. Step 3A Refinement Process

2.0 OBJECTIVES AND APPROACH

The objectives and approach for the Thames River RSC Pilot Study are presented in this section.

2.1 Objectives

The primary objective of this Pilot Study is to determine the current spatial distribution and variability of copper, lead, zinc, arsenic, chromium, total PCB, and total PAH concentrations in Zone 7, Zone 4, and Pier 1 surface sediments using RSC methods. The RSC data will be supplemented by fixed laboratory confirmation data for a subset of sediment samples. Specific objectives include the following:

- Define the nature and extent of contamination in Zone 7, Zone 4, and Pier 1 surface sediments using both RSC results, and fixed laboratory data for a subset of the samples.
- Determine if the dredging that occurred in Zone 7 in 1996 effectively removed some or all of the sediments that previously posed a potential ecological concern.
- In Zones 7 and 4, conduct a SLERA and Step 3a refinement using the fixed laboratory data to determine if sediments potentially pose an unacceptable ecological risk and therefore require further evaluation in a supplemental BERA.
- In Pier 1 area, determine the list of contaminants of potential ecological concern (COPECs) for further evaluation in a BERA.

The RSC Pilot Study data will be used to support the development of data quality objectives (DQOs) and a Work Plan for additional investigation of Pier 1 and a Validation Study of Zones 4 and 7, if required.

2.2 Approach

The RSC Pilot Study approach includes the following elements:

- Collect surface sediment grab samples and analyze using RSC methods to evaluate the nature and extent of contamination and variability in COPEC concentrations.
- Select a subset of sediment samples for fixed laboratory confirmation analysis.
- Using the fixed laboratory confirmation data, perform a SLERA and Step 3a refinement for Zones 7 and 4, and determine the list of COPECs for further evaluation in a BERA for Pier 1.

The data quality objectives (DQOs) for the RSC Pilot Study in Zone 7, Zone 4, and Pier 1 are presented in Tables 1 through 3, respectively. The overall approach is illustrated in Figure 1 and Figure 2 and is described further below. Details regarding field sample collection, including sample locations, are provided in Section 4.0.

Surface sediment samples from Zone 7, Zone 4, and Pier 1 will be analyzed using RSC methods developed by the U.S. Navy's Space and Naval Warfare Systems Center in San Diego, California (SSC SD). These methods provide a fast and accurate screening analysis for selected trace metals (copper, lead, zinc, arsenic and chromium) by X-ray fluorescence (XRF) methods, and total PAHs and total PCBs using immunoassay techniques. It is assumed that the high concentrations of copper, lead, zinc, arsenic and chromium will co-occur with high concentrations of other inorganic COPCs (e.g. nickel and mercury). This assumption was evaluated and validated through an analysis of historical data from Zone 7, Zone 4, and Pier 1 (see Appendix B). This assumption will be verified when fixed laboratory confirmation data are evaluated.

A subset of RSC samples will be analyzed for a full suite of chemical constituents and conventional parameters (metals, PAHs, pesticides, PCBs, total organic carbon [TOC] and grain size) at fixed laboratories. Confirmation samples will be selected based on two criteria: they will represent the three areas of interest (Zone 7, Zone 4, and Pier 1), and will span the range of measured RSC concentrations.

Samples will be ranked based on Effects Range – Median (ER-M) Quotients (ERM-Qs) calculated from the RSC results. Samples for confirmatory analysis will be selected to span the range of ERM-Qs. If either metals or organics are contributing to elevated ERM-Qs, then both types of samples will be included for confirmatory analysis. Finally, spatial representation will be considered in the selection of samples for confirmatory laboratory analysis. The confirmatory laboratory data will be used to verify the RSC results, develop correlation relationships, and also will be used in a SLERA and Step 3a refinement (Zones 7 and 4 only) to determine whether a supplemental BERA is warranted (Figures 1 and 2).

3.0 PROJECT MANAGEMENT

The project management structure for the Thames River RSC Pilot Study is presented in Figure 3. The QAPP in Appendix A defines the roles and responsibilities of each person in the organizational chart.

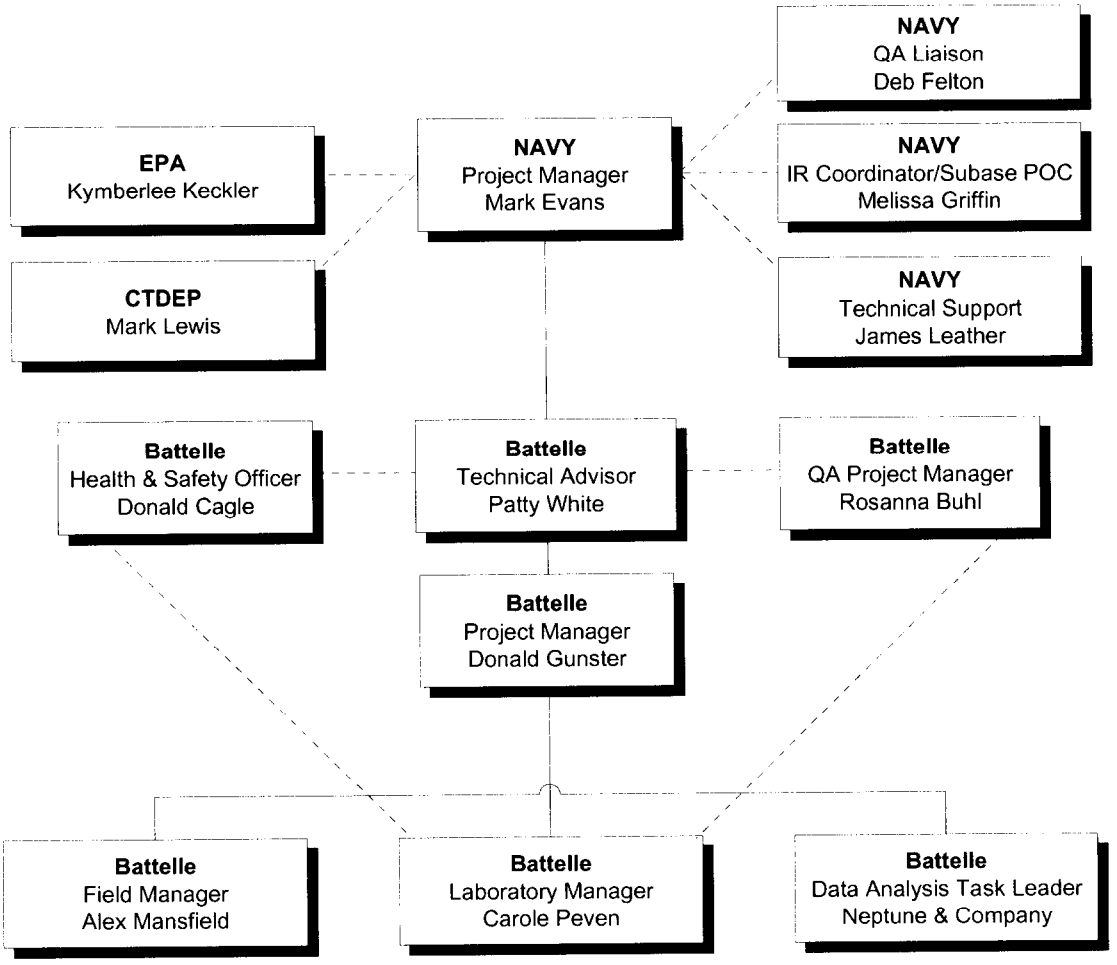

Figure 3. Project Management Structure

4.0 FIELD SAMPLING FOR RAPID SEDIMENT CHARACTERIZATION

4.1 Sample Collection Overview

A representative sample design was developed for the Zone 7, Zone 4, and Pier 1 study areas. A systematic grid was used because of the lack of information on post-dredging conditions in Zone 7, and the small number of historical data points in Zone 4. Grid spacing is smaller in areas that are closer to shore or existing piers, and larger in areas that are farther from potential sources. In the Pier 1 area, sample stations were located to supplement the 1999 SAIC data and help bound the extent of contamination above ambient levels.

Surface sediment samples will be collected from 31 stations in Zone 7, 15 stations in Zone 4, and 12 stations in Pier 1, for a total of 58 site samples. Sample location maps are provided in Figures 4 and 5. Four sediment grab samples will be collected from each of two reference areas for a total of eight reference samples. All reference site samples will be submitted for fixed laboratory analysis; therefore, RSC data for these samples will not be collected. The general location of two upstream reference stations sampled for the Lower Subase Phase II RI (Stations T1SD1 and T1SD2) will be utilized for the RSC Pilot Study. Samples from the reference stations will be collected along two transects at similar distances from shore as the sample stations in Zones 4 and 7 to maximize data comparability. Reference area sample locations are shown in Figure 6.

Samples will be collected from the 24-ft survey vessel R/V Will Do, which is owned and operated by Ocean Survey, Inc. (OSI). The field crew will consist of the boat operator, one Battelle staff member, and one SSC SD staff member. All fieldwork will be coordinated with the Engineering Field Activity Northeast (EFANE) Remedial Project Manager (RPM), Mr. Mark Evans, and the NSB-NLON Environmental Point-of-Contact (POC), Ms. Melissa Griffin, to obtain a sampling schedule and proper security clearances. The field work is expected to require 3 days to complete.

Sediment collected at each station will be homogenized and divided into three containers. One container will be sent to SSC SD for analysis by RSC methods and the other two containers will be sent to Battelle Duxbury Operations (BDO) for storage pending potential confirmatory analysis. The RSC methods to be employed include a modified EPA Method 4020 for PCBs, a modified EPA Method 4035 for PAHs, and a modified EPA SW846 Method 6200 for XRF metals. These methods and associated detection limits are specified in Table A-2 of the Pilot Study QAPP (Appendix A) Other supporting information for the RSC methods is provided in the Pilot Study QAPP tables as follows: Table A-5 defines the sample size and handling requirements and Table A-8 defines the measurement quality criteria. Care will be taken to follow EPA Region 1 Immunoassay Guidelines, to ensure that samples are properly evaluated (EPA, 1996)

A subset of the samples sent to BDO will be analyzed for a full suite of chemical constituents (metals, PAHs, pesticides, PCBs, TOC, and grain size) to provide confirmatory data for the RSC analyses and input data for a screening-level risk assessment. The confirmatory samples will be selected based on the RSC results according to the criteria specified in the DQOs (Tables 1 through 3). The confirmatory samples will span the range of observed RSC concentrations so that a correlation relationship between RSC and fixed laboratory results can be developed. All RSC and confirmatory laboratory data will be evaluated and reported in the Validation/Investigation Study Work Plan. The screening and refinement level risk assessments for Zones 4 and 7 will be included in the Work Plan. Details of the field sample collection effort are provided below.

4.1.1 Field Measurements

Navigation coordinates and water depth will be recorded on the sediment sampling data form provided in Attachment 1 for each sampling station. Station coordinates will be located using OSI's Trimble 4000 RS differential Global Positioning System (DGPS) and recorded during sampling. This DGPS unit has an accuracy of ±2 meters. Coordinates will be recorded in latitude and longitude decimal degrees (NAD83). Prior to the survey, the DGPS unit will be inspected and tested. The DGPS will be checked against an established horizontal benchmark at the beginning of the survey prior to any sample collection. The calibration of the DGPS unit will be checked by the field team prior to each day of sampling using a reference location identified by the Field Team Leader. The location of the reference point will be documented in the field log. Potential benchmarks for DGPS calibration are listed in a table in Attachment 1. All DGPS calibrations will be documented in the field logbook on a DGPS calibration form (Attachment 1). If the DGPS fails to attain a reading that is within 10 meters of the actual position, then the manual should be consulted for sources of error and the reference position verified.

All field measurements, including field location, type of vessel or equipment, and weather will be recorded on the Daily Log Form provided in Attachment 1, and maintained in a paginated and bound field logbook. This Daily Log Form will be used each day for each field location sampled that day. Water depth will be measured using a calibrated lead line. Water depths will be measured to the nearest tenth of a meter (10 cm) and recorded in a paginated and bound field logbook (Attachment 1).

4.1.2 Surface Sediment Collection

Surface sediment samples will be collected from the biotic zone, which is expected to be the upper 5 cm of each grab sample. Samples will be collected using a Ponar or Van Veen grab sampler deployed from a 24-ft survey vessel. One sample will be collected at each station. Sediment will be collected from full or partially full samplers. Approximately 1000 g of sediment are required from each station for RSC and laboratory analysis. Measurements and information pertaining to sediment collection will be recorded on the sediment sampling data form, provided in Attachment 1.

For each sample, the boat operator will maneuver the vessel to within 10 ft of the defined sample location. The boat will be anchored and the grab sampler will be deployed. Each grab will be examined for acceptability (SOP 5-169) before the top 5 cm are removed as described in Section 4.3. Multiple grabs will be necessary to obtain 1000 g of sediment. The number of acceptable vs. unacceptable grabs will be documented on the survey log forms. The SOPs provided in Attachment 2 describe the methods used to collect and handle sediment samples. If no successful grabs are obtained after three attempts to sample a station, then the station will be abandoned, the boat moved slightly, and re-sampling initiated.

Proposed sample locations shown in Figures 4 and 5 cannot be confirmed as available for sampling until the day of sampling. If a sample location must be abandoned due to access limitations, then the Field Team Leader will sample the closest reasonable alternative location. For example, in the event that submarines are moored at piers during fieldwork, sampling stations will be relocated to the nearest available location without compromising NSB-NLON security requirements. The Field Team Leader will coordinate the sampling program with the NSB-NLON POC at least two weeks prior to the initiation of sampling to determine the accessibility of all proposed sampling locations.

The presence of ammonia in porewater will be evaluated at each station in the field using Hach™ test strips. This information will be used to assist in planning for potentially conducting future bioassay tests. Salinity of overlying water will also be measured.

Sample locations in Zone 7, Zone 4, and Pier 1 are shown in Figures 4 and 5. Reference site sample locations are shown in Figure 6. The reference sites are located approximately 0.5 mile upstream from the northern NSB-NLON boundary, between the NSB-NLON and the Dow Chemical Company plant. These reference locations were sampled for the Lower Subase RI (Brown and Root, 1999).

Table 4 provides a list of the sample locations and coordinates. The OSI Trimble 4000 RS DGPS is interfaced with an OSI Maretrack Navigation and Data Logging System. Each grab will be marked in the navigation system to maintain a record of sample coordinates. Unless the differences between grabs at any one station exceed 10 feet, the coordinates of the first successful grab will be used as the coordinates for the station. If individual grab coordinates vary by more than 10 feet, then an average of all grab coordinates will be calculated during data reduction and used as the station location.

4.1.3 Quality Control Samples

Field duplicate samples will be collected from 10 percent of the Pilot Study stations (i.e., seven field duplicates). Two field duplicates will be collected from Pier 1, two from Zone 4, and three from Zone 7. Field duplicate locations are identified in Table 4. In addition, laboratory replicates will be performed on each batch of samples for both the RSC and fixed laboratory analyses. Acceptance criteria for the laboratory replicates are presented in Table A-8 in the QAPP. A complete sample summary is provided in Table 5.

4.2 Equipment Decontamination

Prior to the initiation of the field survey, the grab sampler and all homogenizing equipment will be cleaned by washing with soap and water, follow with a solvent rinse of acetone and then dichloromethane. During the field survey, cross-contamination of surface sediment samples will be avoided by collecting only sample material that is not in direct contact with sample collection equipment, and by cleaning equipment between sampling stations. The grab sampler will be scrubbed with a stiff brush and thoroughly rinsed with site water at the beginning of each day and between each sampling station. Utensils such as kynar scoops, stainless steel bowls, and kynar-coated spoons will be used to collect and homogenize sediment from the samplers (kynar is a chemical-resistant commercial resin). These utensils will be scrubbed with a stiff brush and thoroughly rinsed with site water between each sampling station. These decontamination procedures do not include the use of solvent as specified in SOP 5-169-02, Section 3.2 (Attachment 2). The procedures defined in this WP/FSP represent project-specific modifications. The field methods are designed to eliminate the use of organic solvents in the field by minimizing potential carry-over between stations and contamination from sampling equipment by collecting only sediment that has not contacted the walls of the sampling equipment and using pre-cleaned equipment to collect samples from the grabs.

4.3 Sample Processing and Handling

Approximately 1000 g of sediment are required from each station for RSC and fixed laboratory analysis. RSC samples will not be collected from reference stations because all reference samples will be submitted for fixed laboratory analysis. The upper 5 cm of sediment will be removed from the sampler with kynar-coated scoops that are 5 cm deep. At each station, sediment from all acceptable grabs will be placed in a clean stainless steel bowl. Excessive water should be drained from the sample prior to homogenization to achieve a homogeneous sample. The sediment will be thoroughly mixed in the bowl using a clean spoon until a homogenous color and texture is achieved. The homogenized sediment from each station will be split into three labeled sample jars: one 4-oz glass jar and two 16-oz glass jars, all with Teflon-lined caps.

Particular attention must be paid to water content: sediment should be mixed during this aliquot process to ensure that the water content of all samples is equivalent. Water cannot be decanted once the aliquot process has begun.

The 4-oz jar will be shipped or hand-carried to the SSC SD lab in San Diego for RSC analysis by SSC SD staff. The two 16-oz jars will be shipped to BDO for archival and selected confirmational laboratory analysis. Prior to shipment, sample jars will be stored in a cooler with ice.

During sample collection, field personnel will wear polyethylene or latex gloves as liners under Nitrile® gloves. During sample handling, they will wear polyethylene or latex gloves. Gloves should be changed between stations. Gasoline or diesel motors (boat motors and gas-powered pumps) should be shut down at least 1 minute prior to sampling activities to eliminate the potential for contamination by exhaust gasses.

4.4 Containers and Labeling

Sample containers will be certified as clean, the container lot number will be noted on the custody forms for the samples for tracking purposes, and the certificate of analysis retained.

Sample containers for collecting the sediment samples will be one 4-oz glass jar with a Teflon-lined cap for RSC analysis and two 16-oz glass jars with Teflon-lined caps for potential laboratory analysis or archiving. Each jar will be labeled with waterproof, adhesive-back tape or labels and waterproof ink. Sample labels will provide sufficient detail to uniquely identify each sediment sample. An example is provided below. Sample labels for each station must include a unique sample identification number, sampling area, collection date and time, and sample collector's name.

Survey ID: Thames River RSC Pilot Study 2003
Area (circle one):
Zone 7, Zone 4, Pier I, Ref Area 1, Ref Area 2
Unique Sample ID: CAB-_____
Sample Description (circle one):
RSC, or Archive/Confirmation
Date: _____ Time: _____
Sample Collector:_____
Container ___ of 3

The unique sample ID at each station will include the sample ID and the container number, allowing the data from each sample to be easily tracked to the station. The custody forms will include the jar numbers for tracking purposes. Jar 1 will always be the 4-oz jar for RSC. Jars 2 and 3 will always be the samples targeted for archival and selected confirmation.

4.5 Sample Preservation, Packaging, and Shipment

Samples will be packaged and shipped in accordance with the procedures in the Battelle Duxbury Laboratory SOP 5-210, *Packaging and Shipping of Samples* (Attachment 2). The sediment samples will be preserved by placing each sample container into a cooler filled with ice immediately upon collection and after processing. However, care will be taken to ensure that the samples do not freeze. During field operations, the temperature of the coolers will be monitored and documented in the field log. The intended analyses, storage conditions, and holding times are defined in Table A-5of the QAPP. Samples will either be hand-carried to the analytical laboratory by the field participants, or shipped by overnight carrier. If shipped, the sample custodian should be notified of the number of coolers shipped on the day

that coolers are shipped to allow for early corrective action if samples do not arrive as expected. Sediment samples will be shipped as follows:

The 4-oz glass sediment jar targeted for RSC analysis will be shipped to:

Mr. Jim Leather
SPAWAR Systems Center D362
53475 Strothe Road
San Diego, CA 92152
(619) 553-6240

Attention: Jim Leather

The 16-oz jars for archiving or confirmatory analysis will be shipped to the BDO sample custodian:

Ms. Jessica Fahey (Custodian)
Battelle Duxbury Operations
397 Washington St.
Duxbury, MA 02332
(781) 952-5270

Attention: Carole Peven

The samples selected for analysis will be rehomogenized, split by laboratory staff at BDO, and distributed to the appropriate laboratories. Details regarding the fixed laboratory analyses are provided in the QAPP.

4.6 Sample Custody

Sample custody will be maintained and documented throughout collection, shipping, analysis, and disposal of the sample. Samples will not be left unattended unless properly secured. Sample custody procedures will be in accordance with the Battelle Duxbury Operations SOP 6-010, *Sample Receipt, Custody, and Handling*. The sample custody form (Attachment 2) provides a record of the samples collected and analyses requested. The custody forms completed for RSC will include the jar number (Jar 1) as part of the sample description. Likewise, the custody forms completed for archival and potential confirmatory analysis will include the jar numbers (Jar 2 and 3) as part of the description.

If more than one cooler is sent in one shipment to the laboratory, then each cooler will contain a separate custody record for the samples in that cooler. In addition, the outside of the coolers will be marked to indicate the number of coolers in the shipment (*e.g.*, 1 of 2, 2 of 2).

4.7 Sample Documentation

Sample collection information, compiled at each sampling location, will be hand-recorded on paginated data forms in a bound field logbook. Field logbook forms are provided in Attachment 1. Field samples will be labeled by field personnel as described in Section 4.4. All sample collection forms will be completed using indelible ink, and documentation errors will be corrected by drawing a single line through the error, making the correction, and initialing, dating, and justifying the error.

4.8 Management and Disposal of Investigation-Derived Waste

Field sampling and sample preparation activities will be conducted such as to minimize generation of waste materials. All solid waste will be bagged or otherwise contained prior to disposal in standard refuse containers (dumpsters). In the field, sediment and rinse water from sampling equipment will be washed overboard. It is not anticipated that flammable or solvent waste will be generated in the field; any solvent waste generated in the laboratory will be contained in appropriately labeled containers and disposed of in compliance with state and federal waste handling regulations. Wastewater generated during sample preparation of sediment samples at the BDO, will be managed in compliance with a project-specific wastewater treatment plan as required by that facility. Field samples will be held for six months after delivery of final data. Sediment analytical results can be used to determine whether or not excess sediment requires management as hazardous waste.

5.0 REFERENCES

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SAIC. 2000. *Evaluation of Chemical Data for the Pier 1 Marine Railway, Naval Submarine Base New London, Groton, CT.* Prepared for Naval Facilities Engineering Command, Northern Division. Lester, PA.

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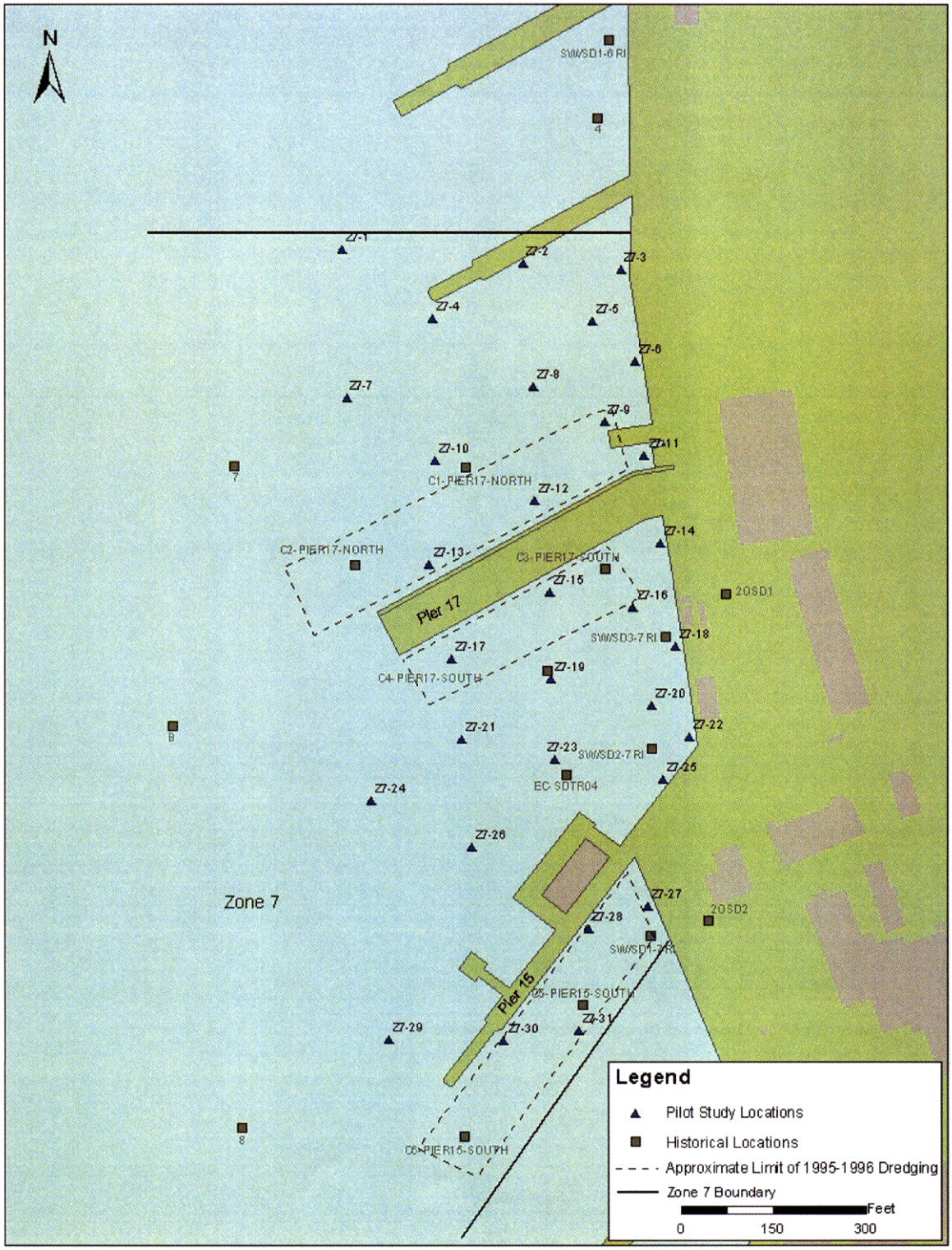

Figure 4. RSC Pilot Study Sampling Locations (Zone 7)

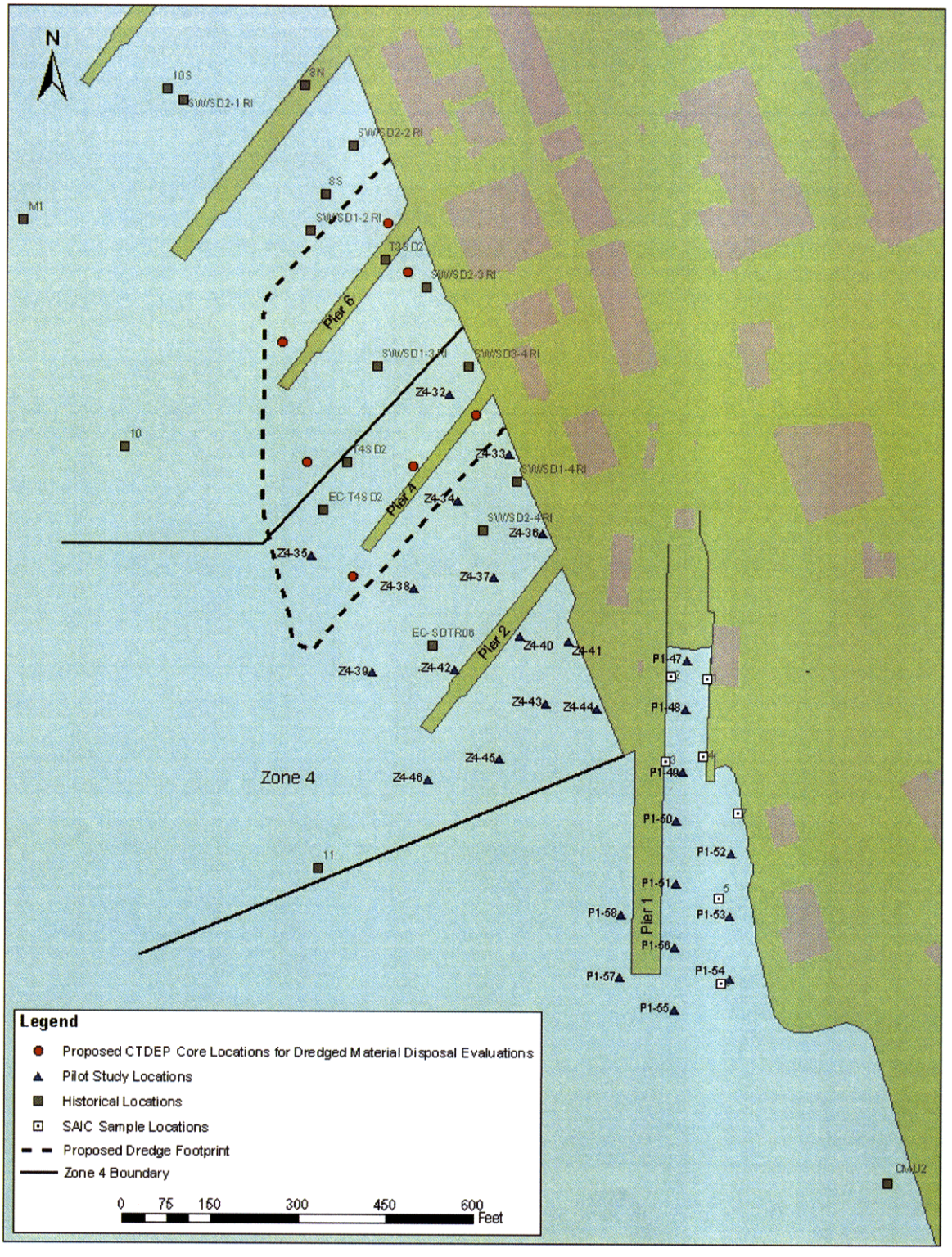

Figure 5. RSC Pilot Study Sampling Locations (Zone 4 and Pier 1)

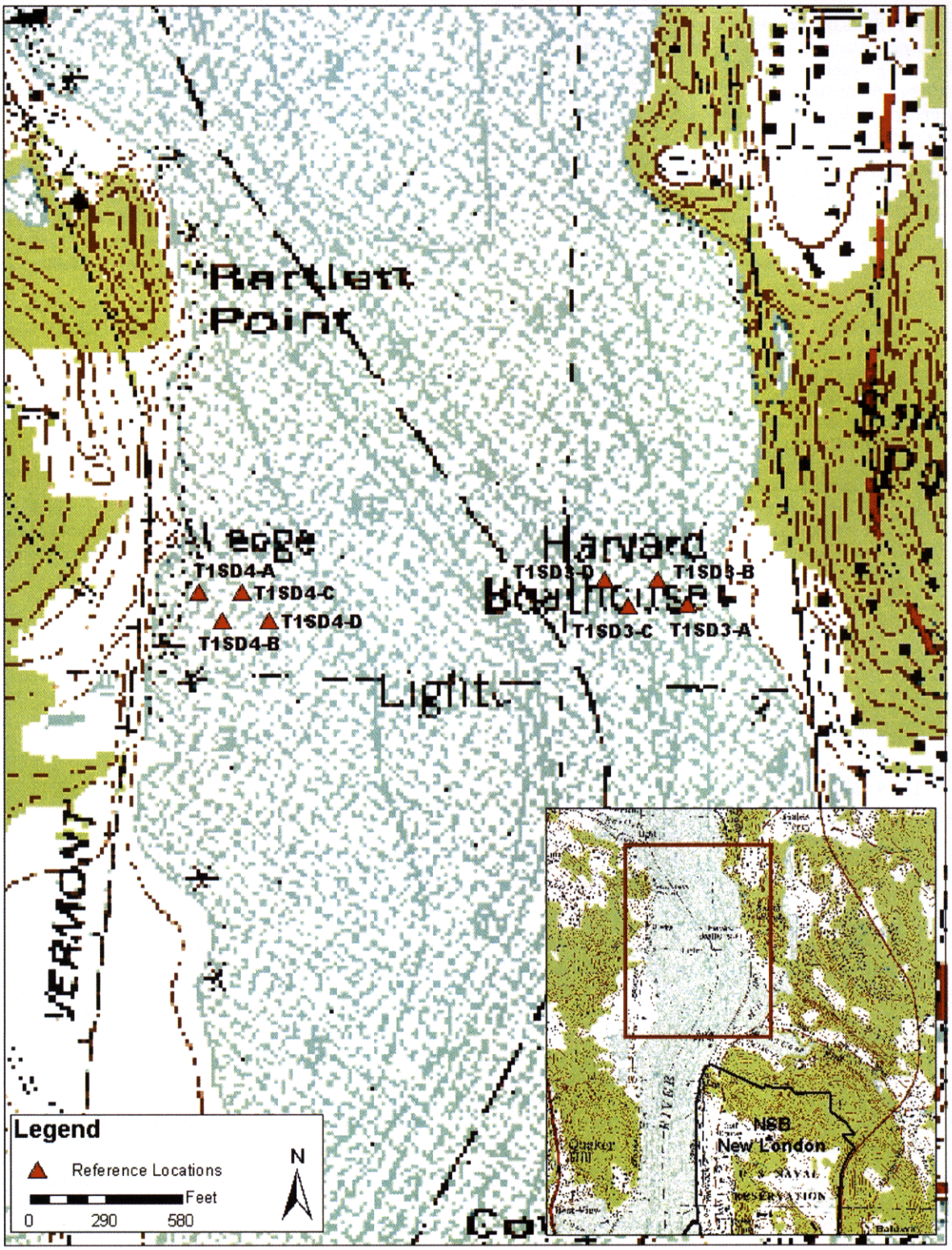

Figure 6. RSC Pilot Study Reference Station Sample Locations

Table 1. Data Quality Objectives for Zone 7

RSC PILOT STUDY FOR ZONE 7, THAMES RIVER

STEP 1: State the Problem

The area offshore of Zone 7 has been the subject of several historical data collection efforts, most recently the analysis of three samples collected in 1997 as part of a RI. The Lower Subase RI concluded that sediments in Zone 7 posed a low to moderate potential ecological risk (Brown and Root, 1999). The RI recommended the collection of additional data to better define the nature and extent of contamination, and evaluate the potential ecological risk associated with sediments in this area.

A substantial portion of Zone 7 was dredged in 1996 during the replacement of Pier 17 and dredging adjacent to Pier 15. One purpose of the Pilot Study is to determine whether the dredging effectively removed some or all of the sediments of potential ecological concern. Historical data from Zone 7 indicate the following:

- Three surface sediment grab samples (0 – 0.5 ft) taken as part of the RI found a few metals (As, Ba, Cr, Fe, Hg) and benzo(a)pyrene at levels exceeding background concentrations, but well below Effects Range – Median (ER-M) values (Long et al., 1995).
- Several metals were measured in sediment samples collected as part of the 1994 SEAWOLF sampling program (Maguire Group Inc., 1995), where composite samples from cores collected in the vicinity of Pier 17 had concentrations of Cu, Pb, and Zn above ER-Ms.
- Percent survival results in *Ampelisca* bioassays performed on two composite sediment samples from the top 3 ft of sediment as part of the Pier 17 replacement Environmental Assessment (EA) (Maguire Group Inc., 1994) were not significantly lower than results for reference sites. *Ampelisca* and *Leptochirus* toxicity bioassays were also performed on one sample as part of the Phase II RI. *Ampelisca* survival was significantly lower than reference; however, the RI does not provide the detailed results, or an evaluation of possible confounding factors. All locations where biological tests were conducted have since been dredged.
- Bioaccumulation of PAHs in clams and worms, and PCBs in worms exposed to sediment collected around Pier 17 was found to occur at levels above reference during the Pier 17 EA. These constituents did not bioaccumulate in caged mussels from a nearby location. Sediments that were tested for bioaccumulation have since been dredged.

STEP 2: Identify the Decision(s)

1. What are the spatial distribution and variability of Cu, Pb, Zn, As, Cr, total PCBs and total PAHs in Zone 7 surface sediments as measured by rapid sediment characterization (RSC) methods?
2. Are COPEC concentrations as measured by fixed laboratory methods below ecological screening threshold values?
3. Do the results of a SLERA and Step 3a refinement using fixed laboratory data and site-specific exposure parameters indicate that sediments potentially pose an unacceptable ecological risk?
4. Are COPECs that are found to pose a potential ecological risk based on the SLERA and Step 3a refinement at concentrations significantly greater than reference area concentrations?

Subordinate Objectives:

1. Do the fixed laboratory confirmation data correlate with the RSC results?
2. Do the fixed laboratory data confirm that the chemical constituents measured by RSC methods co-occur with Hg and Ni measured in the fixed laboratory confirmation samples?
3. Do sediment characteristics (i.e., grain size and organic carbon content) potentially influence the RSC results?
4. Are there porewater or sediment characteristics that might influence the results of potential future toxicity tests (i.e., ammonia, salinity, TOC, grain size)?

STEP 3: Identify Inputs to the Decision(s)

1. Concentrations of Cu, Pb, Zn, As, and Cr (using XRF measurement methods), and total PAHs and PCBs (using immunoassay test methods) representative of Zone 7.
2. Fixed laboratory confirmation data (using NOAA Status and Trends analytical methods) for a subset (approximately 50%) of Zone 7 samples and all reference site samples. Confirmatory samples will be selected based on RSC results and will span the range of RSC concentrations to provide sufficient data to support the development of correlation relationships. Samples will also be selected to provide a reasonable spatial representation of Zone 7 sediments to provide input data for the SLERA and Step 3a refinement.
3. Ecological screening threshold values and exposure parameters and methodologies.[1]
4. Estimates of ammonia concentrations and salinity in porewater, and grain size and TOC data for confirmatory sediment samples.

[1]A project team meeting is proposed during the Pilot Study data collection effort to complete the scoping effort for the ecological risk assessment. Topics of discussion will include the proposed conceptual site model, identification of complete exposure pathways and assessment endpoints, and identification of surrogate receptors and associated exposure parameters for the evaluation of food chain risk.

Table 1. Data Quality Objectives for Zone 7, continued

STEP 4: Define the Study Boundaries

The north and south boundaries of Zone 7 will be the same as those defined in the Lower Subase RI. The western boundary is the Thames River navigational channel, and the eastern boundary is the paved and cantilevered shoreline area. The areas underneath piers and the cantilevered shoreline will not be sampled. The top 5 cm of sediment from each station will be collected.

STEP 5: Develop a Decision Rule

The RSC data will be used to 1) verify the post-dredging concentration and distribution of selected chemicals in Zone 7 surface sediments; 2) provide an estimate of spatial variability; and 3) support, if necessary, the design of a focused sampling plan involving multiple lines of evidence for a supplemental BERA.

Confirmatory laboratory data will be used to 1) determine the degree to which RSC results correlate with fixed laboratory results (at a 95 percent confidence level); 2) identify COPECs by performing an initial screen using conservative ecological screening benchmarks; 3) conduct a SLERA and Step 3a refinement using site-specific exposure parameters to identify likely risk drivers, and 4) determine if any risk drivers identified in Zone 7 sediments occur in concentrations that exceed reference area concentrations.[2]

Field data on porewater salinity and ammonia concentrations, in conjunction with sediment grain size and TOC results, will be used to identify potential confounding factors that may need to be managed during potential future toxicity tests conducted as part of a more detailed validation/investigation study.

[2]The decision logic and procedures to be used to support each of these objectives will be presented to the project team for discussion and concurrence prior to data analysis.

STEP 6: Evaluate Decision Errors

To support the SLERA and Step 3a refinement, qualitative goals were used to design the Pilot Study. Representative sample of the area will be obtained by utilizing a systematic grid sampling. Conservative methods will be employed to ensure that a problem is not overlooked. The conservative approach includes biasing the selection of samples for confirmatory laboratory analysis towards those with elevated RSC results (see Step 7), and the use of a conservative decision rule that uses the maximum observed concentration compared to conservative screening benchmarks to identify COPCs. By employing these two techniques, the probability of incorrectly determining no unacceptable potential ecological risk (false negative) will be limited.

To support the comparison to reference areas that will be used in the Step 3a refinement, a quantitative set of error constraints was developed. A false positive error would result in a determination that a significant difference exists between Zone 7 and the reference area, when in truth the concentration distributions are not different. Four tests will be employed to evaluate differences in distributions. A false positive limit of 0.10 (10%) will be used. A false negative error would result in failing to conclude that a significant difference in concentration distributions exist, when in truth a meaningful difference exists. Specifications regarding both the acceptable probability of a false negative and the size of a distribution shift that is meaningful are as follows. Based on the historically observed variability, it is expected that shifts of between 1.5 and 2 times the reference area values will be observable, with good power (between 75% and 95% of the time). Accordingly, the false negative error rate at a delta of 1.5 is set at 25%, and for a delta of 2 it is set at 10%.

STEP 7: Optimize the Design for Obtaining Data

Because much of Zone 7 was dredged in 1996, little is known about the current distribution and variability of chemical concentrations in sediment. Therefore, a systematic sample approach (i.e., triangular grid) based on a pre-determined number of samples (31) will be used to provide representation of the entire zone. Grid spacing is closer near the shoreline to detect concentration gradients originating from potential onshore sources if they exist. In the reference areas, samples will be collected from eight locations using a triangular grid along a transect perpendicular to the shoreline with a grid spacing approximating that used in Zones 4 and 7.

Samples for fixed laboratory analysis will be selected based on two criteria: they will represent spatial areas of interest, and will span the range of measured RSC concentrations. To achieve the desired error limits for the comparison to reference specified in Step 6, a minimum of 8 samples from the reference area, and 8 or more from Zone 7 are required Fifteen (15) confirmation samples will be collected from Zone 7 as follows: 8-10 samples will represent locations with the highest RSC concentrations, 3-5 will represent locations with medium concentrations, and 2-4 will represent locations with low concentrations. This approach will place greater emphasis on areas that appear to be of greatest concern based on RSC results, while ensuring that low-level samples do not contain elevated levels of COPECs that were not measured by RSC methods.

Table 2. Data Quality Objectives for Zone 4

RSC PILOT STUDY FOR ZONE 4, THAMES RIVER

STEP 1: State the Problem

The area offshore of Zone 4 has been subjected to minimal data collection comprising a total of 5 locations: three surface sediment samples collected in 1997 as part of a RI (Brown and Root, 1999), one surface sample in 1995 to support the SEAWOLF homeporting EIS (Maguire Group Inc., 1995), and one 0 – 5.1 ft core sample collected in 1990. The RI concluded that Zone 4 sediments posed a low to moderate potential risk, and that further data collection would be required to evaluate potential ecological risks associated with sediment is this area. An evaluation of the historical data indicates the following:

- Benzo(a)pyrene, Cr, Cu, Pb, Zn, and Ni were measured at concentrations exceeding ER-Ms; and As, Se and Hg exceeded Effects Range – Low (ER-L) values (Long et al., 1995).
- No toxicity to *Ampelisca* or *Leptochirus* was observed in the one Phase II RI sediment sample that was tested.
- Potential bioaccumulation is only represented by tissue data from caged mussels deployed at a site south of Zone 4. Benzo(a)pyrene and Cr were not detected in the tissues; PAHs and pesticides were detected at low levels.

Dredging is planned in the northern part of Zone 4 in 2005 to support a pier redevelopment project.

STEP 2: Identify the Decision(s)

1. What are the spatial distribution and variability of Cu, Pb, Zn, Cr, As, total PCBs and total PAHs in Zone 4 surface sediments as measured by rapid sediment characterization (RSC) methods?
2. Are COPEC concentrations as measured by fixed laboratory methods below ecological screening threshold values?
3. Do the results of a SLERA and Step 3a refinement using fixed laboratory data and site-specific exposure parameters indicate that sediments potentially pose an unacceptable ecological risk?
4. Are COPECs that are found to pose a potential ecological risk based on the SLERA and Step 3a refinement present at concentrations significantly greater than reference area concentrations?

Subordinate Objectives:

1. Do the fixed laboratory confirmation data correlate with the RSC results?
2. Do the fixed laboratory data confirm that the chemical constituents measured by RSC methods co-occur with Hg and Ni measured in the fixed laboratory confirmation samples?
3. Do sediment characteristics (i.e., grain size and organic carbon content) potentially influence the RSC results?
4. Are there porewater or sediment characteristics that might influence the results of potential future toxicity tests (i.e., ammonia, salinity, TOC, grain size)?

STEP 3: Identify Inputs to the Decision(s)

1. Concentrations of Cu, Pb, Zn, As and Cr (using XRF measurement methods), and total PAHs and PCBs (using immunoassay test methods) representative of Zone 4 sediments.
2. Fixed laboratory confirmation data (using NOAA Status and Trends analytical methods) for a subset of Zone 4 samples and all reference site samples. Confirmatory samples will be selected based on RSC results and will span the range of RSC concentrations to provide sufficient data to support the development of correlation relationships. Samples will be selected to provide a reasonable spatial representation of Zone 4 sediments to provide input data for the SLERA and Step 3a refinement.
3. Ecological screening threshold values and exposure parameters and methodologies.[1]
4. Estimates of ammonia concentrations and salinity in porewater, and grain size and TOC data for confirmatory sediment samples.

[1]A project team meeting is proposed during the Pilot Study data collection effort to complete the scoping effort for the ecological risk assessment. Topics of discussion will include the proposed conceptual site model, identification of complete exposure pathways and assessment endpoints, and identification of surrogate receptors and associated exposure parameters for the evaluation of food chain risk

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STEP 4: Define the Study Boundaries

The north and south boundaries of Zone 4 will be the same as those defined in the Lower Subase RI. The western boundary is the Thames River navigational channel, and the eastern boundary is the paved and cantilevered shoreline area. The areas underneath piers and the cantilevered shoreline will not be sampled. The top 5 cm of sediment from each station will be collected.

Table 2. Data Quality Objectives for Zone 4, continued

STEP 5: Develop a Decision Rule
The RSC data will be used to: 1) verify the concentration and distribution of selected chemicals in Zone 4 surface sediments; 2) provide an estimate of spatial variability; and 3) support, if necessary, the design of a focused sampling plan involving multiple lines of evidence for a supplemental BERA.

Confirmatory laboratory data will be used to: 1) determine the degree to which RSC results correlate with fixed laboratory results (at a 95 percent confidence level); 2) identify COPECs by performing an initial screen using conservative ecological screening benchmarks; 3) conduct a SLERA and Step 3a refinement using site-specific exposure parameters to identify likely risk drivers, and 4) determine if any risk drivers identified in Zone 4 sediments occur in concentrations that exceed reference area concentrations.[2]

Field data on porewater salinity and ammonia concentrations, in conjunction with sediment grain size and TOC results, will be used to identify potential confounding factors that may need to be managed during potential future toxicity tests conducted as part of a more detailed validation/investigation study.

[2]The decision logic and procedures to be used to support each of these objectives will be presented to the project team for discussion and concurrence prior to data analysis.

STEP 6: Evaluate Decision Errors
To support the SLERA and Step 3a refinement, qualitative goals were used to design the Pilot Study. Representative sample of the area will be obtained by utilizing a systematic grid sampling. Conservative methods will be employed to ensure that a problem is not overlooked. The conservative approach includes biasing the selection of samples for confirmatory laboratory analysis towards those with elevated RSC results (see Step 7), and the use of a conservative decision rule that uses the maximum observed concentration compared to conservative screening benchmarks to identify COPCs. By employing these two techniques, the probability of incorrectly determining no unacceptable potential ecological risk (false negative) will be limited.

To support the comparison to reference areas that will be used in the Step 3a refinement, a quantitative set of error constraints was developed. A false positive error would result in a determination that a significant difference exists between Zone 4 and the reference area, when in truth the concentration distributions are not different. Four tests will be employed to evaluate differences in distributions. A false positive limit of 0.10 (10%) will be used. A false negative error would result in failing to conclude that a significant difference in concentration distributions exist, when in truth a meaningful difference exists. Specifications regarding both the acceptable probability of a false negative and the size of a distribution shift that is meaningful are as follows. Based on the historically observed variability, it is expected that shifts of between 1.5 and 2 times the reference area values will be observable, with good power (between 75% and 95% of the time). Accordingly, the false negative error rate at a delta of 1.5 is set at 25%, and for a delta of 2 it is set at 10%.

STEP 7: Optimize the Design for Obtaining Data
Little is known about the current distribution and variability of chemical concentrations in sediment in Zone 4 because of the small number of historical sample locations. Therefore, a systematic sample approach (i.e., triangular grid) based on a pre-determined number of samples (15) will be used to provide representation of the entire zone. Grid spacing is closer near the shoreline to detect concentration gradients originating from potential onshore sources if they exist. Fewer samples are located within the proposed dredge envelope for the planned Pier 6 reconstruction project because these sediments will undergo a dredged material disposal evaluation under a separate program. In the reference areas, samples will be collected from eight locations using a triangular grid along a transect perpendicular to the shoreline with a grid spacing approximating that used in Zones 4 and 7.

Samples for fixed laboratory analysis will be selected based on two criteria: they will represent spatial areas of interest, and will span the range of measured RSC concentrations. To achieve the desired error limits for the comparison to reference specified in Step 6, a minimum of 8 samples from the reference area, and 8 or more from Zone 4 are required. Eleven (11) confirmation samples will be collected from Zone 4 as follows: 7 samples will represent locations with the highest RSC concentrations, 2 will represent locations with medium concentrations, and 2 will represent locations with low concentrations. This approach will place greater emphasis on areas that appear to be of greatest concern based on RSC results, while ensuring that low-level samples do not contain elevated levels of COPECs that were not measured by RSC methods.

Table 3. Data Quality Objectives for Pier 1

RSC PILOT STUDY FOR PIER 1, THAMES RIVER

STEP 1: State the Problem

The area to the east of Pier 1 was sampled in 1999. Seven composite surface grabs (top 15 cm) and one composite core sample (30-60 cm) were collected and analyzed for a range of contaminants. The results indicate a gradient of contamination, with the highest concentrations at the head of the inlet east of Pier 1. Concentrations of mercury, copper, lead, nickel and zinc exceeded ER-Ms. Chromium, silver and arsenic exceeded ER-Ls, but were below ER-Ms. PAH concentrations also exhibited a gradient across the area with several individual PAHs and HPAH totals exceeding ER-Ms throughout the area, and LPAHs exceeding ER-Ms at stations near the head of the inlet. PCBs were detected in a similar pattern, with highest concentrations at the head of the inlet. Total PCBs exceeded the ER-M in five of the eight samples. Pesticides and butyltins were generally not present at elevated concentrations. Additional investigation is required to evaluate the potential ecological risk associated with the Pier 1 sediments. Based on the presence of elevated concentrations of multiple chemicals, the Pier 1 area will require further investigation to provide data to support the baseline ecological risk assessment (BERA).

STEP 2: Identify the Decision(s)

1. What are the spatial distribution and variability of Cu, Pb, Zn, Cr, As, total PCBs, and total PAHs in Pier 1 surface sediments as measured by rapid sediment characterization (RSC) methods?
2. What COPECs, as measured by fixed laboratory methods, exceed ecological screening threshold values or are detected and are known to bioaccumulate, and therefore should be carried forward for evaluation in a BERA?

Subordinate objectives:

1. Do the fixed laboratory confirmation sample data correlate with the RSC results?
2. Do the fixed laboratory data confirm that the chemical constituents measured by RSC methods co-occur with Hg and Ni measured in the fixed laboratory confirmation samples?
3. Do sediment characteristics (i.e., grain size and organic carbon content) potentially influence the RSC results?
4. Are there sediment or porewater characteristics that might be confounding factors in potential future toxicity tests (i.e., ammonia, salinity, grain size, TOC)?

STEP 3: Identify Inputs to the Decision(s)

1. Concentrations of Cu, Pb, Zn, As, Cr (using XRF measurement methods), and total PAHs and PCBs (using immunoassay test methods) in Pier 1 sediments.
2. Fixed laboratory confirmation data (using NOAA Status and Trends analytical methods) for three (3) Pier 1 samples and all reference site samples. Confirmatory samples will be selected based on RSC results and will span the range of RSC concentrations to provide sufficient data to support the development of correlation relationships.
3. Ecological screening threshold values.
4. Estimates of ammonia concentrations and salinity in porewater, and grain size and TOC data for confirmatory sediment samples.

STEP 4: Define the Study Boundaries

The Pier 1 area is defined by the head of the inlet on the north, Pier 1 on the west, and the shoreline on the east. The study will extend to a point approximately 150 ft south of the end of Pier 1. Sediment underneath Pier 1 will not be sampled. The top 5 cm of sediment from each station will be collected.

STEP 5: Develop a Decision Rule

The RSC data will be used to: 1) determine the distribution and concentrations of chemical constituents in Pier 1 sediments; 2) provide an estimate of spatial variability; and 3) support the design of a focused sampling plan involving multiple lines of evidence for an ecological risk evaluation.

Confirmatory laboratory data will be used to: 1) evaluate the degree to which the RSC results correlate with the fixed laboratory results; and 2) develop the list of COPECs for analysis in the BERA.

Field data on porewater salinity and ammonia concentrations, in conjunction with sediment grain size and TOC results, will be used to identify potential confounding factors that may need to be managed during potential future toxicity tests conducted as part of a more detailed validation/investigation study.

Table 3. Data Quality Objectives for Pier 1, continued

STEP 6: Evaluate Decision Errors The primary objective of the Pilot Study is to support the validation/investigation study DQO process and work plan development. Inadequate representation of sediment COPC concentrations in the Pier 1 area could result in an under- or overestimate of the spatial variance. This could lead to calculating incorrect sample sizes for the BERA study. The study area (Pier 1) was subdivided into two regions: inner and outer. The inner region is the location where historical data showed elevated PCB concentrations in several samples ranging from 0.14 to 9.2 ppm. Given the extreme variability and high concentrations in this area, a qualitative sampling design will be implemented to augment the existing data. The outer area historically had lower PCB concentrations ranging from 0.05 to 0.17 ppm. In this area, data will be collected to test the hypothesis that the mean PCB concentration is less than the ER-M, 0.18 ppm. The working hypothesis (or 'null' hypothesis) is that the mean value in the outer region is equal to or exceeds the PCB ER-M. The alternative hypothesis is that the mean value is less than the PCB ER-M. A false positive error would result in incorrectly determining that the area is below the threshold level. A 5% tolerance was established for this error type. A false negative error would result in incorrectly accepting the null (that the mean is equal or greater than the ER-M), when it is really below the threshold level. A 10% tolerance was established for this error type, and a gray region of 0.7 ppm was deemed acceptable.
STEP 7: Optimize the Design for Obtaining Data A systematic sample approach (i.e., triangular grid) based on a pre-determined number of samples (3) will be used to provide representation of the inner area. For the outer area, a systematic triangular grid with a random start was used to locate 9 samples to represent the area of interest. This sample size provides the desired power of 90% of determining that the mean concentration is below the 0.180 ppm threshold when the true mean is at the lower end of the gray region (0.110 ppm or less) Sample sizes were based on the historical variability of the samples collected in this region using Visual Sampling Plan (VSP), and a one sided t-test. Three confirmation samples for fixed laboratory analysis will be selected based on the RSC results. Confirmation samples will span the range of measured RSC concentrations to provide data for development of correlation relationships.

Table 4. Sampling Locations for RSC Pilot Study

Station ID	Sample ID	Latitude NAD83 in Decimal Degrees	Longitude NAD83 in Decimal Degrees
Zone 7			
Z7-1	CAB-1	41.400178	-72.095156
Z7-2	CAB-2	41.400111	-72.094090
Z7-3	CAB-3	41.400078	-72.093509
Z7-4	CAB-4	41.399869	-72.094624
Z7-4 Field Dup	CAB-5	41.399869	-72.094624
Z7-5	CAB-6	41.399849	-72.093677
Z7-6	CAB-7	41.399670	-72.093428
Z7-7	CAB-8	41.399514	-72.095139
Z7-8	CAB-9	41.399560	-72.094031
Z7-9	CAB-10	41.399403	-72.093611
Z7-10	CAB-11	41.399235	-72.094620
Z7-11	CAB-12	41.399247	-72.093378
Z7-12	CAB-13	41.399013	-72.094006
Z7-13	CAB-14	41.398737	-72.094640
Z7-14	CAB-15	41.398852	-72.093290
Z7-14 Field Dup	CAB-16	41.398852	-72.093290
Z7-15	CAB-17	41.398684	-72.093997
Z7-16	CAB-18	41.398571	-72.093452
Z7-17	CAB-19	41.398414	-72.094542
Z7-18	CAB-20	41.398397	-72.093203
Z7-19	CAB-21	41.398253	-72.093943
Z7-20	CAB-22	41.398130	-72.093348
Z7-21	CAB-23	41.397986	-72.094475
Z7-22	CAB-24	41.397988	-72.093128
Z7-23	CAB-25	41.397892	-72.093922
Z7-24	CAB-26	41.397713	-72.095014
Z7-25	CAB-27	41.397799	-72.093282
Z7-26	CAB-28	41.397504	-72.094419
Z7-27	CAB-29	41.397234	-72.093380
Z7-28	CAB-30	41.397133	-72.093728
Z7-29	CAB-31	41.396650	-72.094919
Z7-30	CAB-32	41.396636	-72.094238
Z7-30 Field Dup	CAB-33	41.396636	-72.094238
Z7-31	CAB-34	41.396680	-72.093794
Zone 4			
Z4-32	CAB-35	41.392153	-72.091004
Z4-33	CAB-36	41.391866	-72.090634
Z4-34	CAB-37	41.391656	-72.090951
Z4-35	CAB-38	41.391402	-72.091866
Z4-36	CAB-39	41.391496	-72.090424
Z4-36 Field Dup	CAB-40	41.391496	-72.090424
Z4-37	CAB-41	41.391293	-72.090732
Z4-38	CAB-42	41.391245	-72.091227
Z4-39	CAB-43	41.390852	-72.091488
Z4-40	CAB-44	41.391015	-72.090570
Z4-41	CAB-45	41.390991	-72.090265
Z4-42	CAB-46	41.390864	-72.090979

Table 4. Sampling Locations for RSC Pilot Study (continued)

Station ID	Sample ID	Latitude NAD83 in Decimal Degrees	Longitude NAD83 in Decimal Degrees
Z4-43	CAB-47	41.390700	-72.090408
Z4-44	CAB-48	41.390676	-72.090093
Z4-45	CAB-49	41.390442	-72.090702
Z4-46	CAB-50	41.390346	-72.091149
Z4-46 Field Dup	CAB-51	41.390346	-72.091149
Pier 1			
P1-47	CAB-52	41.390900	-72.089531
P1-48	CAB-53	41.390672	-72.089539
P1-48 Field Dup	CAB-54	41.390672	-72.089539
P1-49	CAB-55	41.390376	-72.089553
P1-50	CAB-56	41.390150	-72.089595
P1-51	CAB-57	41.389854	-72.089602
P1-52	CAB-58	41.389997	-72.089259
P1-53	CAB-59	41.389702	-72.089266
P1-54	CAB-60	41.389407	-72.089273
P1-55	CAB-61	41.389264	-72.089616
P1-56	CAB-62	41.389559	-72.089609
P1-56 Field Dup	CAB-63	41.389559	-72.089609
P1-57	CAB-64	41.389416	-72.089952
P1-58	CAB-65	41.389711	-72.089945
Reference			
T1SD3 – A	CAB-66	41.418632	-72.090162
T1SD3 – B	CAB-67	41.418896	-72.090570
T1SD3 – C	CAB-68	41.418623	-72.090983
T1SD3 – D	CAB-69	41.418900	-72.091301
T1SD4 – A	CAB-70	41.418807	-72.096964
T1SD4 – B	CAB-71	41.418509	-72.096647
T1SD4 – C	CAB-72	41.418796	-72.096362
T1SD4 – D	CAB-73	41.418499	-72.095989

Table 5. Pilot Study Sample Summary

	Number of RSC Samples	Number of RSC Field Duplicates	Number of Confirmatory Lab Samples	Number of Confirmatory Lab Field Duplicates[1]
Zone 7	31	3	15	0
Zone 4	15	2	11	0
Pier 1	12	2	3	0
Reference Stations	0	0	8	0
Total	58	7	37	0

[1]Laboratory quality control samples include 2 method blanks, 2 laboratory control samples, 2 matrix spikes, 2 matrix spike duplicates or laboratory duplicates, and 2 standard reference material samples.

ATTACHMENT 1

Field Logbook Forms

FIELD LOG

BATTELLE DUXBURY OPERATIONS
397 Washington Street, Duxbury, MA 02332
Duxbury Project Number: G4863057700
Project Title: Thames River RSC Pilot Study

Survey Number ID: CAB
Facility Name: Naval Submarine Base New London
Edgewater Drive, Suite 747, Oakland, CA 94621

THAMES RIVER RSC PILOT STUDY

NAVAL SUBMARINE BASE
NEW LONDON, CT

Prepared for:

DEPARTMENT OF THE NAVY
ENGINEERING FIELD ACTIVITY, NORTHEAST
NAVAL FACILITIES ENGINEERING COMMAND
10 Industrial Highway
Mail Stop, #82
Lester, PA 19113-2090

Contract No. N62472-00-D-1300

Battelle Project No. G486305

IF THIS LOGBOOK IS LOST AND YOU FIND IT, PLEASE CONTACT:

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After May 15, 2003
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Duxbury, MA 02332
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OR contact: Patty White (781)952-5279
397 Washington Street Duxbury, MA 02332

BATTELLE DUXBURY OPERATIONS
397 Washington Street, Duxbury, MA 02332
Duxbury Project Number: G4863057700
Project Title: Thames River RSC Pilot Study

Survey Number ID: CAB
Facility Name: Naval Submarine Base New London
Edgewater Drive, Suite 747, Oakland, CA 94621

PERSONNEL AUTHORIZED TO MAKE ENTRIES IN THIS LOG BOOK INCLUDE:

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FREQUENCY OF REVIEW: ***Data Sheets Weekly; Entire Logbook at Completion of Work***
CONCURRED BY: _____ ***, PROJECT MANAGER***
DATE: _____

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NAVY/NEW LONDON ONSITE				
Dick Conant	Navy Liaison	860-694-4899	860-694-5176	mailto:ConantD@subasenlon.navy.mil

LABORATORY SHIPPING ADDRESS:

Analytical Lab
Ms. Jessica Fahey (Custodian) ***Battelle Duxbury Operations*** ***397 Washington Street*** ***Duxbury, MA 02332*** ***Notification Letter Attention:*** ***Mr. Bob Lizotte*** ***Tel: (781) 952-5235*** Fax: 781-934-2124

TAILGATE SAFETY MEETING RECORD FORM

BATTELLE DUXBURY OPERATIONS
397 Washington Street, Duxbury, MA 02332
Duxbury Project Number: G4863057700
Project Title: Thames River RSC Pilot Study

Survey Number ID: CAB
Facility Name: Naval Submarine Base New London
Edgewater Drive, Suite 747, Oakland, CA 94621

Date:_____ Time:_____

Specific Location: _____

Type of Work:_____

_____

Chemicals Present:_____

SAFETY TOPICS DISCUSSED

Protective Clothing/Equipment: _____

_____

Hazards of Chemicals Present: _____

_____

Physical Hazards: _____

_____

Special Hazards: _____

_____

Other Topics: _____

_____

ATTENDEES

Name (printed)	Signature

DAILY LOG FORM

BATTELLE DUXBURY OPERATIONS
397 Washington St., Duxbury, MA 02332
(781) 934-0571 Fax (781) 934-2124
Project Number: G486305

Survey Number: CAB
Facility Name: New London Naval Submarine Base
Project Title: Thames River RSC Pilot Study

DATE_____ **INITIALS**_____ **START TIME**_____ **STOP TIME**_____

FIELD LOCATION: _____

_____

VESSEL NAME _____

PERSONNEL	AFFILIATION

WEATHER

TIME	TEMP °C	SALINITY (ppt)	PRECIP	SKY	WIND

Refractometer SN:

COMMENTS

_____

BATTELLE DUXBURY OPERATIONS
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Edgewater Drive, Suite 747, Oakland, CA 94621

GPS CALIBRATION FORM

Check GPS at Established Benchmark at Survey start, prior to sample collection
Check GPS at least one reference checkpoint at beginning of each day.

Established Benchmark Name _____ **Established by** _____

Benchmark Location

Units and Datum	Northing / Latitude	Easting / Longitude
State Plane NAD 83 (Ft)		
State Plane NAD 27 (Ft)		
Lat Long NAD 83 (Decimal Minutes)		
Lat Long NAD 83 (Deg, min, sec)		

Comments_____

GPS Reference Checkpoint Name _____

Reference Checkpoint 1 Location

Units and Datum	Northing / Latitude	Easting / Longitude
State Plane NAD 83 (Ft)		
State Plane NAD 27 (Ft)		
Lat Long NAD 83 (Decimal Minutes)		
Lat Long NAD 83 (Deg, min, sec)		

Date/Time _____ **Vessel** _____

Unit Make/Model _____

Benchmark or Reference Checkpoint (circle one) Name _____

Established coordinates from table _____

Measured coordinates _____

Difference _____feet _____meters _____ Within 100 meters? Yes/No

If no, check operation, re-measure, or replace unit and repeat calibration check.

Benchmark or Reference Checkpoint (circle one) Name _____

Date/Time _____ Vessel _____

Unit Make/Model _____

Measured coordinates _____

Difference _____feet _____meters _____ Within 100 meters? Yes/No

If no, check operation, re-measure, or replace unit and repeat calibration check.

BATTELLE DUXBURY OPERATIONS
397 Washington Street, Duxbury, MA 02332
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Survey Number ID: CAB
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Edgewater Drive, Suite 747, Oakland, CA 94621

POTENTIAL GPS BENCHMARKS

Station Designation/PID	Location/Brief Description	Latitude	Longitude
PLANT LX5210	UConn, Avery Point property. Disk set in a rock outcrop.	41 18 57.88446 (N)	072 03 58.24651 (W)
STA 32022 LX7657	UConn, Avery Point property. Disk set on concrete monument on roof of building.	41 19 00.17389 (N)	072 03 50.25791 (W)
846 1490 K TIDAL BM	New London. Disk set in rock outcrop near Amtrak Railroad.	41 21 42.99760 (N)	072 05 41.02290 (W)

DETAILED DESCRIPTION OF EACH BENCHMARK LOCATION

Station Designation/PID	Description of Location
PLANT LX5210	The station is located about 3.0 mi south of Groton, on the property of the University of Connecticut at Avery Point located on the mouth of the Thames River, set in a massive ledge of rock outcrop, about 33.53 m (110.01 ft) northeast of the east bank of the Thames River an on the southwest side of West Circle Road which passes by the rock ledge. The station is an NGS triangulation station disk cemented in a drill hole on the southwest side of the massive rock ledge about 1.53 m (5.02 ft) above the bank of the river. Ownership: University of Connecticut. Contact Coast Guard R&D one day in advance before occupying the station. Phone: (860)441-2609. Alternate contact: Ms. Carolyn Budziszewski (Directors Office). Phone: (860)405-9010.

STA 32022 LX7657	The station is located about 3.45 mi southeast of New London, 3.0 mi south of Groton, 2.0 mi east-northeast of Ocean Beach, at the mouth and on the east side of the Thames River at the site of the University of Connecticut Southwestern Branch Marine Science Institute and Coast Guard Research and Development Center, at Avery Point. It is on the roof of the U.S.C.G. Research Center Building 23. The station is set in the top of a 18-inch square concrete monument which is bolted to the concrete roof of the building and projects about 4 inches above the roof level. Located about 12 ft (3.7 m) south of the north roof railing, 33 ft (10.1 m) north of the south roof railing and 60 ft (18.3 m) east of the west roof railing. Ownership: University of Connecticut. Contact Mr. Jay Spalding. Phone: (860)441-2687.
846 1490 K TIDAL BM	The station is located in New London about 0.15 mi west of the entrance road of the State Pier (Port of New London Terminal) on the northwest side of State Pier Road and on the southeast side of the Amtrak Railroad in a rock outcrop. It is located about 12.2 m (40.03 ft) northeast of an opening in a chain link fence and on the northwest side of the fence in a 4 X 24-ft rock outcrop projecting about 0.46 m (1.51 ft) above the ground. It is located 34.14 m (112.01 ft) northwest of the center of State Pier Road, 26.9 m (88.3 ft) west of a power pole with a guy wire, 7.47 m (24.51 ft) southeast of the southeast rail of the tracks, 2.29 m (7.51 ft) east of a metal post anchored in the outcrop (old fence post) and 1.41 m (4.63 ft) northwest of an NGS metal witness post attached to a fence post in the chain link fence. Ownership: Amtrak. Contact the Amtrak Police Department at (800)331-0008 before occupation.

SEDIMENT SAMPLING DATA FORM

BATTELLE DUXBURY OPERATIONS
397 Washington St., Duxbury, MA 02332
(781) 934-0571 Fax (781) 934-2124
Project Number: G486305

Survey Number: CAB
Facility Name: New London Naval Submarine Base
Project Title: Thames River RSC Pilot Study

DATE (mm/dd/yy) _____ INITIALS _____ AREA-STATION ID _____

ON STATION (time) _____ WATER DEPTH _____ Ft M Fm

STATION POSITION (NAD 83) Latitude or Northing _____ Longitude or Easting _____

SAMPLER USED (circle one) 0.1 m^2 van Veen Grab Ponar Grab or Petite Ponar Grab Push Core Other:

Rejection Codes:

OP	**Overpenetrated**	**DB**	**Debris interference**
DS	**Disturbed surface**	**NS**	**No sediment in sampler**

Time of First Attempt:

Attempt #	Accepted, Rejected	Comments, Reason for Rejection
	A R	
	A R	
	A R	
	A R	
	A R	
	A R	
	A R	
	A R	

Attempt #	Accepted, Rejected	Comments, Reason for Rejection
	A R	
	A R	
	A R	
	A R	
	A R	
	A R	
	A R	
	A R	

OVERALL SEDIMENT CHARACTERISTICS

SEDIMENT COLOR: Black Dark Gray Gray Brown Other _____

SEDIMENT TYPE: Rock Cobbles Gravel Sand (coarse med fine) Silt Clay Wood Chips

Other: _____

RPD measurement (redox) _____

Label with Unique Sample ID (attach here):

Comments

_____

_____

_____

BATTELLE DUXBURY OPERATIONS
397 Washington Street, Duxbury, MA 02332
Duxbury Project Number: G4863057700
Project Title: Thames River RSC Pilot Study

Survey Number ID: CAB
Facility Name: Naval Submarine Base New London
Edgewater Drive, Suite 747, Oakland, CA 94621

Entered by: _____ ***Date*** _____

The following information is (check one)
[ ] miscellaneous documentation
[ ] a deviation from Protocol, Work Plan or QA Plan (give title)

_____

[ ] a deviation from SOP No. _____

Description:

Impact on Project:

APPROVED BY:

_____ _____
Date

[1] Planned change (12 h prior notification): Don Gunster (Patty White), Rosanna Buhl (Deb Felton)
Minor Deviations (4 h notification): Don Gunster (Patty White), Rosanna Buhl (Deb Felton)
Major Deviations: (4 h notification): Don Gunster (Patty White), Rosanna Buhl (Deb Felton)

ATTACHMENT 2

Field Standard Operating Procedures

Battelle Duxbury Operations
Standard Operating Procedure
for
Collection and At-Sea Processing of Benthic Grab Samples
SOP 5-169-02

Battelle Duxbury Operations
Standard Operating Procedure

for

COLLECTION AND AT-SEA PROCESSING OF BENTHIC GRAB SAMPLES

Summary of Changes to this version: The procedures in the SOP have been updated to reflect the current processing routine and equipment used for benthic sediment collection by grab sampling.

1.0 OBJECTIVE

This SOP describes the collection and at-sea processing of benthic grab samples for chemical, biological and geophysical analyses.

2.0 PREPARATION

2.1 SUPPLIES AND EQUIPMENT

- Grab Sampler: Young-modified Van Veen, Van Veen, Smith-McIntyre, or Ponar
- Wooden base or stand for the sampler
- Bucket with pour spout
- Plastic syringes, outer diameter 2.54cm
- Sieving table with bucket rocking assembly
- Water filter unit with extra clean filters (5 micron nominal pore size)
- Hoses and fittings for the filter unit
- Seawater pump for filter unit
- Sieves, mesh size 0.3mm, 0.5 mm, 1.0mm, or as specified in the Quality Assurance Project Plan (QAPP)
- Sample containers: Plastic wide-mouth jars in various sizes for infauna, Whirlpak™ bags for Grain Size, glass or plastic jars with teflon-lined screw caps for chemistry, sterile specimen cups for microbiology, or as specified in the QAPP
- Squirt bottles
- Funnels
- Tape: electrical and teflon tape for sealing sample jar lids, and clear packing tape for securing/protecting the computer generated barcode labels
- grease pencils

2.2 REAGENTS

- Formalin (37-40% solution of formaldehyde)
- Borax (to buffer the formalin)

2.3 SOLVENTS (FOR CLEANING EQUIPMENT BETWEEN STATIONS)

- Acetone
- DCM (Dichloromethane)
- ETOH (Ethanol)

2.4 PREPARATION

1. Set the grab sampler on a wooden stand under the boom and attach the cable termination to the grab shackle with another shackle. Use an appropriate swivel with the shackles when performing deep-water operations.
2. Place the sieve table in a convenient location on deck and lash it tightly to cleats or eye bolts on deck. Reinforce the tables with c-clamps if necessary. Position the outlet hose from the sink of the table through the scupper or over the side so that the sieved sediment runs overboard.
3. Secure the water filter unit to the table with bracket mounts and insert a clean filter. Run one hose from the seawater source to the filter assembly, and another one from the filter to the sieving table.
4. If necessary, use a Y connector to run another hose from the filter to the grab sampling area for washing the grab between samples. (The grab should be washed with filtered seawater during sample collection)

2.5 OPERATION

2.5.1 Young-Modified Van Veen Sampler

1. Secure the doors on the top of the grab by tightening the wing nuts at the edge of each door.
2. Remove tension from the cable attached to the sampler. This allows the arms of the firing mechanism to flatten and the jaws to open. The arms of the sampler should be parallel to the deck.
3. Secure the two metal hooks on the upper arms onto the pegs on the arms attached to the jaws.
4. While holding the hooks in place, instruct the winch operator to apply tension to the cable, to hold the arms firmly in place.

2.5.2 Van Veen Sampler

1. Secure the doors on the top of the grab by tightening the wing nuts at the edge of each door.

2. Turn the release device at the end of the cable until the suspension chains are taught. This holds the jaws of the sampler open.
3. While holding the release device in place, instruct the winch operator to apply tension to the cable, this will hold the desired position until deployment.

2.5.3 Smith-McIntyre Sampler

1. Secure the lids on top of the sampler buckets by snugly tightening the screws. This prevents the loss of sample through the top.
2. Lift the bucket arms up until the catches on the support bar of the pressure plates are engaged. The buckets are held open by these catches.
3. Insert the hook of the cocking bar through the ring on top of the sampler.
4. Lift both pressure plates. This moves the firing assembly up. This usually requires two people.
5. Pull the cocking bar downward to compress the firing springs.
6. To engage the cam and hold the bucket assembly in the cocked position, slam the pressure plates down.
7. Check the catches to be sure that they are securely in place. If they are not securely in place the sampler could unintentionally fire and flip the cocking bar out of place away from the sampler.
8. Carefully remove the cocking bar. Note the pressure of the bar. If no pressure is detected, it means that the sampler is cocked; if pressure is detected, the sampler is not cocked and steps 4-8 must be repeated.
9. Insert the safety pins (attached to the sampler by a chain) through the cams. This will prevent the accidental firing of the sampler while it is on deck and not in use.

2.5.4 Ponar Sampler

1. Remove tension from the cable attached to the sampler. Align the arms of the sampler so that they lay parallel on the deck.
2. Secure the metal tab locks, located on the upper arms, underneath the lower arms.
3. While holding the metal tab locks in place, instruct the winch operator to apply tension to the cable, to hold the locks in place.

3.0 PROCEDURES

3.1 COLLECTION OF BENTHIC SEDIMENT SAMPLES

1. When vessel is on station, direct the winch operator to slowly lift the grab from its stand. During its ascent and while the boom swings overboard, the grab can be steadied with hands, lines or boat hooks. Once the boom is outboard, the winch operator should deploy the grab by paying out the cable.
2. When the cable goes slack, the grab is on the bottom. Instruct the NAVSAM™ operator to hit the event marker at this time. Alternatively, site coordinates can be manually recorded on

the station log. Initiate recovery slowly, until the grab is free from the bottom. After that, retrieve the cable at a steady rate, until the grab is visible near the surface. When the grab is visible, slow the rate of ascent so that it can be steadied as it is brought on board.

3. Set the sampler on the wooden stand, open the lid and inspect the sample for acceptability. An acceptable grab is one that displays the following characteristics:

   - Sampler is not overfilled with sediment, the jaws are fully closed and the top of the sediment is below the level of the open doors.
   - The overlying water is not excessively turbid.
   - The sampler is at least half full, indicating that the desired penetration has been achieved.
   - The sediment is level on at least one side.

   In certain locations, slight over-penetration may be accepted, at the discretion of the chief scientist. The chief scientist will make the final decision regarding acceptability of all grabs. The overall condition of the grab (i.e. "slightly sloped on one side") should be noted on the station log (Attachment 1). Penetration depth can be controlled to some extent by the addition or subtraction of weights and/or "mud shoes" on the frame of the grab.

4. All grabs taken are recorded by the NAVSAM™ operator on the station log. If the grab is rejected, record the reasons on the station log, along with other pertinent station information. (See Attachment 1: *Station Log for Benthic Sediment Grab Samples*)

5. If the sample is rejected, empty the grab, wash it thoroughly with seawater, and re-cock the sampler. Note that decontamination cleaning procedures (section 3.2) are not required when the grab is redeployed at the same station. The sampling procedure is repeated until an acceptable grab is obtained.

3.2 DECONTAMINATION CLEANING PROCEDURES

Sediment collection for infaunal analysis requires that the grab be cleaned with soap and seawater only between stations. Generally, for other types of sample analyses, the cleaning procedures to be followed between stations are as follows:

1. Chemistry, *organic and inorganic*: Wash the grab with soap and water, follow with a solvent rinse of acetone and then DCM.
2. Microbiology (*C. perfringens, enterococcus*, or fecal coliform): Wash the grab with soap and water, follow with an ethanol rinse.

Note that all solvents must be captured and disposed of in labeled aqueous waste containers. All instruments that come into contact with the sample (i.e. syringe, ruler, collection buckets) must be cleaned in the same manner as the grab.

3.3 COLLECTION OF SEDIMENT SAMPLE FROM THE GRAB

3.3.1 General

1. Once the grab is deemed acceptable, processing can begin. Measure the penetration depth of the grab by inserting a clean ruler into the sediment near the center of the sample. This depth may be compared to a chart of penetration depth versus volumes (Attachment 2), to determine the approximate volume of the grab. Record the depth and corresponding volume

on the station log (Attachment 1). It is important that all sediment is retained if the grab is collected for infaunal analysis (see section 3.3.2). If the grab is going to be analyzed for infauna, then the ruler should be rinsed over the grab so that all of the adhering sediment washes back into the sample.

2. For most programs, an estimate of the apparent Redox Potential Discontinuity (RPD) will be measured. Insert a 2.54 cm diameter syringe into the sediment and withdraw a core. Estimate the distance from the surface of the sediment to the upper portion of the black subsurface sediment (if visible) to the nearest .5 cm and record the distance on the station log (Attachment 1). If the grab is collected for infaunal analysis, the contents of the syringe and all adhering sediment must be washed back into the sample as described above. For all other analyses, the core may be disposed of.

3.3.2 Infaunal Samples

As discussed in Section 3.3.1, all sediments collected for infaunal analysis must be retained, paying particular attention to organisms visable in overlying water or stuck to the sides of the grab or the lids of the screen. Thorough and gentle washing of the entire grab sample into a clean collection bucket is necessary to ensure a representative sample.

3.3.3 Chemical, Physicochemical and Microbiological Samples

A subsample from the top two centimeters of the grab is required for samples collected for chemical, physicochemical and microbiological analyses. Samples obtained for chemical analyses (organic and inorganic) are collected with a Kynar-Coated grab to reduce the possibility of contamination. Once the grab has been deemed acceptable, remove the top two centimeters of sediment using a contaminant free (Kynar-coated or teflon) utensil. Place the sediment in a clean receptacle and gently homogenize for 1-2 minutes. Following homogenization, partition the sediment into the appropriate sample containers, in the amount specified in the QAPP. Samples to be analyzed for TOC, organics, and metals can be frozen immediately. Grain size and microbiology samples should be refrigerated, not frozen, unless otherwise specified in the QAPP.

3.4 INFAUNAL SAMPLE PROCESSING

Remove the entire sample from the grab according to Section 3.3.2. Once the entire sample is collected in the bucket, place the bucket in the rocking mount on the sieving table, with the spout directed toward the center of the table. Place a clean, 12-inch diameter 0.3mm screen in the table, over the sink, underneath the spout of the bucket. Add filtered seawater to the bucket while gently decanting the sample onto the screen. When the screen starts to fill up with sediment, direct the seawater onto the screen, and try to remove as much of the fine sediment as possible. While sieving, it is important to make sure that the sediment in the bucket is covered with water, and that the sides of the bucket have been washed down, to prevent organisms from drying out. The portion of the sample remaining on the screen after sieving is retained for analysis. Wash the contents of the screen to one side of the sieve using a gentle flow of filtered seawater. Place a funnel in an appropriately sized sample container (the sample material should ideally fill ½ to ¾ of the container) and carefully wash the sample through the funnel into the sample container with filtered seawater. Be sure to rinse the funnel and to cap the jar to prevent loss from spilling. Continue this process until the bucket is empty.

Once the entire sample has been sieved and collected in the sample jar, add buffered formalin to obtain a final concentration of 10% formalin (e.g. 100 mls of formalin in a 1L container), and fill the jar to the threads with seawater. In many programs, a heaping tablespoon of Borax is added to the sample to ensure

adequate buffering of the slightly acidic formalin. Gently swirl the contents of the jar to ensure complete mixing of the sample and the formalin. Affix the sample label and cover it with clear packing tape. Seal the jar tightly and tape the lid with teflon and/or electrical tape to prevent leakage and escape of fumes during transport.

If the sample is made up of heavy material that will not wash through the sieve (i.e. course sand, rocks, shell hash) it may be necessary to modify the sieving scheme to avoid injuring the organisms. This is accomplished by an elutriation procedure. The contents of the bucket are flooded with seawater and gently swirled to encourage the small infaunal organisms to float to the top. The elutrient is then poured off onto the screen. The procedure is repeated until organisms are no longer visible in the elutrient. The portion of the sample retained on the screen is referred to as the light density fraction, the portion remaining in the bucket is the heavy density fraction. The two fractions are rinsed into separate, labeled sample jars. Whenever a sample is divided into more than one jar, for any reason, the jar label must reflect the number of jars. The NAVSAM™ operator should be instructed to print the appropriate number of labels and the number of jars should be hand-written on the label (i.e. 1 of 2). The number of jars should also be noted on the custody form (Attachment 3).

4.0 CALCULATIONS

There are no calculations necessary for this procedure.

5.0 QUALITY CONTROL

Field replicates and equipment blanks for chemistry analysis will be collected according to the QAPP. Any deviations from this SOP must be documented on the station log in the survey logbook Careful attention to the procedures described in this SOP by trained, qualified personnel will ensure the quality of the samples collected.

6.0 TRAINING

A technician training in these methods must first read this SOP in its entirety. The trainee will then practice the procedures in the field under the watch of the instructor until proficiency is demonstrated.

Individuals performing grab sample processing must be trained by a qualified sample processor. A qualified sample processor is considered to be an individual who has performed the processing in the past (within 2 years) and for whom a training certificate or equivalent documentation of experience exists. The methods in Section 3.0 are used as the training standard.

A trainee should begin by reading this SOP in its entirety; all questions should be directed to a qualified sample processor. A qualified sample processor must then demonstrate the procedures listed in Section 3.0 in their entirety with the trainee and then observe the trainee perform the procedures independently.

Once proficiency has been verified, a certificate of training (Attachment 4) will be completed for the trainee. The original certificate is maintained in the Quality Assurance Unit.

7.0 SAFETY

All personnel should wear protective clothing, safety glasses, and gloves when handling sample containers or reagent bottles containing the formaldehyde or the solvents. All collectors should wear life vests and hard hats when handling the grab on deck.

During training, the qualified sampler should explicitly point out hazards associated with the reagents and or equipment that are used:

- **Formaldehyde** and its vapors are dangerous.
- **DCM (Dichloromethane)** is a suspected cancer hazard. It is harmful if swallowed, inhaled or absorbed through the skin.
- **Acetone** is an extremely flammable liquid. It may be harmful if absorbed through the skin. Can cause severe skin and eye irritation.
- **ETOH (Ethanol)**

Gloves, safety glasses (with side shields) and protective clothing must be worn when handling any of the above chemicals. Open containers must remain in a fume hood or used on deck at sea. If the analyst is exposed to any of the above chemicals the following procedures should be followed:

Skin contact. Remove contaminated clothing immediately, directing a stream of water under clothing while it is being removed, if possible. Wash affected area 15-20 min and ensure no evidence of chemical remains.

Eye contact. Wash eyes immediately with large amounts of water while lifting upper and lower lids for at least 15-20 min. Get medical attention immediately.

Ingestion. If victim is conscious, give large quantities of water immediately. **DO NOT INDUCE VOMITING.** Milk or activated charcoal may also be administered by trained medical personnel. Get medical attention immediately. Contact Poison Control Center and adhere to their advice.

Inhalation. Remove from exposure area to fresh air immediately. If not breathing, give artificial respiration. If breathing is difficult, give oxygen.

Analysts using this procedure must read the material safety data sheets (MSDS) associated with these materials thoroughly. MSDS are located in the laboratory and on the boat. In the event of an emergency, the MSDS should be submitted to the appropriate medical personnel.

ATTACHMENTS

Attachment 1. Example of Station Log
Attachment 2. Grab Penetration Depth to Sediment Volume Conversion Chart
Attachment 3. Example of Chain of Custody form
Attachment 4. Example of Training Certificate

APPROVALS

Author	Janine D Boyle Name	3/16/99 Date
Technical Concurrance	Wayne R. Trulli Name	3/16/99 Date
Quality Systems Manager	Dana Burke Name	3-17-99 Date
Field Operations Section Manager	Wayne R. Trulli Name	3/16/99 Date

ATTACHMENT 1. Example of a Benthic Survey Station Log

STATION LOG

For Benthic Sediment Grab Samples

Project Name:

SURVEY: DATE: TIME ON STATION:	STATION DEPTH:	Recorded By:
Comments	Sample ID Label	Field Measurements
		Grab Size: 0.04-m^2 0.1-m^2
		Grab Penetration (cm):
		Sediment Texture:
		Redox Depth (cm):
		Analyses: (circle all applicable) Organics Metals TC GR CL EN/FE FA
		Comment:
		Grab Size: 0.04-m^2 0.1-m^2
		Grab Penetration (cm):
		Sediment Texture:
		Redox Depth (cm):
		Analyses: (circle all applicable) Organics Metals TC GR CL EN/FE FA
		Comment:
		Grab Size: 0.04-m^2 0.1-m^2
		Grab Penetration (cm):
		Sediment Texture:
		Redox Depth (cm):
		Analyses: (circle all applicable) Organics Metals TC GR CL EN/FE FA
		Comment:
		Grab Size: 0.04-m^2 0.1-m^2
		Grab Penetration (cm):
		Sediment Texture:
		Redox Depth (cm):
		Analyses: (circle all applicable) Organics Metals TC GR CL EN/FE FA
		Comment:

PLEASE RETURN THE ORIGINAL OF THIS COMPLETED STATION LOG FORM TO BATTELLE

TC= total organic carbon, GR = grain size, CL=*C perfringens*, EN/FE= Enterococcus and Fecal Coliform, FA = Infauna

ATTACHMENT 2.
Example of Penetration to Volume Conversion Chart

Chart Used to Convert Grab Penetration Depth (cm) to Sediment Volume (L) for the 0.1-m^2 van Young-modified Van Veen grab sampler.

Sediment Volume (L)	Grab Penetration Depth (cm)
3.5	5.5
4.0	6.0-6.5
4.5	7.0
5.0	7.0
5.5	7.5
6.0	7.5
6.5	8.5
7.0	8.5
7.5	9.0
8.0	9.5
8.5	10.0
9.0	10.0
9.5	10.5-11.0
10.0	11.5-12.0
10.5	12.5
11.0	13.0
11.0+	13.5 maximum

ATTACHMENT 3. Example of Chain of Custody Form

ATTACHMENT 4.

BATTELLE DUXBURY OPERATIONS CERTIFICATE OF TRAINING

SOP No. 5-169-02

8.0 SOP TITLE: COLLECTION AND AT-SEA PROCESSING OF BENTHIC GRAB SAMPLES

Trainee: _____

Instructor: _____

Date SOP Read: _____

Date Training Completed: _____

Approved: _____ **Date:** _____

Battelle Duxbury Operations
Standard Operating Procedure
for
Packaging and Shipping of Samples
SOP 5-210-03

Battelle Duxbury Operations
Standard Operating Procedure

For

PACKAGING AND SHIPPING OF SAMPLES

Summary of changes in this version: An attachment that provides instructions for field personnel, additonal information for sample packaging and shipping, and a training section are added.

1.0 OBJECTIVE

The purpose of this SOP is to define the procedures, responsibilities, and documentation associated with the packaging and shipping of samples.

2.0 RESPONSIBILITIES

The sample custodian or designee is responsible for the proper packaging and shipping of samples from the laboratory. The Project Managers are responsible for informing the sample custodian or designee as to when and where the samples or sample containers are going to be shipped. The project manager or designee is responsible for contacting the recipient of the material to be shipped to notify them of a pending delivery.

3.0 EQUIPMENT

Field Pack Equipment
Coolers
Bubble Wrap
Teflon Tape
Black Ball Point Pens **NO "SHARPIES"**
Blank custody forms
Proper Jars for sampling
Packaging Tape
Chain-of-custody (COC) Seals

Sample Transmittal Equipment
Coolers
Bubble Wrap
Sample Transmittal Forms
Samples
Cover Letter
Zip Lock Bag

4.0 PREPARTION

4.1 Cooler Preparation

Coolers should be washed inside and outside with soap and warm water to avoid any possible contamination of the samples. The coolers should have two sturdy handles, a working top, and be in good shape. Do not use any coolers that are damaged or are contaminated.

4.2 Cooler Labeling

It is critical that cooler labels are secured to the cooler to ensure that samples are not lost.

- The shipping label (typically Fed Ex) should be permanently attached to the cooler lid, NOT the cooler handle. In order to ensure that the label doesn't fall off, scrub the cooler lid and rinse with a solvent (e.g., methanol). Stick the label on the lid and tape over it with packing tape.
- In addition to the shipping label, a full label with the recipient's name and address as well as the sender's name and address should be attached to the outside of the cooler.
- The sample custody form should include the full addresses of the recipient and the shipping organizations, as well as a contact name at each organization.

5.0 PROCEDURE

There are two types of shipping performed by the sample custodian or designee. The most common is Sample Transmittal or sample transfer, this occurs when the laboratory custodian ships samples to an outside contractor. The second type of shipping is the preparation and shipment of "Field Packs."

5.1 Shipping Samples

The sample custodian or designee packs the samples securely in a cooler with bubble wrap and adds blue ice or crushed ice to achieve the proper temperature and to ensure that the samples stay at a constant temperature for their entire trip. The cooler should have at least one inch of bubble wrap placed on the bottom of the cooler and the samples should be wrapped in bubble wrap if breakable or crushable containers are used. Cubitainers are soft plastic and are easily punctured. Cubitainers should not be packaged with anything that has sharp edges. The samples must be packed tightly and not be able to move freely in the cooler; they must be secure. An upper weight limit of 70 pounds per cooler is suggested. All paper work is signed, the original custody form is placed in a zip lock bag with a cover letter, and taped to the top of the cooler to avoid moisture.

Cover letter accompanying samples should include
- the name of the Battelle technical contact;
- a statement about the number of coolers being shipped;
- a description of the work to be performed or a reference to the appropriate document (e.g., contract, QAPP); and
- a request that the receiving laboratory return the signed custody forms.

When one sample shipment is contained in multiple coolers, the custody forms should be copied, placed in Zip-lock bags, and attached to the inside top of each cooler[1]. Copies should be clearly labeled as such and they should indicate which samples are contained in each cooler. The individual coolers should be numbered 1 of 3, 2 of 3, etc. In addition, the Federal Express (or other transporter) label should be completed to indicate the cooler number and total number of coolers in the shipment (1 of 3, 2 of 3, etc.). Each cooler shipped by Federal Express receives a unique tracking number. Copies of all paper work associated with a sample shipment are stored in the custody form logbook for tracking purposes. (Note that it is Battelle policy that all cover letters receive one-over-one approval).

Shipping over national holidays should be avoided whenever possible.

[1] Some projects may require that a separate, cooler-specific custody form be prepared to inventory the contents of each cooler. This requirement should be communicated to the sample custodian by the project manager.

5.2 Shipping Field Packs

The second form of shipping is a "field pack." In this type of shipping the empty jars and coolers are sent to clients for sampling in the field. They might consist of just an empty cooler or include a complete kit of PC (certified) grade jars, pens, packing tape, bubble wrap, Teflon tape, custody forms and warm blue ice to be frozen in the field. This type of packaging needs to be secured in the same manner as actual samples (described above). Field packs must be accompanied by Attachment 1, which describes sampling and documentation requirements for field personnel to ensure the integrity of the samples.

If the sample jars are shipped from Battelle, the certificate which comes with the jars certifying that they are precleaned must be maintained in the Sample Jar Logbook. The custodian notes the jar lot on the sample custody forms that are shipped to the client. If the sample jars are drop-shipped directly to the field then Battelle is not responsible for retaining the jar certificates unless they are shipped to Battelle with the samples. The handling of the cleaning certificate should be specified in the cover letter. The project manager should specify whether the cleaning certificate should be returned to Battelle or maintained with the field records.

6.0 TRAINING

The sample custodian is normally responsible for the shipment of samples off-site. Therefore, the sample custodian and alternates must receive training in this SOPs. Another individual may perform the activities described in this SOP under the supervision of the sample custodian or another trained staff member. A person who is being trained to ship samples from Battelle must first read this SOP. The person may then perform specific tasks under the supervision of a qualified instructor. A certificate of training (Attachment 2) is issued upon completion of training and provided to the Quality Assurance Unit.

ATTACHMENTS
Attachment 1 Instructions for field sampling teams
Attachment 2 Certificate of training

APPROVALS

Author	Roanna Buhl	4-02-01
Laboratory Supervisor	[illegible]	4.3.01
Quality Systems Manager	Roanna Buhl	4-4-01
Analytical Services and Field Operations Resource Manager	Carlos J. McCarthy (Name)	4/3/2001 (Date)

Attachment 1
Instructions For Field Sampling Teams (Page 1 of 3)

RESPONSIBILITIES

The person collecting the samples (sample collector) is responsible for

- Collect and preserve samples in accordance with approved procedures, as specified in the project specifications or the attached guidance
- Collect sufficient sample for the intended analyses
- Adjust the pH to < 2 if the sample is intended for volatile organics analysis (and also adding sodium thiosulfate if total residual chlorine is present)
- Assign a sample number or code at the time of collection that uniquely identifies that sample
- Label each sample container with the sample number, project identification, date of collection, collector's initials, and storage requirements (room temperature, frozen, chilled). Labeling system must be water resistant and use indelible ink. Sharpies cannot be used for VOA samples.
- Document sample collection (location, date and time of collection, collector's name), sample type (matrix), preservation, and any special remarks on the custody form
- Collect samples into proper sample containers (see table below)
- Package samples for shipment in a manner that minimizes the risk of breaks and leaks and to ensure that the samples are maintained at the appropriate temperature
- Complete and sign the custody form completely, accurately, and legibly
- Enclose the custody form in a sealed plastic bag, and attach the bag to the inner top of the cooler
- Ensure integrity of the samples by sealing or locking the shipping container(s) and applying custody tape (if required)
- Arrange timely transportation of samples to the laboratory; identify on the shipping label the name of the person to whom the samples should be delivered
- Ship samples with ample time to meet holding time requirements and ample volume for the intended analyses
- Call the laboratory to notify the laboratory that the samples have been shipped and when they will arrive

If the samples show signs of damage or contamination, contact the sampling team project manager immediately to determine if samples should be re-collected. Compromised samples must be segregated and shipped separately to avoid potential cross-contamination.

DECONTAMINATION

All sampling equipment should be thoroughly decontaminated prior to the field sampling efforts. The employer of the sample collector is responsible for this training and for documenting the decontamination procedures. Decontamination must be appropriate for the types of samples and intended analysis.

SHIPPING REQUIREMENTS

It is Battelle policy that samples are not received outside of regular business hours unless the project manager has made specific arrangements with the laboratory manager and the sample custodian in advance.

Attachment 1
Instructions For Field Sampling Teams (Page 2 of 3)

REJECTION OF SAMPLES

It may be necessary for Battelle to reject samples if any of the following conditions are noted upon receipt:

- The integrity of the samples is compromised (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
- VOC vials contain bubbles of sizes greater than 1% of the vial volume
- Incomplete sample custody forms: the sample collector or the intended analysis is not documented, or the custody forms are not signed and dated by the person who relinquished the samples
- Samples are designated for VOA analysis but no VOA trip blank is provided.

Attachment 1
Instructions For Field Sampling Teams (Page 3 of 3)

HOLDING TIMES AND CONDITIONS

The employer of the field sampling team is responsible for determining, documenting, and communicating to the field sampling team the holding times required for the intended analysis. In the absence of other instructions, the attached criteria should be applied.

Compound Class	Containers (Volume)	Preservation		Holding Time
		Temperature	Other	
WATER (1 L except TBT 2 L)				
Pesticides and PCBs	Glass with Teflon lined caps	Cool 4°C ±2°C	pH 5-9 if held longer than 72 hours	7 days
SVOA, TPH or Fingerprinting	Glass with Teflon lined caps	Cool 4°C ±2°C	Store in dark	7 days
VOA	Glass with Teflon lined caps	Cool 4°C ±2°C	pH<2 Headspace ≤1% of sample No bubbles	14 days
TBT	Polycarbonate Teflon lined caps	Freeze ≤20°C		90 days
Metals	Teflon	Cool 4°C ±2°C	Acidify with 0.2% nitric acid < 2pH	28 days (Hg) 6 months (Other metals)
SEDIMENT OR SOIL (50 g)				
Pesticides and PCBs	Glass with Teflon lined caps	Cool 4°C ±2°C or Frozen <20°C		14 days 1 year
SVOA or TBT	Glass with Teflon lined caps	Cool 4°C ±2°C or Frozen <20°C		14 days 1 year
VOA, TPH, or Fingerprinting	Glass with Teflon lined caps	Cool 4°C ±2°C		14 days
Metals	Polystyrene or Glass with Teflon lined caps	Cool 4°C ±2°C		28 days (Hg) 6 months (Other metals)
TISSUE (50 g)				
All classes	Glass with Teflon lined cap or for organic analysis solvent-rinsed foil	Freeze ≤20°C		1 year

Attachment 2

RECORD OF TRAINING
For
SOP No. 5-210-03
Packaging of Samples for Shipment

The above mentioned SOP is relevant to your work. Your signature below signifies that you have read and understand the requirements associated with this procedure.

Trainee

Instructor

Date SOP read and understood

Comments:

Approval_____ Date_____

Battelle Duxbury Operations
Standard Operating Procedure
for
Sample Receipt, Custody and Handling
SOP 6-010-11

Battelle Duxbury Operations
Standard Operating Procedures
for

SAMPLE RECEIPT, CUSTODY, AND HANDLING

Summary of changes in this version: A new Sample Custody Corrective Action Form has been added. The Sample Receipt Form has been modified. Details on the evaluation of preserved samples and pre-cleaned bottles have been added. A sample log-in flowchart has been added.

1.0 OBJECTIVE

Sample control is a vital aspect of any environmental monitoring program which generates data that may be used for regulatory purposes or as evidence in a court of law. Additionally, the complexity of many environmental sampling programs, which may involve the collection and analysis of samples of various media from different sites to be analyzed for several parameters, makes a sample control system essential. This standard operating procedure (SOP) defines the procedures, organizational responsibilities, and documentation requirements associated with the Laboratory sample control system.

The routine flow of samples through the laboratory is illustrated in Attachment 1. Additional sample control procedures may be required to meet the needs of specific projects. These procedures will be defined in the project plan.

2.0 DEFINITIONS

Custody Records — The administrative records associated with the possession history of each sample from the time of collection, through analysis, to final disposal.

Chain-of-Custody Records — 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.

Legal or Evidentiary Chain of Custody (COC) – A special type of sample custody which requires that the physical possession, transport and storage of a sample be documented in writing. The records must account for all periods of time from sample container acquisition through sample disposal.

Sample control — The formal system designed to provide sufficient information to reconstruct the history of each sample, including collection, shipment, receipt and distribution within the laboratory, analysis, storage or disposal, and data reporting.

Sample custody — 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
- The samples are in a secure area

3.0 RESPONSIBILITIES

Sample Collector — The person collecting the samples is responsible for

- Collecting and preserving samples in accordance with approved procedures, as specified in the project-specific plan and SOPs
- Adjusting the pH to < 2 if the sample is intended for volatile organics analysis (and also adding sodium thiosulfate if total residual chlorine is present)
- Assigning a number or code at the time of collection that uniquely identifies that sample
- Labeling each sample container with the sample number, project identification, date of collection, collector's initials, and storage requirements (room temperature, frozen, chilled)
- Documenting sample collection and preservation
- Packaging samples for shipment in a manner that minimizes the risk of breaks and leaks and to ensure that the samples are maintained at the appropriate temperature
- Completing and signing the chain-of-custody records accurately and legibly
- Ensuring integrity of the samples by sealing or locking the shipping container(s) and applying custody tape (if required)
- Arranging timely transportation of samples to the laboratory, including identifying on the shipping label the name of the person to whom the samples should be delivered

Laboratory Sample Custodian — The responsibilities of the Laboratory Sample Custodian include:

- Receiving samples (for details see Section 4.1).
- Maintaining records of sample receipt, movement in and out of storage, and release, archival, and disposal
- Distributing completed custody forms according to Section 4.1.5.
- Returning the shipping cooler to the client or shipper.
- Communicating sample custody problems to the project manager and implementing corrective action as directed (Section 4.8).

Alternate Sample Custodian — The Alternate Sample Custodian is responsible for assisting the Laboratory Sample Custodian and for performing the above tasks in the absence of the Laboratory Sample Custodian.

Project Manager — The Project Manager is responsible for communicating

- expected receipt dates and project-specific receipt requirements to the Sample Custodian
- the potential presence of total residual chlorine to the Sample Custodian
- sample custody-related problems to the client
- corrective action to the sample custodian and laboratory manager

Laboratory Manager — The Laboratory Manager is responsible for designating the Laboratory Sample Custodian and the Alternate Sample Custodian and for ensuring that these individuals are trained to perform the tasks specified in this SOP.

4.0 PROCEDURES

4.1 SAMPLE RECEIPT

4.1.1 Hours

Samples that are admitted to the laboratory during normal business hours are either delivered to the front desk or to a designated area in the Chemistry North Building. The Sample Custodian is notified immediately.

It is Battelle policy that samples are not received outside of regular business hours unless the project manager has made specific arrangements with the laboratory manager and the sample custodian in advance. Samples received in the laboratory outside of normal business hours are placed in a secure area under the appropriate storage conditions until they can be formally released to the Laboratory Sample Custodian.

- If samples are shipped by commercial carrier, the person receiving the samples should document the date and time of receipt on the container, and place the samples in a secure location.

- If samples are transported from the field to the laboratory by Battelle sampling personnel, the samples should be placed in a pre-arranged, secure location until they can be formally relinquished to the Laboratory Sample Custodian.

In either case, on the next business day, the Laboratory Sample Custodian logs in the samples. (Note that the receipt form allows for separate entries of receipt and log-in date).

Upon receipt of the samples, the Laboratory Sample Custodian will move the shipping containers to the sample custody room.

4.1.2 Sample Handling

The shipping container should only be opened under the vented hood. The sample custodian must determine whether the sample condition upon receipt is acceptable. That is, that the sample temperatures, pH, and containers, are appropriate for the intended analysis; and that the samples have been received within the required holding times. Attachment 2 defines acceptable sample handling and holding times. If sample containers, preservation, or timely delivery do not meet the criteria in Attachment 2 and section 4.1.3, then the sample custodian must notify the project manager who in turn must notify the client (section 4.8).

The sample custodian must review and document the following for proper receipt of the samples:

1. Method of delivery (i.e. commercial carrier, hand delivered) and presence/absence of chains-of-custodies.

2. Inspect the shipping container(s) for the presence/absence and condition of custody seals.

3. Inspect each sample for the presence/absence and condition of samples and custody seals.

4. Inspect each sample for breaks or leaks (see Section 5.0 for safety instructions).

5. Review the accompanying records for completeness and accuracy of sample labels and sample transmittal forms.

6. Measure and record the temperature of each container to document whether or not the samples were maintained at the appropriate temperature (frozen, cool, or room temperature) during shipment. The temperature of a cooler blank (if available), melt water, or the external temperature of the sample containers should be measured and documented. (Thermometers or probes are never inserted into a sample container).

7. Measure and record initial pH of water samples unless otherwise directed by the laboratory manager, project manager, or QAPP (see Section 4.2.1).

8. If the project manager indicates that TRC may be present, measure samples for total residual chlorine (TRC) and treat samples with sodium thiosulfate (Section 4.2.2).

9. Inspect VOC vials for bubbles of sizes greater than 1% of the vial volume. If present, notify the Project Manager immediately.

10. Upon completion of the sample inspection, the Laboratory Sample Custodian formally acknowledges receipt of the samples by signing, dating, and noting the current time on the sample transmittal form(s).

11. Log-in and assign unique laboratory identification numbers to each sample (see Section 4.3).

12. Storage of samples in the appropriate storage location until samples are ready to be further processed. VOC samples to be stored in a separate storage location than samples for other organic analyses. This includes releasing samples to the laboratory and to outside contractors.

13. Communicate sample custody problems to the project manager and implement corrective action as directed.

14. Distribute completed custody forms according to Section 4.1.5.

15. Return the shipping coolers to the client or shipper, if necessary.

4.1.3 Sample Acceptance/Rejection Criteria

Under some circumstances Battelle will place itself at risk by accepting samples for analysis because data that are generated from samples that do not meet chain of custody or handling requirements (Section 4.1.1) may be rejected by EPA. Battelle may currently analyze samples for the following regulatory programs

- Resource Conservation and Recovery Act (RCRA)

- Comprehensive Environmental Response, Compensation and Liability Act (Superfund) (CERCLA)
- Clean Water Act (CWA)

It is the responsibility of the sample custodian to ensure that the following conditions are recorded on the Sample Receipt Form. The Laboratory Sample Custodian will notify the Project Manager and Laboratory Manager in writing (See Section 4.8 and Attachment 3) of sample receipt, condition, and problems (e.g., breakage, leakage, missing samples, excessive temperatures). Upon completion of sample inspection, the Laboratory Sample Custodian formally acknowledges receipt of the samples by signing, dating, and noting the current time on the sample transmittal form(s).

It is the responsibility of the project manager to specify in the QAPP that project samples are being analyzed for compliance monitoring. In these cases samples could be rejected if:

- The integrity of the samples is compromised (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
- VOC vials contain bubbles of sizes greater than 1% of the vial volume
- Incomplete sample custody forms: 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.
- Samples are designated for VOA analysis but no VOA trip blank is provided.

If the sample custodian identifies any of the above conditions the project manager must be notified (Section 4.8).

4.1.4 Documentation

Documentation of sample receipt includes the original sample custody forms (or copies if the originals are returned to the shipper), any additional records of transmittal, the shipper's air bill (if applicable), and the Sample Receipt form. Sample custody records are filed by date in the Custody Logbook which is kept in the access controlled sample custody room. A record of Battelle Laboratory ID numbers, including cross-reference to original field IDs, is entered into the custody database and hard copies are stored in the custody room.

The condition of the samples, integrity of the custody seals, discrepancies between sample labels and transmittal forms, and unusual events or deviations from the project work plan or SOPs are documented in detail on a Sample Receipt Form (Attachment 4). Any problems are also recorded on the original custody forms, if present.

Occasionally, samples are received with only a letter of transmittal or no transmittal forms at all. In these cases the Sample Custodian should complete the sample log-in procedures (Section 4.3) and attach a printout from the Chemistry Laboratory Sample Receipt Database to the Sample Receipt form to provide a record in the Custody Log of the samples received.

4.1.5 Distribution

The sample custodian will provide the Project Manager, the QAU, and Lab Manager with a copy of all documentation that accompanied the samples.

The custodian should make the following distribution of custody forms:

- Copies of the sample receipt and custody forms are provided to the project manager, laboratory manager, and the quality assurance officer.
- If the custody forms that are received with the samples are multi-copied then the copies should be distributed as indicated on each copy (e.g., laboratory, customer (client), shipper).
- If the custody forms that are received with the samples are not multi-copied then the custodian should consult the project manager to determine if a copy of the custody records should be returned to the client and/or shipper.

4.2 SAMPLE PRESERVATION ADJUSTMENTS

4.2.1 pH Measurement and Adjustment

If water samples will not be extracted within 72 hours then the pH is typically adjusted according to the preservation requirements in Attachment 2. To measure pH, withdraw a small volume of water (0.1 mL) from the sample container using a baked Pasteur pipette. Place 1 drop on a narrow range pH paper strip and follow the instructions included with the pH paper to read the results. Record the pH on the Sample Receipt form (Attachment 4). If water sample pH is NOT between pH 5 and 9 consult the project and/or laboratory manager and make pH adjustments as directed by these managers. The decision to adjust sample pH is based on the target analyte list and is therefore made on a case-by-case basis.

To adjust the sample pH, sulfuric or hydrochloric acid (1+1) or 10 N NaOH is added until the sample pH is between 5 and 9. (Note that NaOH solutions must be pre-extracted prior to use to prevent sample contamination). The final pH and the volume of solution added in making the pH adjustment is recorded on the Sample Receipt form.

The following matrix should be used in the determination of bottle preservation and the pre-cleaned bottles:

Preservation	**Pre-cleaned bottles**
Yes=C-O-C and bottle match ,or documented on C-O-C and not on bottle	*Yes*=Sent out and received
No=C-O-C and bottle unmarked	*No*=Not sent and not received
Unknown=documented on the bottle and not on C-O-C	*Unknown*=Sent out and not received

For pre-cleaned bottles the PM should include in a cover letter to the client that the C-O-C should include documentation if Battelle has supplied bottles.

Any problems are documented on the original custody forms. If C-O-C signed without comments then the samples are considered acceptable upon receipt. The C-O-C should never be altered, comments can be added, but information should never be crossed off or written over.

Samples intended for VOA analysis should NOT be opened. pH adjustments must be made in the field.

4.2.2 Total Residual Chlorine Measurement and Treatment.

If the project manager indicates that samples may contain total residual chlorine (TRC) then the sample

must be treated with sodium thiosulfate according to Attachment 2 (PCB/Pest, PAH). The sample custodian must work directly with the project manager in these cases. Ideally, the sample is treated for chlorine in the field. However, the sample custodian should verify the absence of TRC using a commercial test kit. If TRC is detected then sodium thiosulfate is added at the ratio of 80 mg/L sample. This treatment is documented on the sample receipt form.

Samples intended for VOA analysis should NOT be opened. Treatment for TRC must be performed in the field.

4.3 SAMPLE LOG-IN

The receipt of all samples received by the Chemistry Laboratory will be recorded in the Chemistry Laboratory Sample Receipt Database (SOP 6-007). Each incoming sample is assigned a unique ID number, which is clearly and indelibly marked on each sample container and the custody form. Samples that contain more than one jar for the same analysis will be labeled with the same Lab ID and the jar number (e.g. 1 of 2 and 2 of 2). Alternatively, a separate ID number can be assigned to each container. The full ID including the jar number will be called out in the preparatory records. Upon completion of log-in procedures, samples are placed in a limited-access area at the appropriate temperature. The storage area is documented on the Sample Receipt form.

4.4 SAMPLE STORAGE

Upon completion of sample log-in procedures, samples are transferred to a secure location for storage. This location may be a room, refrigerator, or freezer, depending on the storage requirements of the samples, but must be an area that can be locked from the outside. The initial storage location is documented on the Sample Receipt form. Only the sample custodian, organic prep lab group leader, and the facilities manager have keys to these controlled-access areas.

The following storage requirements are applied to samples received at Battelle unless otherwise specified in the QAPP: tissue and sediment samples: ≤20°C; water samples: 4±2°C. Samples collected for compliance monitoring according to EPA regulatory methods are stored according to the conditions specified in Attachment 2 and should be specified in the QAPP.

Samples that are to be analyzed for volatile organic compounds must be stored in a separate storage location from the samples being analyzed for semi-volatile organic compounds.

4.5 SAMPLE TRACKING

Sample custody is transferred from the Sample Custodian to the sample prep Task Leader when sample preparation is initiated. The transfer of custody to laboratory personnel and all sample movement within the laboratory is documented on Daily Sample Tracking forms (Attachment 5) that are maintained with the prep records. Each technician is responsible for the care and appropriate storage of the samples in his/her custody, and for documenting the conditions under which the samples are maintained. Labs/areas which house samples in-progress must be controlled-access (locked) during non-working hours.

4.6 SAMPLE SPLITTING

The aliquotting of samples for multiple analyses is documented on Sample Split and Transfer Logs (Attachment 6). Split samples retain their original Laboratory Sample identification number. Sample

Split Logs are maintained with the original sample custody records. Lab Ids are distinguished by the analyses for which the sample was split.

4.7 SHIPMENT OF SAMPLES

Distribution of samples that are aliquotted at Battelle and shipped to external laboratories for analyses is normally documented on the Battelle Sample Split and Transfer Log. If the samples were collected or generated by Battelle, sample custody is tracked on Battelle Custody forms (Attachment 7) that are shipped with the samples. In either case the person who has custody of the samples is responsible for

- packing the samples for shipment such that temperatures requirements are maintained and samples are protected from breaks or leaks
- arranging for transportation.
- split forms and/or custody forms must be included with the samples shipment.

A *copy* of the transfer or custody form should be retained with the original custody records for tracking purposes. The custodian at the receiving laboratory documents sample receipt and condition on the form and retains the yellow copy. The white (original) and pink copies are returned to Battelle. The original is maintained with the original custody records and the pink copy is sent to the client, if requested. The preparation of field kits, custody of sample containers, and sample packing procedures are defined in SOP 5-210.

4.8 CLIENT NOTIFICATION

The client must be notified immediately if problems are noted during sample receipt and log-in so that corrective action may be initiated. The sample custodian may communicate directly with the client custodian or representative if discrepancies between sample labels and custody forms are noted or if samples are missing. The project manager should communicate other problems (e.g., holding time exceedences, preservation issues, incomplete or improper custody records – see Section 4.1.3). This notification and the clients directions for corrective action is documented on the Corrective Action form (Attachment 3). It must be specifically documented if the client approves analysis of the samples. All corrective action is communicated to the sample custodian or laboratory manager in writing.

Specific samples may include other client notification requirements (e.g., if permit thresh hold limits are exceeded the client must be notified within 24 hours of verified sample data). The project manager should define requirements these in the QAPP.

4.9 SAMPLE ARCHIVAL AND DISPOSAL

Sample extracts and unextracted field samples are returned to the custody of the sample custodian. Once sample analysis is considered final the samples can be archived.
The decision to archive samples should be made by the client and the Project Manager when the project is initiated. Sample disposition and the length of storage should be defined in the project plan. In the absence of other directives, unexpended samples are archived for six months after the delivery of the final data. Unless otherwise specified by the client, the samples will be discarded in the proper waste stream after this period. The project manager will be notified prior to the disposal of samples.

Sample extracts are held for one month after delivery of the final data. Unless otherwise specified by the

client the extracts will be discarded in the proper waste stream after this period. The project manager is not notified of extract disposal. It is the responsibility of the project manager to include sample disposition requirements in the QAPP and to communicate them to the sample custodian. The following documentation is required:

1. Samples or extracts for archival are boxed by batch.
2. The project numbers, title/clients, batch numbers, and extract fractions are labeled on the box.
3. A copy of the label is filed in the Sample Archival logbook and the archive location of the box (box number and freezer number) is documented in the logbook.

Sample disposal is documented in the Sample Archival Log Book by documenting "Discarded" with the date and initials of the custodian directly on the label copy. The appropriate handling and disposal procedures for sample and sample extract are discussed in SOP 5-114.

5.0 SAFETY

Sample handling must always assume that samples are potentially "contaminated." Therefore, sample shipping containers are always opened in a vented fume hood, and personnel protective equipment is worn when unpacking samples (safety glasses, lab coat, and gloves).

Occasionally, samples are received broken. Because the potential hazard may be unknown all spills must be treated as if the material is hazardous. Clean-up materials should be maintained in the sample custody room. These consist of

absorbent (e.g., speedi-dry)
dust pan and brush
glass disposal container
heavy-duty gloves
paper towels
plastic bags
solid waste stream container

The hazardous waste coordinator should be contacted to determine the proper disposal procedures for spilled sample. In general, water samples are absorbed into chemical absorbent; sediment, soil, or tissues are placed in heavy-duty plastic bags. These are both disposed of in the laboratory's solid waste stream. Broken glass containers are placed in the glass disposal container.

6.0 TRAINING

A person who is being trained as a sample custodian must first read this SOP. The person may then perform specific tasks under the supervision of a qualified instructor (Laboratory Sample Custodian or Alternate). Tasks performed by the trainee are reviewed and co-signed by the Laboratory Sample Custodian or Alternate until it has been established that the trainee is able to perform these tasks without supervision. A certificate of training (Attachment 8) is issued upon completion of training and provided to the Quality Assurance Unit.

ATTACHMENTS

1a. Sample Flowchart
1b. Sample Log-in Flowchart
2. Sample Handling Requirements

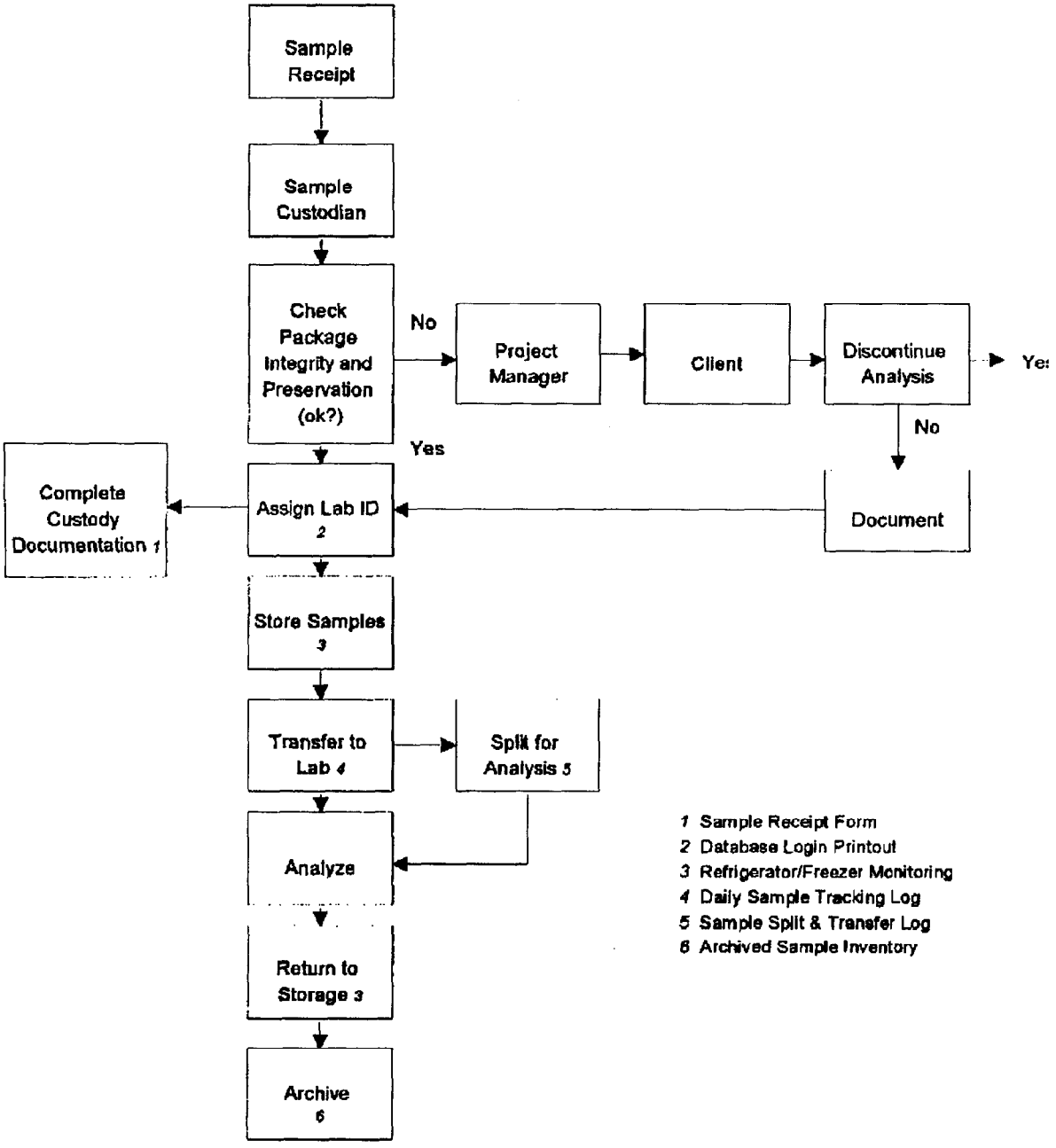
3. Sample Custody Corrective Action Form
4a. Sample Receipt Form
4b. Sample Receipt Auxiliary Form
5. Daily Sample Tracking Form
6. Sample Split and Transfer Log
7. Battelle Chain-of- Custody Record
8. Certificate of Training

APPROVALS

Author		7·10·00
Laboratory Manager		7·10·00
Quality Systems Manager		8-4-00
Environmental Chemistry Section Manager	Name	Date 8/7/2000

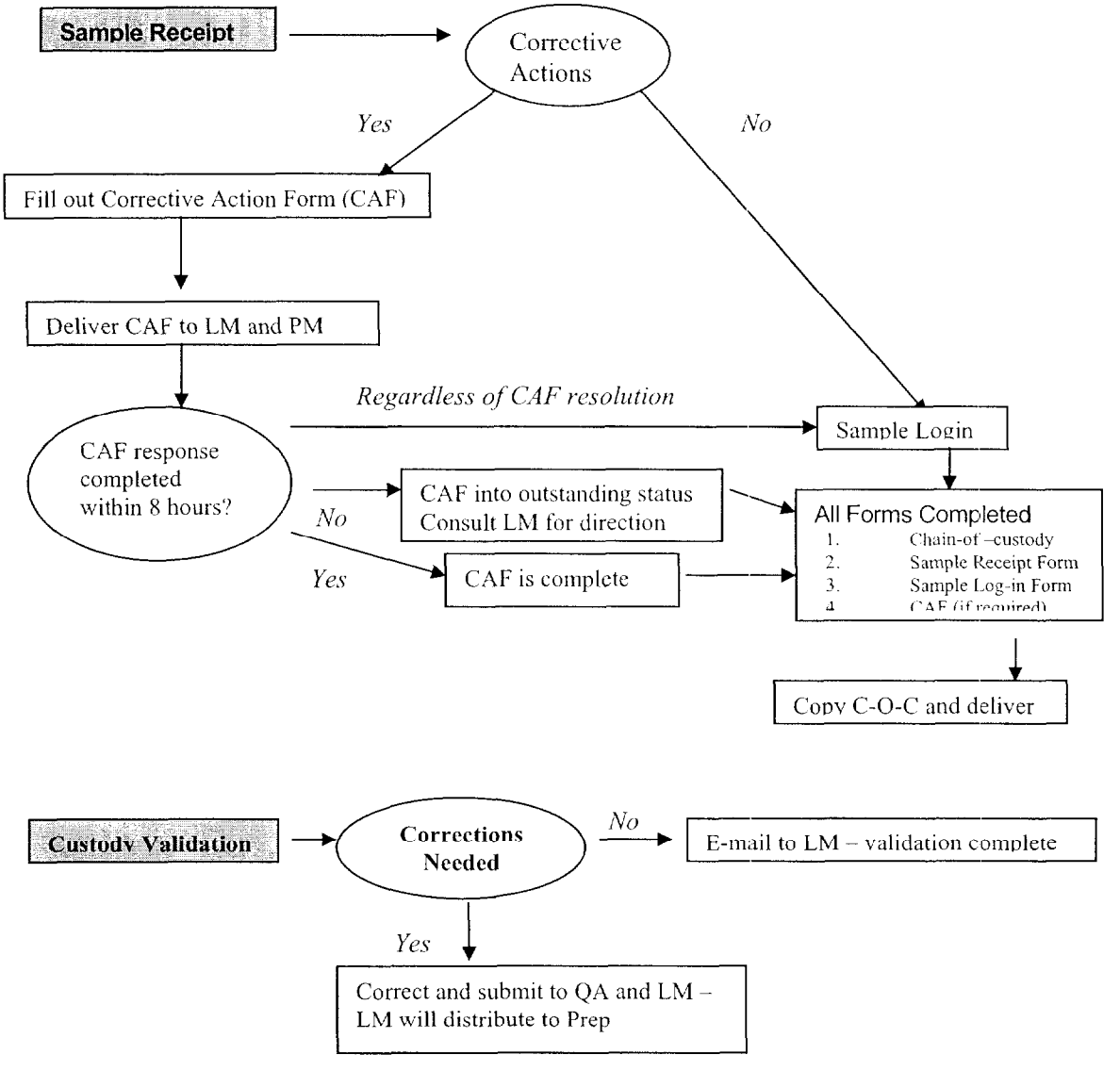
ATTACHMENT 1a
Battelle Duxbury Operations
Sample Flowchart

N:\6-010.AF2 Wednesday, August 7, 1996 9:34 AM



ATTACHMENT 1b
Battelle Duxbury Operations
Sample Log-in Flowchart

The following is the proper chain of events:



ATTACHMENT 2
Sample Handling Requirements

WATER

Compound Class	Containers	Preservation		Holding Time
		Temperature	Other	
PESTICIDES[1]	Glass with Teflon lined caps	Cool 4°C ±2°C	pH 5-9 if held longer than 72 hours Store in dark	7 days until extraction, 40 days after extraction
PCBs[1]				
PAH[1]			Store in dark	
Other SVOA • Haloethers • Phthalate Esters • Nitroaromatics • Isophorones • Nitrosamines				
TPH or FINGERPRINT				
VOA[1]			pH<2 Headspace ≤1% of sample	14 days
TBT		Freeze ≤20°C		90 days

[1]If Residual Chlorine is present in the sample it must be treated with sodium thiosulfate.

SEDIMENT/SOIL

Compound Class	Containers	Preservation		Holding Time
		Temperature	Other	
PESTICIDES **PCBS**	Glass with Teflon lined caps	Cool 4°C ± 2°C		14 days until extraction, 40 days after extraction
PAH **SVOA**		Freeze ≤20°C		1 Year
VOA		Cool 4°C ± 2°C		14 days
TPH or FINGERPRINT		Cool 4°C ± 2°C		14 days until extraction, 40 days after extraction
TBT		Freeze ≤20°C		1 Year

All tissue samples are stored frozen (≤20°C).

ATTACHMENT 3
Battelle Duxbury Operations
Sample Custody Corrective Action Form

Project Number _____ Client _____

Description of Problem:			**Explanation:**
*Client ID:**		Jars and C-O-C do not have matching IDs	
		Extra samples not listed on the C-O-C	
		Missing samples listed on the C-O-C	
		Either label or C-O-C cannot be verified	
Temperature and Preservation:		Receipt temperature outside of acceptability	
		Preservation discrepancy on C-O-C	
		Not documented as being compliant with the SOP	
VOA Analysis:		Water VOA samples >1% headspace or bubbles	
		No trip blank included	
Sample Container Integrity:		Sample Broken	
		Cap broken	
		Sample Leaking	
		Custody Seal broken	
*Custody:**		Jars and C-O-C do not match for time/date	
		Samples received outside of holding time	
		Samples not relinquished from sender	
		Sample Collector not identified	
		Incomplete Sample Custody Forms	
Other:			

* The C-O-C must reference this form for each sample issue

Project Manager and Laboratory Manager must be notified upon receipt of the samples when problems are identified

Documentation of project manager notification:

Sample Custodian: _____ _____
Laboratory Manager: _____ _____
Project Manager _____ _____
Signature Date

Documentation of client notification (should be completed by project manager within 24 hrs):

On _____ I contacted _____ at _____
Date Name of client contact Name of client organization

Results of communication with client (Describe any corrective action directed by the client):

Date this form was received back to the custodian:_____

ATTACHMENT 4a
Battelle Duxbury Operations
Sample Receipt Form

Project Number: _____ Client: _____
Received by: _____ Date/Time Received: _____
No. of Shipping Containers _____ _

SHIPMENT

Method of Delivery: _____ Commercial Carrier (Air bill No. _____)
_____ Hand Delivered
_____ US Mail (RPS No. _____)

COC Forms: _____ Shipped with samples _____No forms

Cooler(s)\Box(es) were sealed with: _____ Tape _____ Custody Seals_____(Other specify)
Were the seals intact for each shipping container?_____Yes_____No_____NA
If NO, see Sample Custody Corrective Action Form

SAMPLES

Sample Labels: _____ Sample labels agree with COC forms
_____ Discrepancies (see Sample Custody Corrective Action Form)*

Container Seals: _____ Tape _____ Custody Seals_____(Other specify)
_____ Seals intact for each shipping container
_____ Seal broken (list impacted samples):

Condition of Samples: _____ Sample containers intact
_____ Sample containers broken/leaking (see Sample Custody Corrective Action Form)*

Temperature upon receipt (°C): _____Temperature blank used _____Yes____No
(Note: If temperature upon receipt differs from required conditions, list impacted samples):

Samples Acidified? ___Yes ___No ___Unknown

Initial pH 5 - 9? (Y/N):___ *If no, individual sample adjustments on the Auxiliary Sample Receipt Form.*

Total Residual Chlorine Present? (water) (Y/N): _____
If yes, individual sample adjustments on the Auxiliary Sample Receipt Form.

Head Space <1% in samples for water VOC analysis _____Yes_____No
Individual sample deviations listed below.

Sample Containers:
Samples returned in PC-grade jars? ___Yes ___No ___Unknown / Lot No._____

Storage Location: _____ **BDO IDs Assigned**: _____

Holding Times:	Water -	Sediment-	Tissue-

Additional Comments:
Samples logged in by: _____ Date/Time: _____
* Must also be noted on the C-O-C.

ATTACHMENT 4b

Battelle Duxbury Operations
Auxiliary Sample Receipt Form

Sample ID	pH initial	pH Final	Vol. Added Acid/Base	Initial TRC	Final TRC	Vol. Added Sodium Thiosulfate

Work Perform by/Entered by _____ **Date**_____

ATTACHMENT 5
Battelle Duxbury Operations
Example Daily Sample Tracking Form[1]

BATTELLE - DUXBURY OPERATIONS

DAILY SAMPLE TRACKING PAGE

Samples Relinquished by Custodian :	Date :	Time:
Location from which retrieved :	Date :	Time:
Samples received for sample prep by :	Date :	Time:
Storage until prep initiated :	Date :	Time:
Samples Returned to Custodian :	Date :	Time:
Location Stored :	Date :	Time:

This Batch Contains The Following Samples:

Date/Time Extracts Removed :	Initials:	Location removed from:
Date/Time Extracts Returned :	Initials:	Storage Location:
Date/Time Extracts Removed :	Initials:	Location removed from:
Date/Time Extracts Returned :	Initials:	Storage Location:
Date/Time Extracts Removed :	Initials:	Location removed from:
Date/Time Extracts Returned :	Initials:	Storage Location:
Date/Time Extracts Removed :	Initials:	Location removed from:
Date/Time Extracts Returned :	Initials:	Storage Location:

VALIDATION CHECKLIST

Check sample custody records to verify client Ids	
Verify all samples in batch are reported and all samples reported are in the prep batch	
Complete surrogate and internal standard spikng forms	
Check sample dilution, grav weights, and correction factors	
Check HPLC tables 100%	
Check dry weight and lipid weight tables	
Ensure sample transfer and documented by proper sign-off	

[1] Separate forms should be initiated for each sample prep batch and for each analysis.
If sample is not consumed, document the storage of the remainder.

ATTACHMENT 6

Battelle Duxbury Operation
Sample Split and Transfer Log

Battelle
. . . Putting Technology To Work

Sample Split and Transfer Log

Project Number _____ Date of Work _____

Project Title _____

Analysis Type(s) _____

Splitting Procedure _____

_____

(include description of amount or weight of split, packaging, storage)

Name _____ Date _____

Sample ID	Sample ID	Sample ID	Sample ID	Sample ID	Sample ID

Released	Received
Signature/Date:	Signature/Date:
Storage Location/Conditions:	Storage Location/Conditions:
Released	**Received**
Signature/Date:	Signature/Date:
Storage Location/Conditions:	Storage Location/Conditions:

ATTACHMENT 7
Battelle Duxbury Operations
Chain-of-custody Record

Battelle
. . . Putting Technology To Work

Chain of Custody

Proj. No	Proj. Name

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

Relinquished by:	Date/Time	Received by:	Date/Time
Relinquished by:	Date/Time	Received by:	Date/Time

Comments:

ORIGINAL

ATTACHMENT 8
Battelle Duxbury Operations
Certificate of Training

SOP No. 6-010

SOP Title: Chemistry Laboratory Sample Control

Trainee_____

Instructor_____

SOP Read:_____ _____
Signature Date

Date Training Completed:_____

The above mentioned trainee has satisfactorily completed the training requirements associated with this SOP. Supporting documentation (if applicable) is attached.

Comments:

Approved By/Date: _____

APPENDIX A

Quality Assurance Project Plan

FINAL

THAMES RIVER
RAPID SEDIMENT CHARACTERIZATION PILOT STUDY
NAVAL SUBMARINE BASE - NEW LONDON
GROTON, CONNECTICUT

QUALITY ASSURANCE PROJECT PLAN
(Version 1)

Prepared for:

DEPARTMENT OF THE NAVY
NAVAL FACILITIES ENGINEERING COMMAND
NORTHERN DIVISION
10 Industrial Highway
Mail Stop, #82
Lester, PA 19113-2090

Prepared by:

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Fairfax Station, VA 22039

Contract No. N62472-00-D-1300

Task Order 0005

Project No. G486305

June 12, 2003

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FINAL

THAMES RIVER RAPID SEDIMENT CHARACTERIZATION PILOT STUDY

QUALITY ASSURANCE PROJECT PLAN

Prepared by:

BATTELLE

NEPTUNE AND COMPANY

June 2003

REVIEW AND APPROVALS

Project Manager, Battelle Don Gunster	Donald Gunster	6/12/03
Technical Advisor, Battelle Donald Gunster	Patty White	6/12/03
Project QA Manager, Battelle Rosanna Buhl	Rosanna Buhl	6-12-03
Field Manager, Battelle Alex Mansfield	Robert E Heath for AM	6/12/03
Navy Project Manager: Mark Evans	M A Evans	6/11/03
Navy QA Manager: Deb Felton	Deborah L. Felton	6/11/2003
	Name	Date

DISTRIBUTION LIST

This document will be distributed to the following project participants once all approval signatures have been received:

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Jim Leather	
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Dean Neptune	
Carole Peven	
Jennyfer Smith	
Liz Vonckx	
Patty White	

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FIGURE

Figure A-1. Organizational Structure for the Thames River Rapid Sediment Characterization Pilot Study. A-10

ATTACHMENT

Attachment A: Analytical Documentation Severn-Trent Laboratory – Burlington

ACRONYMS AND ABBREVIATIONS

BDO	– Battelle Duxbury Operations
BSL	– Battelle Sequim Laboratory
CFR	– Code of Federal Regulations
CLP	– Contract laboratory program
cm	– Centimeter
COPEC	– Contaminant of potential environmental concern
CVAA	– Cold vapor atomic absorption
CVAF	– Cold vapor atomic fluorescence
DGPS	– Differentially-corrected global positioning system
DOD	– Department of Defense
DQA	– Data quality assessment
DQO	– Data quality objective
ECD	– Electron-capture detector
EDD	– Electronic data deliverable
EPA	– U.S. Environmental Protection Agency
FSP	– Field sampling plan
g	– Gram
GC	– Gas chromatography
GC/ECD	– Gas chromatography/ Electron-capture detector
GC/MS	– Gas chromatograph/mass spectrometer
GFAA	– Graphite furnace atomic absorption
HAZWOPER	– Hazardous waste operations and emergency response
HASP	– Health and Safety Plan
ICP	– Inductively coupled plasma
ICP-AES	– Inductively coupled plasma-Atomic Emission Spectroscopy
ICP-MS	– Inductively coupled plasma-mass spectroscopy
ID	– Identification
LCS	– Laboratory control sample
LCSD	– Laboratory control sample duplicate
MB	– Method or procedural blank
MDL	– Method detection limit
mg/kg	– Milligrams per kilogram
MQC	– Measurement Quality Criteria
MS	– Matrix spike
MSL	– Marine Sciences Laboratory (*i.e*, BSL)
MSD	– Matrix spike duplicate
NA	– Not applicable
NEDTS	– National Environmental Data Transfer Standards
NFESC	– Naval Facilities Engineering Service Center
NIST	– National Institute of Standards and Technology
NOAA	– National Oceanic and Atmospheric Administration
NPL	– National priorities list
NS&T	– National Status and Trends

PAH	– Polycyclic aromatic hydrocarbon
PCB	– Polychlorinated biphenyl
PD	– Percent difference
PE	– Performance evaluation
PFTBA	– Perfluorotributylamine
PT	– Performance test
QA	– Quality assurance
QADU	– Quality assurance (laboratory) duplicate sample
QAPP	– Quality assurance project plan
QC	– Quality control
QL	– Quantitation limit
RF	– Response factor
RI	– Remedial investigation
RIS	– Recovery internal standard
RPD	– Relative percent difference
RSC	– Rapid Sediment Characterization
RSD	– Relative standard deviation
SA	– Selective availability
SIM	– Selective Ion Monitoring
SIS	– Surrogate internal standard
SOP	– Standard operating procedure
SRM	– Standard reference material
SSC SD	– SPAWAR Systems Center - San Diego
TIC	– Tentatively identified compound
WP/FSP	– Work Plan/Field Sampling Plan
XRF	– X-Ray Fluorescence

A.1.0 INTRODUCTION

The purpose of this Quality Assurance Project Plan (QAPP) is to describe the sampling, analysis, and reporting that will be conducted by Battelle at the Naval Submarine Base New London (NSB-NLON) in Connecticut. The current work plan and this QAPP are prepared for the Naval Facilities Engineering Command Northern Division (NORTHDIV) under Contract No. N62472-00-D-1300 to describe the Rapid Sediment Characterization (RSC) Pilot Study that will be conducted in June 2003. The organization of this QAPP follows the format defined by EPA QA/R-5 (2001). Table A-1 provides a cross-walk between the organization of this document and the requirements of EPA Region I. All tables are located after the text in Section A.6.

The objective of this project is to assist the Navy in the evaluation of data gaps in specific areas of the Thames River adjacent to NSB-NLON that were identified during the Lower Subase Remedial Investigation (RI) (Brown and Root, 1999). Additionally, the project is being initiated to assist the Navy in additional investigation of sediment in the Pier 1 Marine Railway area that were found to contain elevated levels of polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), and metals during sampling completed in October 1999 and documented in SAIC (2000). The primary objectives of this project are to:

- Review historical data;
- Conduct a pilot study to support the development of data quality objectives (DQOs) and a Work Plan; and
- Develop DQOs and associated Work Plan to complete an ecological screening and refinement evaluation for Zones 4 and 7 and a preliminary investigation to better characterize sediment contaminants and support a Phase I ecological screen for Pier 1.

This QAPP focuses on Task 2, conduct of a RSC Pilot Study at NSB-NLON. The project will utilize the U.S. Navy's Space and Naval Warfare (SPAWAR) Systems Center in San Diego, California (SSC SD) to conduct fast and accurate screening analysis. A subset of these samples will also be analyzed for a full suite of chemical analysis using National Oceanic and Atmospheric Administration (NOAA) Status and Trends methods to provide confirmation data of the rapid screening analysis.

A combined Work Plan/Field Sampling Plan (WP/FSP) describes the overall study objectives and field activities. This Quality Assurance Project Plan (QAPP) is Appendix A of the WP/FSP; it defines Quality Assurance and Quality Control requirements for the project. A Health and Safety Plan (HASP) that defines the potential risks associated with sampling at the New London base is distributed as a separate document.

A site visit and kick-off meeting were conducted by the Battelle project and field managers on January 23, 2003. The final WP/FSP, QAPP, and HASP address both Navy and agency review comments Field work will be completed by June 20, 2003 and a rough draft report of the results of the pilot study provided to the Navy Project Manager by October 27, 2003. Based on the Agency review of the results, a draft investigation/validation study work plan will be provided to the Navy Project Manager by December 31, 2003 and finalized after agency review by March 29, 2004.

A.2.0 PROJECT MANAGEMENT

A.2.1 Project and Task Organization

Figure A-1 presents the organizational structure of the Thames River RSC Pilot Study.

Mr. Mark Evans is the Navy Project Manager. He is responsible for providing final approval for conducting all field activities, directing the technical team, and approving selected subcontractors, executing contracts, and approving the release of study reports. He is responsible for the implementation of the field and analytical activities associated with the Thames River RSC Pilot Study.

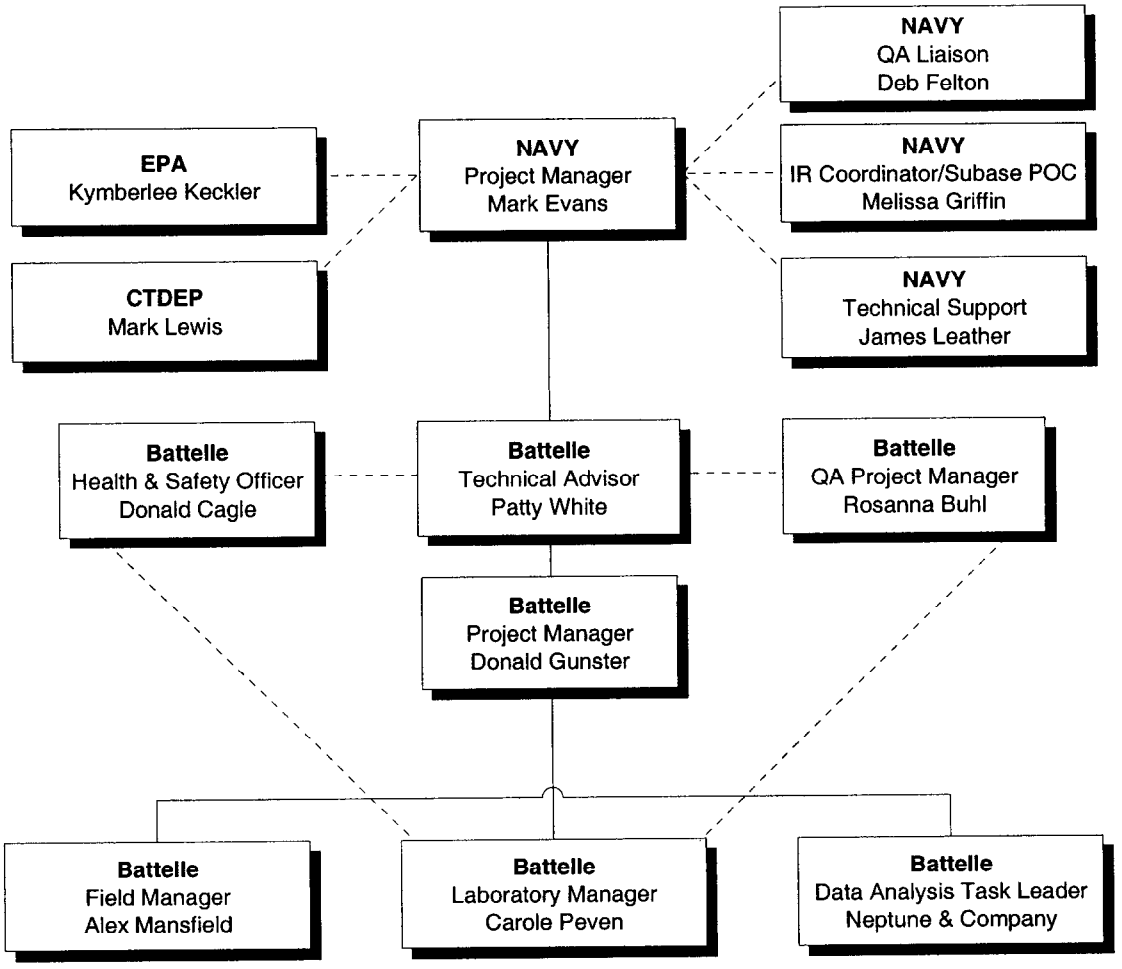

Figure A-1. Organizational Structure for the Thames River Rapid Sediment Characterization Pilot Study.

Ms. Deb Felton is the Navy QA Officer for this project. In that role, she is responsible for QA oversight of the Thames River RSC Pilot Study. Her responsibilities include review and approval of the QAPP and FSP for completeness, consistency, and adequate quality control; review of the design process to ensure that it is complete, technically sound, and well-documented; ensuring that all contractors are certified for the work being performed; communicating with the Battelle Project QA Manager and identifying

programmatic issues; reviewing the results of data validation and addressing issues that could compromise the project; and communicating issues to the Navy Project Manager and the Project QA Manager. Ms. Felton performs an independent QA function and is authorized to suspend field activities if Navy QA requirements are not met.

The roles and responsibilities of EPA and state regulators associated with this project are defined by their respective agencies.

The Battelle Thames River RSC Pilot Study Team consists of the Project Manager, Health and Safety Officer, QA Manager, Field Manager, and Laboratory Manager.

Dr. Gunster is the Battelle Project Manager. He coordinates technical activities as a liaison between the Navy Project Manager, and the Field and Chemistry Laboratory Managers. He communicates directly with the project team to coordinate activities, enforces schedules and deadlines, and ensures that all decisions that impact field or laboratory activities are dispatched in real time. He is responsible for ensuring that responses to QA reports are adequate and that corrective action is implemented, as needed. He is responsible for preparation and review of the Thames River validation/investigation work plan, and final deliverables. He reports to and coordinates Thames River RSC Pilot Study activities with the Navy Project Manager. He communicates directly with the Project Health and Safety Officer, Project QA Manager, and the Project Manager and reports program status to, and implements the directives of, the Navy Project Manager. He is authorized to stop work for cause if data quality or staff safety are threatened.

Mr. Donald Gunster will also serve as the technical advisor for this project. He provides expertise in validation/investigation and ecological risk assessment activities of this project. He coordinates program-level activities for Navy projects conducted at Battelle Duxbury Operations.

Ms. Heather Kitchen is the Project Health and Safety Officer. She is responsible for reviewing the Thames River RSC Pilot Study Health and Safety Plan, ensuring that the field personnel have received appropriate health and safety training for work at the NSB-NLON, and that the training is documented. She reports issues and concerns directly to the Project Manager and has the authority to stop work.

Ms. Rosanna Buhl is the Project QA Manager. She is responsible for ensuring that the QA systems required by the Navy for laboratories performing work under the Installation Restoration Guidelines are adequately addressed in QA documents that describe project activities: the QAPP, the FSP, and Standard Operating Procedures (SOPs). She prepares the QAPP and must approve the final version. She conducts project reviews, monitors corrective action, and reports the results of these oversight activities to the Project Manager. She is the point of contact with the data validation firm. She is authorized to stop work for cause if data quality or staff safety is threatened.

Mr. Alex Mansfield is the Project Field Manager. He is responsible for coordinating field logistics, providing the FSP to the field crew, conducting a kick-off meeting prior to sampling activities, and ensuring that the field team is adequately trained in field sampling procedures. He is responsible for ensuring that all technical logistics are identified and addressed and for arranging access to the naval facility, scheduling the sampling trip, arranging for equipment and vessels, and escorts, where required. He verifies that field equipment and instruments have been adequately maintained and tested, and that appropriate calibration and decontamination between sites and samples is conducted and documented. Mr. Mansfield is responsible for ensuring that samples are collected, handled, preserved, and shipped as specified in the FSP and QAPP, and that documentation is detailed, accurate, and legally defensible. He is responsible for ensuring that samples are collected and handled under custody. He reports to the Project Manager.

Ms. Carole Peven-McCarthy is the Project Laboratory Manager. She is responsible for ensuring that analytical procedures are performed as defined in the QAPP, and for providing technical expertise to the analytical laboratories. She is responsible for performing a management review of analytical data reports to ensure that the data, including quality control data, are acceptable. She ensures that the status of laboratory analyses and potential problems are reported to the Project Manager.

The field team is responsible for conducting all field activities according to the QAPP and FSP and for communicating problems to the Project Field Manager.

The analytical laboratories are responsible for conducting all analytical activities according to the NFESC (1999), the Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM, 2000), the QAPP, and the WP/FSP. Laboratories are responsible for maintaining sample custody records throughout processing and analysis, conducting analysis according to specified SOPs, reviewing QC data and implementing corrective action, as appropriate, and contacting the Project Laboratory Manager to communicate any issues that could affect sample integrity, data quality, or schedule.

The SSC SD is responsible for conducting all rapid sediment screening analyses for this study according to internal procedures established at this facility.

Each laboratory is responsible for appointing an independent QA Officer who will monitor the study, conduct laboratory inspections and data audits, and report findings to management. Ms. Rosanna Buhl and Ms. Deborah Coffey are the Laboratory QA Officers at Battelle's Duxbury Operations (BDO) and Battelle's Sequim Laboratory (BSL), respectively. The SSC SD is responsible for implementing internal QA policies at the SSC SD laboratory for this project.

A.2.2 Problem Definition/Background

NSB-NLON is located along the Thames River and lies within the towns of Ledyard and Groton, CT. The Lower Subase is a narrow strip of land that generally forms the western boundary of NSB-NLON and parallels the Thames River. During RI activities completed in the Lower Subase, the seven offshore areas were assessed for risk using a weight-of-evidence approach. Data gaps that need to be filled in order to proceed to the Feasibility Study were identified at two of these zones (Zone 4 & 7). The need for further investigation at another site, the Pier 1 Marine Railway, was identified when a small-scale study showed exceedences of benchmarks for metals, PAHs and PCBs. The current Thames River RSC Pilot Study will assess sediment contaminant levels at these three stations.

A.2.3 Project/Task Description

The primary objective of the Thames River RSC Pilot Study is to supplement historic data for the development of DQOs and an associated Work Plan in support of a validation/investigation study.

Surface sediment samples will be screened by the SSC SD laboratory (San Diego, CA) for selected trace metals, total PAHs, and total PCBs using semi-quantitative analytical techniques. RSC sample results will be used to develop maps showing the distribution of the selected contaminants in surface sediments in the areas of interest. Confirmation analysis using quantitative, low-level laboratory techniques will be performed on 37 of these samples to verify the accuracy of the screening results. The confirmatory laboratory data will be used to verify the RSC results and also will be used in a screening-level risk assessment with site-specific exposure parameters to determine whether a baseline ecological risk assessment is warranted.

Once data collection is complete the assessment tasks will include compilation of data and, preparation of a comprehensive work plan.

A.2.4 Quality Objectives and Criteria for Measurement Data

A.2.4.1 Data Quality Objectives

DQOs are defined using a systematic planning process that defines the quality objectives and the performance criteria. The sampling design is a product of the DQOs. For the Thames River RSC Pilot Study, the sampling and analysis design is intended to provide an overview of sediment COPEC concentrations. The DQOs are tabulated in the project WP/FSP to which this QAPP is appended. The consequences of making an incorrect decision are addressed as part of the DQOs. Based on this analysis of consequences, the QAPP defines the project quality objectives in quantitative terms.

Potential data quality concerns result primarily from incorrect interpretation of the analytical results. Based on the proposed use of the data these concerns include, but are not limited to:

- collecting an adequate number of samples to characterize the areas of interest (the number of samples could be inadequate, for example, if measurement or sampling variability exceeds expectations);
- limiting handling-related contamination of samples to insignificant levels; and,
- maintaining an acceptable level of data quality to allow for statistically valid evaluation or pooling of these data within each area of interest (Zones 4 and 7, Pier 1 and the reference area) to obtain spatial estimates of chemical constituent variability.

The DQO planning process resulted in a WP/FSP and QAPP that addresses these data quality concerns.

A.2.4.2 Measurement Quality Objectives

Quantitative measurement quality objectives can be expressed in terms of accuracy, precision, completeness, and sensitivity goals. Qualitative quality objectives are expressed in terms of comparability, and representativeness.

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 monitored through the analysis of quality control samples (Section A.3.5).

Precision is defined as degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves. Precision is usually expressed as standard deviation, variance, or range, in either absolute or relative terms. Precision is monitored through the analysis of quality control samples (Section A.3.5).

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%. For the reference area, 100% of samples are needed to meet the DQOs specified for this investigation. However, the project will not be compromised if 90% of the other field samples are collected and analyzed with acceptable quality. Completeness is calculated by the project manager.

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). The results of rapid sediment screening analysis conducted by SSC SD will be comparable with other rapid sediment screening data. Confirmation data will be used for risk assessment and are not intended to be comparable with past analyses that used traditional EPA Methods.

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 sample 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.

Sensitivity is the capability of a test method or instrument to discriminate between measurement responses representing different levels (*e.g.*, concentrations) of a variable of interest. Sensitivity is addressed primarily through the selection of appropriate analytical methods, equipment, and instrumentation. The NOAA Status and Trends analytical methods selected for the Thames River RSC Pilot Study were chosen to provide the sensitivity required for the end-use of the data. Sensitivity is monitored through the determination of method and reporting limits, instrument calibration, calibration verification, and the analysis of procedural blanks with every analytical batch.

Method Detection Limits (MDLs) for organic compounds in sediments are determined according to 40 CFR Part 136 Appendix B by spiking clean sediment or a solid matrix such as prebaked sodium sulfate with all parameters of interest and processing them according to the methods defined in Section A.3.4. These MDLs are checked on a quarterly basis. MDLs for Gas chromatography/Electron-capture detector (GC/ECD) analysis are determined on the primary column. MDLs for PCBs and pesticides must also be determined on a confirmation column if data from confirmatory analyses will be reported. In these instances, the MDLs determined from confirmation column analysis must be less than those determined from the primary column. Quantification on confirmation columns is not, however, anticipated for this investigation.

MDLs for trace metals are determined annually according to 40 CFR Part 136 Appendix B. MDLs for sediment samples are performed using clean quartz sand as the matrix. If metal-free matrices for sediments are not available, MDLs for metals in sediment samples are generated by a fresh water MDL study converted to concentration per weight using a representative sample mass and volume. MDLs for fresh water samples are determined by spiking deionized water with all metals of interest and processing them according to the methods defined in Section A.3.4.

For risk assessment purposes, the sample-specific MDL (adjusted for sample size and dilutions) will be inserted into the value field for non-detected chemical parameters with the National Environmental Data Transfer Standards (NEDTS) data qualifier "U."

Reporting Limits (RLs) for organic compounds are empirical values based on instrument sensitivity and day-to-day operations. For organic compounds, the RL is calculated as

RL = (Low Standard Concentration)(Pre-injection volume)(Dilution Factors)(1/ Sample Size)

The reporting limits of two COPECs (dieldrin and endrin) are higher than the benchmark values. The actual reporting limit can be lowered by increasing the sample size and decreasing the pre-injection volume of the sample. Detected values that are less than the reporting limit or for which the target detection limit was not achieved will be qualified as estimates (J) and used with caution during any assessment.

For trace metals, the RL is calculated by multiplying the target analyte MDL by 3.18. The value 3.18 is based on the Student's-t value for 7 replicates, the number of replicates usually analyzed to generate the MDL. If a greater number of replicates are analyzed, the 3.14 value will be replaced according to the appropriate t statistic $_{(n-1)\ (alpha+0.995)}$. The NEDTS data qualifier "J" will be added to any reported values that are less than the sample-specific RL.

The DoD Quality Systems Manual (2000) includes the following note: *There may be numbers reported to the client that are below the reporting limit. These numbers must be flagged appropriately. When the analysis demonstrates a non-detect at the MDL, the data shall be flagged with a "U." The value reported to the client is the MDL, adjusted by any dilution factor used in the analysis. When an analyte is detected between the lower quantitation limit and the MDL, the data shall be flagged with a "J." The value reported is an estimation.*

A.2.5 Special Training/Certification

A.2.5.1 Training Requirements

Documented training is required for each individual performing activities in support of environmental data collection or analysis. Each laboratory technician and analyst must complete an initial demonstration of capability before processing or analyzing samples for this project. At least annually, technicians and analysts must demonstrate continued proficiency for the analyses that they are performing. The procedures used to ensure that staff training is current and documented is defiled in laboratory SOPs. The applicable laboratory manager is responsible for determining specific training and certification needs, and for ensuring that any required training is documented.

Field and data management personnel must have documented experience or direct training in the procedures that they will be performing for this project, including any applicable SOPs.

A.2.5.2 Special Training

Special training and certification required for the Thames River RSC Pilot Study include the following:

- Any field team members involved with sample collection or handling must receive certification of training in hazardous waste handling and emergency response (HAZWOPER – 29 CFR 1910.120) is required for any team members. This is a 40-hour course.
- Field team members must have the 40-hour HAZWOPER course, and, when necessary, the 8-hour refresher training course within the last 12 months.
- The Project Field Manager must complete an additional 8-hour supervisor training course (HAZWOPER – 29 CFR 1910.120).
- Any other safety-related training defined in the project HASP.
- Vessel operators will be experienced and have demonstrable experience in small boat handling under the conditions expected at the site.

A field orientation must be conducted to establish guidelines for field observations to ensure repeatability between members of the field team. This orientation is the responsibility of the Project Field Manager.

A.2.5.3 Navy Certification

Battelle's laboratories in Duxbury, Massachusetts (BDO) and Sequim, Washington (BSL) will be performing non-standard methods (NOAA Status and Trends methods). Both laboratories have obtained Navy approval through NFESC for the methods that will be implemented for this study. Severn-Trent Laboratories (STL) in Burlington will be performing TOC and grain size analysis for this study. Grain size is not a certifiable procedure, although STL routinely performs this procedure for Navy projects; STL certification by NFESC of their TOC in sediment procedure is in progress.

A.2.6 Documentation and Records

A.2.6.1 Document Control

It is critical that project personnel have the most recent versions of the QAPP, WP/FSP, and SOPs. Version control is maintained by defining the version and date on each of these documents. A distribution list is maintained for each controlled document. When a new version is approved, it is distributed and the old versions must be discarded. Version control is maintained and documented through the document header blocks, which identify the document, version, and effective date. Versions of the QAPP prepared for review, include preliminary draft and draft versions, are not assigned a version number. The final QAPP will be identified as "Version 1" with the effective date. If the final QAPP is subsequently revised the header block will identify the updated document as "Version 2."

A.2.6.2 Field Documentation

The field team members will maintain bound, paginated field logbooks to provide a daily record of field activities, observations, and measurements during sampling. Field data, observation, and all information pertinent to sampling will be recorded in the logbooks in real time using activity-specific forms. The WP/FSP defines the specific records and data that must be maintained for each field activity to ensure that samples and data are traceable and defensible. Documentation of sample collection must include both date and time. All field records and documentation must comply with the documentation requirements defined in Section A.2.6.4.

A.2.6.3 Laboratory Documentation

Documentation is critical for tracking data and evaluating the success of any activity. Laboratory documentation requirements are defined in laboratory SOPs. Laboratory documentation will be recorded using activity-specific forms, bound, pre-paginated logbooks, or the laboratory information management system (LIMS). Required documentation includes, but is not limited to, the following:

- Sample receipt and custody records
- Sample processing or preparation procedures such that it is traceable to sample receipt records.
- Calibration and maintenance records for all instruments and equipment involved in the collection of environmental data.
- Preparation of calibration standards, spiking solutions, and dosing solutions such that each unique preparation can be tracked to the original (neat) material.

- Lot numbers for all standards, stock solutions, reagents, and solvents.
- All sample analyses and results of analyses. All rejected data are accompanied by explanations of the failure and the corrective action.
- All data reduction formulas such that reported data is uniquely traceable to raw data.

A.2.6.4 Documentation Standards

Each organization performing activities in support of environmental data collection must have SOPs for all methods and procedures related to the collection, processing, analysis, reporting, and tracking of environmental data. SOPs must be readily available to laboratory personnel. SOPs are controlled documents and, as such, must be approved by management and dated. The laboratory must maintain a master list of SOPs in accordance with Navy Installation Restoration Data Quality Manual requirements. All SOPs that are used for environmental data collection activities must be reviewed annually and updated as needed. Any sampling procedures defined in the WP/FSP by reference to the SOP number or another citation must be included as an Appendix to the WP/FSP.

All data generated during the course of this project must be able to withstand challenges to their validity, accuracy, and legibility. To meet this objective, documentation of all environmental data collection activities must meet the following minimum requirements. Other specific documentation requirements are discussed throughout this QAPP and the associated SOPs:

- Data must be entered directly, promptly, and legibly. All reported data must be uniquely traceable to the raw data. All data reduction formulas must be documented.
- Handwritten data must be recorded in ink. All original data records include, as appropriate, a description of the data collected, units of measurement, unique sample identification (ID) and station or location ID (if applicable), name (signature or initials) of the person collecting the data, and date of data collection.
- Any changes to the original (raw data) entry must not obscure the original entry. The reason for the change must be documented, and the change must be initialed and dated by the person making the change.
- The use of pencil, correction fluid, and erasable pen is prohibited.

Any changes to the QAPP or WP/FSP must be documented and approved. Changes that are anticipated up to 12 hours prior to the intended field or laboratory activities must be documented and submitted to the Project Manager for approval prior to implementation of the changes.

A.2.6.4.1 Changes and Deviations

During the conduct of this study, it may be necessary to modify the planned activities. Modifications that are anticipated prior to field or laboratory work will be reported to the Project Manager, who will assess the potential impact and contact the Navy Project Manager if the changes are major (e.g., those that would impact the study objectives, design, or data quality). All modifications will be described in the final report. The Navy and Battelle Project Managers will determine whether modifications are significant enough to either update the WP/FSP or QAPP or prepare an addendum to those documents. For this study, the final report is the site Investigation/Validation Work Plan.

Changes that are not anticipated prior to the planned activities are deviations and must be communicated to the Project Manager as soon as possible, documented, and submitted for approval to the Project Manager. Documentation should include an assessment of any impact that the deviation has on data quality and the corrective action. Minor deviations (*e.g.*, those that would not impact the study objectives, design, or data quality) will be reported to and approved by the appropriate team manager and the Project Manager. Major deviations (*e.g.*, those that could impact the study objectives, design, or data quality) will additionally be reported to the Project Manager and the Project QA Manager. A discussion of major deviations and potential impact on the project objectives will be included in the final report.

A.2.6.4.2 Definition of Raw Data

Raw data are defined as any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (and verified accurate by signature) then the exact copy or exact transcript may be substituted (NELAC Chapter 1 Glossary June 2000). Raw data will be archived at each participating laboratory for 5 years from the date of the final report.

A.2.6.5 Contents of Data Packages

The analytical laboratories that are performing confirmatory (quantitative) chemistry analysis (BDO, BSL, STL-Burlington) will provide the prime contractor with full data packages, which contain all information required for validation. (Section A.5.0 discusses data validation requirements). All data packages must contain any of the following elements that are applicable to the analysis because the data will be validated:

- Title page;
- Table of contents;
- Data package narrative (contents defined in Navy Installation Restoration Data Quality Manual) and this QAPP;
- Final data report tables (see Section A.2.6.7 for contents);
- Analytical records:
  - Instrument tuning (GC/MS methods);
  - Degradation control (pesticide analyses);
  - Retention times (GC methods);
  - Calibration data;
  - Calibration verifications;
  - Mass spectra for all detected GC/MS samples selected for Level IV validation;
  - Surrogate recoveries (GC/MS and GC methods);
  - Internal standard response and retention times; and

  - All QC data required by the analytical method or the QAPP (blanks, laboratory control samples (LCS)/LSC duplicates (LCSD), matrix spike samples (MS)/MS duplicates (MSD), duplicates);
- Required supporting information:
  - Entire package of sample custody documentation, including sample receipt form;
  - Sample processing and spiking records;
  - Copies of standard preparation logs for each standard used in sample preparation and instrument calibration;
  - Run logs (see Navy Installation Restoration Data Quality Manual for specific requirements);
  - Raw data associated with field and QC data;
  - Chromatograms;
  - Instrument calibration records and calibration results;
  - Results of all QC samples required by the QAPP; matrix spike solution compounds in concentration units;
  - Sources of control limits for surrogates and LCS; and
  - Source of LCS;
- Summary of internal standard retention times and response;
- Description of manual integration procedures; and,
- List of current method detection limits (MDLs) for the preparation and analysis methods used for sample processing.

The summary data packages for analytical chemistry must contain all information required for independent validation.

A.2.6.6 Reports

Data reports, consisting of a QC narrative and summary data tables, will be generated once the internal data review process is satisfactorily completed. The Laboratory Manager at each analytical laboratory is responsible for preparing these reports. Data reports must include the following:

  - Complete field sample identification;
  - Sample identification numbers assigned by the laboratory;
  - Dates of sample collection, processing, and analysis;
  - Sample matrix;
  - Analytical SOP number and base EPA method (if applicable);
  - Results (with clearly defined concentration units) for each targeted analyte;
  - Electronic file identification codes (when applicable, identify instrument data files);
  - Data qualifying flags;
  - Dilution factor(s);

- Method detection Limits;
- Reporting Limits;
- Date of report; and,
- Review date and signature of the laboratory manager.

A comprehensive work plan will include the results of the field survey and a summary of analytical results. In addition to the data report requirements defined above and in Section A.3.10.2, the work plan will summarize the field sampling activities. Information will include

- a chronology of events;
- a table of field statistics (date/time, depth, salinity, porewater ammonia, and coordinates of each station);
- a table of samples collected, together with location coordinates, date/times, sample IDs, amount of sample and intended analyses; and,
- a summary of problems encountered, deviations, and corrective actions.

A.2.6.7 Data Storage and Disposal

All electronic and hardcopy raw data, data packages, and final data will be retained by the laboratory for a minimum of 5 years after final data submittal. If raw data will be stored on tape or CD then the magnetic tape storage device or other similar storage device must be capable of recording data for long-term, off-line storage. Battelle is responsible for maintaining data generated for this project for 5 years. At the end of that period Battelle will contact the Navy RPM or their representative to determine if Navy wants to take possession of the data. If so, Battelle submits that data, including electronic data, to the Navy. The Navy is responsible for maintaining that data in accordance with their prescribed time requirements. Sample archival and disposal is discussed in Section A.3.3.4.

A.3.0 DATA GENERATION AND ACQUISITION

This section describes the method requirements for all aspects of data measurement and acquisition for collection, handling, and analysis of samples, quality control procedures and requirements, and data management. The procedures described in this section are selected to ensure that data of the appropriate type and quality are collected in support of the Thames River RSC Pilot Study.

A.3.1 Sampling Process Design (Experimental Design)

The DQOs, sampling design, station locations, and associated maps are provided in the WP/FSP. Table A-2 lists the COPECs for the Thames River RSC Pilot Study. Table A-3 defines critical vs. non-critical measurements. Table A-4 contains a list of all standard operating procedures that apply to this study. The Thames River RSC Pilot Study tasks include:

- Collection of sediment samples at 58 Subase stations;
- Collection of 4 sediment samples at each of two references stations;
- Collection of 7 field duplicate samples;
- A qualitative measurement of porewater ammonia concentrations will be obtained from each sample using Hach™ test strips;
- Rapid Sediment Characterization (RSC) analysis of 58 samples using XRF (Cu, Pb, Zn, Cr and As) analyses for trace metals, and immunoassay procedures for total PCBs and total PAHs;
- Archival of all sediment samples for potential quantitative analysis for organics compounds (pesticides, PCBs, PAHs), and trace metals;
- Confirmatory analysis of 37 samples that span the range of concentrations detected during RSC, including all samples collected from the reference stations; and,
- Physical characterization of laboratory confirmation samples for percent moisture, grain size and total organic carbon content.

A.3.2 Sampling Methods

To perform analytical tests, sediment samples must be collected according to standard protocols. The following field procedures are relevant for this study, which are detailed in the WP/FSP:

- Station coordinates (latitude and longitude) will be determined at each sampling location using a Trimble 4000 RS DGPS receiver interfaced with an Ocean Survey Inc. (OSI) Maretrack Navigation and Data Logging System.
- Station depths will be measured using a lead line.
- Porewater ammonia will be measured using a Hach®Kit.
- Surface salinity will be measured using a Refractometer.
- A Ponar grab sampler or Van Veen grab sampler will be used to collect undisturbed sediment to a penetration depth of up to 10 cm.

- The top 5 cm will then be collected for analysis. The sample will be homogenized in the field and aliquotted between three sample jars for RSC techniques and archival for potential confirmatory analyses and physical characterization.
- Field duplicate samples will be collected at the rate of 1:10 field samples (7 duplicates). The location of the duplicates is defined in the WP/FSP.

Table A-5 summarizes the container type, required sample volumes, and preservation requirements for all sample analyses, as well as the maximum holding times to sample extraction and analysis, as necessary.

If sampling requirements cannot be met due to sampling or measurement system failure, field conditions or other factors that cannot be controlled, the Project Manager will be contacted. A corrective action will be agreed upon based on the critical/non-critical nature of the problem, documented in the field log, and communicated to the sampling team. In general, if critical measurements or samples cannot be collected, then sampling will be re-scheduled. If a non-critical measurement or sample cannot be collected, then the deviation will be documented. The Project QA Manager will review corrective actions to assess their effectiveness.

A.3.3 Sample Handling and Custody

Sample handling and custody requirements are designed to ensure sample integrity and traceability. All field procedures, including sampling, decontamination, and handling, are detailed in the WP/FSP. Labeling requirements are defined in Section A.3.3.2.

A.3.3.1 Sample Processing

Sample collection at the NSB-NLON will be coordinated by Battelle with in-field assistance from a SSC SD staff member and an OSI boat operator. Sample collection procedures are detailed in the WP/FSP.

Field personnel should wear Nitrile® gloves during sample collection and polyethylene or latex gloves during handling activities. Gloves should be changed between stations. Gasoline or diesel motors (boat motors and gas-powered pumps) should be shut down at least one minute prior to sampling activities to eliminate the potential for contamination by exhaust gases. If boats cannot be shut down then they must be positioned to minimize the affects of exhaust fumes.

Approximately 1000 g of sediment are required from each station for screening and lab analyses. The upper 5 cm of sediment will be removed from the sampler with a stainless steel spoon and placed in a clean stainless steel bowl. Multiple grabs may be required at each station in order to achieve the required sample volume. The sediment will be mixed in the bowl using a clean stainless steel spoon until a homogeneous color and texture is achieved. The homogenized sediment from each station will be split into three labeled sample jars: 20 g in a 4 oz glass jar with a Teflon-lined cap for rapid sediment screening analysis at SSC SD and the remainder in two 16 oz glass jars with Teflon-lined caps for archiving and potential laboratory analysis. Samples will be placed on ice immediately after sampling, and maintained on ice throughout the field holding period. Temperatures will be monitored daily and recorded in a bound logbook throughout storage.

Samples will be hand-carried or shipped by the field sampling team to the destination laboratory by overnight courier in coolers with ice as follows:

The 4 oz glass sediment jars targeted for rapid sediment screening analyses will either be hand-carried to the laboratory by Mr. Jim Leather, or will be shipped from the field at the end of the survey to:

Mr. Jim Leather
SSC SD D362
53475 Strothe Road
San Diego, CA 92152
(619) 553-6240

Attention: Jim Leather

The two 16–oz sediment jars for archiving and potential laboratory confirmation analysis will either be hand-carried to the laboratory by Mr. Alex Mansfield or shipped from the field to the Battelle Duxbury Operations sample custodian:

Ms. Jessica Fahey (Custodian)
Battelle Duxbury Operations
397 Washington Street
Duxbury, MA 02332
(781) 952-5270

Attention: Carole Peven-McCarthy

Upon arrive at BDO, samples will be frozen. Thirty seven (37) of the sediment samples will undergo full laboratory (confirmatory) analysis. These samples will be selected based on the screening results (WP/FSP Section 2.0). The samples selected for analysis will be thawed, homogenized, split by laboratory staff at Battelle Duxbury Operations and distributed as follows.

Sediment in one 16-oz jar will be homogenized and split for organic compound analysis and trace metals analysis. The aliquots intended for organic compound analysis will be retained at BDO. The aliquots intended for trace metals will be shipped to the BSL sample custodian:

Ms. Carolynn Suslick (Custodian)
Battelle Marine Sciences Laboratory
1529 West Sequim Bay Road
Sequim, WA 98382
(360) 681-3624

Attention: Jill Brandenburg

Sediment in the second 16-oz jar, intended for TOC and grain size analysis, will be shipped to:

Mr. Frank Bessette (Custodian)
Severn-Trent Laboratory – Burlington
55 South Park Drive
Colchester, VT 05480
(802) 655-1203

Attention: Kirk Young

Separate aliquots are not collected for percent moisture determination. Each analytical chemistry laboratory will determine the percent moisture of the sample aliquot that they received, and will use that value to determine dry weight concentration data.

A.3.3.2 Sample Custody

Sample custody 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. The custody record also serves as a record of the samples collected and analyses requested. SOPs 6-010 and MSL-A-002 define field and laboratory custody procedures.

Sample containers will be labeled with waterproof, adhesive-back labels, and reinforced with clear packing tape. Sample labels must provide sufficient detail to uniquely identify each sediment sample and allow tracking to field activities. Sample identification numbers will be in the format

CAB-001

Where,

C is the year (2003)
AB is the survey ID
001 is a unique, sequential number

Sample labels must include a unique sample identification number, sampling area, collection date, and container number and total number of containers (e.g., 1 of 2; 2 of 2), and sample collector's name. The unique sample ID at each station is comprised of the unique sample ID and the container number, allowing the data from each sample to be easily tracked to the station. The custody forms will include the jar numbers for tracking purposes. Jar 1 will always be the 4-oz jar for RSC. Jars 2 and 3 will always be the samples targeted for archival and potential confirmation.
An example is provided below.

Survey ID: Thames River RSC Pilot Study 2003
Area (circle one):
Zone 4, Zone 7, Pier I, Ref Area 1, Ref Area 2
Unique Sample ID: CAB-_____
Sample Description (circle one):
RSC, or Archive/Confirmation
Date: _____ **Time:** _____
Sample Collector:_____
Container ___ of 3

Sample custody will be documented throughout the life of the sample. Samples should not be left unattended unless properly secured. Each laboratory 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.

The custody form summarizes the samples collected and analyses requested. The custody form tracks sample release from the field to the initial receiving laboratory. If the receiving laboratory performs compositing or initial processing that creates a "new" sample then a new custody form with "new" sample numbers is generated. Each sample custody form will be signed by the person relinquishing samples once that person has verified that the custody form is accurate; i.e., that all samples present in the shipping container are listed on the form, and that the sample descriptions, requested analytical methods, and sampling dates are accurate. The original sample custody forms accompany the samples; the shipper will

keep a copy. Upon receipt at the sample destination, sample custody forms will be signed by the person receiving the samples once that person has verified that all samples identified on the custody forms are present in the shipping container. Any discrepancies will be noted on the form (in addition to any internal laboratory documentation policy) and the sample receiver will immediately contact the project manager to report missing, broken, or compromised samples. The original sample custody forms must be submitted to Battelle by each analytical laboratory with the hard copy data packages.

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.

Occasionally, multiple coolers of samples will be sent in one shipment to the laboratory. Each cooler will contain a separate custody record for the samples in that cooler. In addition, the outside of the cooler will be marked to indicate the cooler number and the number of coolers in the shipment (e.g., 1 of 2, 2 of 2). All coolers must be shipped under a bill of lading that identifies the total number of coolers in the shipment. Separate tracking numbers will be assigned to each cooler.

A.3.3.3 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 custody 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 Project Field Manager for resolution of the problem. The discrepancy, its resolution, and the identity of the person contacted must be documented in the project file. The following conditions may cause sample data to be unusable and must be communicated to the laboratory team leader:

- 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; or,
- Required sample temperatures were not maintained during transport.

The custodian must verify that sample conditions, amounts, and containers meet the requirements for the sample and matrix (Table A-5). A unique sample identifier must be assigned to each sample container received at the laboratory, including multiple containers of the same sample.

A.3.3.4 Sample Handling

Sample holding conditions and holding times are defined in Table A-5. Holding times are calculated from the time of sample collection. Documentation must be sufficient to track sample holding, processing, and analysis times to ensure that holding times are met. Samples must be held in a controlled area with limited access. Deviations from the defined storage requirements must be documented and reported with the data even if alternative holding times are requested by the client (not anticipated for this study).

Field samples will be held for six months after delivery of final data; sample extracts and digestates will be held for one month. Disposal records for unextracted samples, extracted samples, sample containers, and sample extracts must be sufficient to provide tracking from collection, through laboratory receipt, to sample disposal.

A.3.4 Analytical Methods

A.3.4.1 Field Measurement

Field analyses performed during the Thames River RSC Pilot Study will be limited to those that directly support station or sample characterization.

- Sampling coordinates (Connecticut State Plane NAD83) will be determined by OSI using DGPS procedures.
- Station depths to the nearest 10 cm will be determined using a lead line.
- Porewater ammonia will be determined using a Hach®Kit.
- Surface water salinity will be measured by refractometer.

A.3.4.2 Physical Characterization

The following protocols will be used to determine physical/chemical properties of the sediment.

- *TOC sampling and analysis* is used to determine how much total organic carbon is in the sediment. TOC will be analyzed in each of the 37 of the samples targeted for quantitative analysis.
- *Grain size sampling and analysis* is used to describe the distribution of particle sizes in samples of sediment. Grain size influences chemical and biological variables, and can, therefore, be used to determine the most acceptable test organism. Grain size varies from coarse (gravel-sand) fractions to fine (silt-clay) fractions. Grains size will be determined in each of the samples targeted for quantitative analysis (37).
- *Percent moisture sampling and analysis* is used to determine the amount of water present in collected sediments. Percent moisture is determined using a well-homogenized aliquot of sediment. Percent moisture will be determined for each analytical aliquot and applied to the data generated at each laboratory.
- *Porewater ammonia* will be analyzed in the field using Hach™ test strips.

A.3.4.3 Laboratory Analyses

Two types of analyses will be conducted for the Thames River RSC Pilot Study: Semi-quantitative RSC analyses and low-level laboratory confirmation quantitative analyses. The 58 sediment samples collected from Zone 4, Zone 7, and Pier 1 will be analyzed at the SSC SD laboratory to determine general levels of selected COPECs. Once the rapid sediment screening is complete, 37 of the samples will be selected for laboratory confirmation using quantitative techniques for the full COPEC list. The COPEC list, with the required reporting limits and detection limits, is provided in Table A-2. Laboratory procedures are defined in Table A-6. COPEC analyses will be performed by BDO, BSL, and STL laboratories.

The Rapid Sediment Screening samples submitted to the analytical laboratory for quantitative analysis should represent the range of concentrations measured using the screening techniques, include samples with significant hits and negative results. Significant hits include those rapid sediment screening samples with high quantitation in strategic areas. Confirmation of negative results is necessary to determine the occurrence of false negatives. Section x.x of the WP/FSP presents the sample selection process for confirmatory analysis.

A.3.4.3.1 Rapid Sediment Screening Analysis

Sixty-five (65) sediment samples, including field duplicates, will be analyzed at the SSC SD using RSC techniques for total PAHs, total PCBs, and trace metals (Cu, Pb, Zn, Cr and As). Total PCBs will be measured by extracting 10 g of sediment with methanol and measured by immunoassay techniques using a modification of EPA Method 4020 (EPA, 1996a). Total PCBs will be measured by extracting 10 g of sediment with methanol and measurement by immunoassay techniques using a modification of EPA Method 4035 (EPA, 1996a). Strategic Diagnostics Inc. Rapid Assay test kits are used for both PCB and PAH analyses.

Sample aliquots for trace metals will be homogenized and analyzed directly by X-Ray Fluorescence (XRF) using a modification of EPA Method 6200 (EPA, 1998). The modification in sample prep includes running the wet sediment samples directly without further drying and grinding. If heterogeneity leads to poor sample precision measurements, more standard sample prep with drying and grinding can be incorporated. A Spectrace Benchtop QuanX XRF is used for this analysis.

The RSC procedures that will be implemented for this study are consistent with the Immunoassay Guidelines for Planning Environmental Projects developed by EPA Region I (1996b). Specifically,

- Immunoassay kits will be stored at the manufacturer's recommended temperatures prior to use and when not in use;
- Kits will be operated within the temperature range specified in the kit instructions;
- Kits will be used within the shelf life defined by the manufacturer.
- Samples with percent moisture of greater than 30% will be decanted, filtered, or centrifuged to reduce the water content to less than 30%.

A.3.4.3.2 Grain Size

The analysis of grain size will be performed by Severn-Trent Laboratories in Burlington using ASTM method D422. Particles greater than 75 μm (gravel to fine sand) are determined by sieving and particles less than 75 μm (silts and clay) are determined by sedimentation using a hydrometer. The STL-Burlington SOP is provided in Attachment A.

A.3.4.3.3 TOC

The analysis of TOC will be performed by Severn-Trent Laboratories in Burlington using the Lloyd Kahn technique defined in Table A-7. The STL-Burlington SOP is provided in Attachment A.

A.3.4.3.4 Low-Level Analysis

BDO will perform the analysis of organic COPECs according to low-level methods developed for the NOAA Status and Trends Program. The concentrations of compounds detected using these quantitative techniques will be compared to the RSC values detected in the same samples to determine the precision and accuracy of the RSC analysis. Laboratory procedures are defined in Table A-6.

Sample cleanup is a critical component of low-level organic compounds analyses; therefore, a variety of cleanup options may be employed to purify the sample extracts. Sample cleanup options are incorporated into the sample processing SOPs; all sample cleanup procedures will be documented. Sample cleanup procedures will be implemented on a batch-wide basis to ensure comparability of results and to assess cleanup effects on QC samples.

Laboratory analyses must be performed using instruments and columns that are capable of achieving the sensitivity and separation to achieve the reporting limits defined for the COPECs.

- Pesticide and PCB parameters are analyzed by GC/ECD, with a confirmatory column to qualitatively verify peak identification.
- Only Pesticide and PCB peaks confirmed on both columns will be considered "hits."
- All GS/MS analyses will utilize the selected ion monitoring (SIM) method.
- Sample data will not be surrogate corrected.
- No data will be blank-corrected.

Manual integrations are also a key element of low-level organic compounds analyses and are implemented routinely for low-level GC and GC/MS data to separate data system baseline integration features from peaks that can be distinguished at greater than 3:1 signal:noise ratio. Manual integration

- will not be used preferentially for QC samples and must not be used to satisfy QC criteria requirements;
- must be identified, and must be signed and dated by the analyst; and,
- must be justified in the final data report and all manually integrated data must be flagged in the raw data.

All GC/ECD chromatographic peaks are manually integrated. This will be documented in the miscellaneous documentation form included with each individual batch.

A.3.4.3.5 Trace Metals

BSL will perform the analysis of low-level trace metals. The specific method will depend on the concentrations of trace metals detected in the field samples. Analyses will proceed from inductively coupled plasma-mass spectroscopy (ICP-MS) and inductively coupled plasma-Atomic Emission Spectroscopy (ICP-AES) to graphite furnace atomic absorption (GFAA) or hydride generation atomic absorption (HGAA) using a flow-injection analysis system (FIAS). Mercury determination in sediments

will be analyzed by cold vapor atomic absorption (CVAA); Mercury determination in the equipment blank (water) will be analyzed by cold vapor atomic fluorescence (CVAF). The reported analysis will be based on the method that achieves a clear detectable signal, or the method that achieves the reporting limit. Laboratory procedures are defined in Table A-6.

A.3.4.3.6 General Requirements

Each laboratory performing COPEC analyses for the Thames River RSC Pilot Study must comply with the certification and training requirements defined in Section A.2.5. In addition, a demonstration of capability must be completed, method accuracy and precision defined, MDLs verified annually, and a descriptive SOP prepared.

A laboratory batch is defined as a group of ≤ 20 field samples of a similar matrix that is processed as a unit with the same reagents and solvents, simultaneously with the required QC samples, and analyzed in the same method sequence. A procedural blank must be analyzed in each analytical sequence. For the purposes of this study, all sediments are considered a "similar" matrix.

Analytical failures must be assessed and corrected. In most cases an analytical failure will stop the flow of work until it is reviewed, the root cause is identified, and corrective action is implemented. Most analytical failures are associated with QC results or instrument performance. Corrective action for these areas is addressed in Sections A.3.5 and A.4.1. Any deviations from the approved methods must be documented and discussed in the report narrative.

Spent samples, solvent, and acid waste will be discarded in the appropriate waste stream according to SOPs and the sample custody requirements defined in Section A.3.3.

A.3.5 Quality Control Requirements

This section defines the quality control (QC) program for the Thames River RSC Pilot Study. Appropriate field and laboratory QC procedures are designated in order to assess data quality through the measures of accuracy and precision. If data fall outside the specified accuracy or precision criteria defined for a procedure or measurement, or if problems affecting comparability are identified, the field or laboratory team leader must contact the Project QA Manager to discuss options available for rectifying the out-of-control situation. The Battelle Project Manager has final authority on decisions made to address problems.

A.3.5.1 Field Sampling

QC for sample collection is designed to support estimates of total measurement system precision and bias by media and analyte. For this study, field assessment sampling is integrated in the WP/FSP and is designed to assess sampling reproducibility through the collection of field duplicates. Table A-7 provides a definition of the field quality control samples that are incorporated into the study design, as well as the collection frequency.

A.3.5.2 Analytical Laboratory

The study design and QC samples are intended to assess the major components of total study error, which facilitates the final evaluation of whether environmental data are of sufficient quality to support the related decisions. The QC sample requirements are designed to provide measurement error information that can be used to initiate corrective actions with the goal of limiting the total measurement error.

QC samples and frequency applicable to analytical chemistry laboratories are detailed in Table A-7. Table A-8 defines the required accuracy and precision for QC samples, along with corrective actions that must be implemented if QC criteria are not met. Table A-9 provides formulas for the calculation of QC sample assessment statistics. SOP 7-029 and the BSL QA Management Plan define the calculation of QC statistics at BDO and BSL.

All QC sample failures and associated corrective actions will be documented. If data must be reported with failing QC results, then data qualifiers will be assigned to the QC sample data. Table A-10 defines data qualifiers.

A.3.5.3 Control Charts

Laboratory control charts for organic chemistry procedures are established and maintained using the percent recovery results of the LCS. The control chart average, warning (2σ), and control limits (3σ) must be based on at least 20 individual percent recovery values generated within a calendar year vs. a "true value" calculation. Control charts for organic compounds at Battelle are maintained for representative compounds of interest for each method (*i.e.,* the same SOP). BSL uses a performance-based approach to assess and evaluate LCS and reference samples for trace metals. Criteria for monitoring control charts, for detecting warning or control limits, and for verifying that results fall within the acceptable limits are specified in the control chart SOPs or specific analytical procedures.

A.3.6 Instrumentation/Equipment Testing, Inspection, and Maintenance

A.3.6.1 Field Equipment

Battelle and its subcontractors will provide field sampling equipment, the boat, DGPS, and other supplies for the field-sampling program.

Maintenance requirements for field instruments are provided in Table A-11. The DGPS will be inspected and tested prior to use in the field. The DGPS manual and SOP must be available in the field. Any problems with the operation of this unit must be documented, along with corrective action and the results of performance verification.

A.3.6.2 Laboratory Equipment

All analytical instruments and equipment must be maintained according to SOPs and the manufacturers' instructions. Equipment and instrument and maintenance and frequency are defined in SOPs and are summarized in Tables A-11 and A-12. All routine maintenance and non-routine repairs are to be documented in a bound logbook. The information recorded should include analyst initials, date maintenance was performed, a description of the maintenance activity, and (if the maintenance was performed in response to a specific instrument performance problem) the result of re-testing to demonstrate that the instrument performance had been returned to acceptable standards prior to re-use. The return to analytical control is demonstrated by successful calibration.

A.3.7 Instrument/Equipment Calibration and Frequency

Field and laboratory equipment will be calibrated according to the manufacturers' recommendations. Field equipment refers to articles used for on-site monitoring and testing, whereas laboratory equipment refers to articles used in the laboratory in support of data collection (*e.g.*, refrigerators). Laboratory instruments are units used for sample analysis (*e.g.*, GC/MS).

A.3.7.1 Field Equipment

Table A-13 lists the calibration verification requirements for the DGPS. The Trimble 4000 DGPS Receiver DGPS is self-calibrated. The integrity of the unit is verified by conducting a comparison measurement at a known position (e.g., benchmark) versus the position that is acquired by the GPS unit. The defined accuracy of the DGPS is ±2 m. If the DGPS fails to attain a reading that is within 10 meters of the actual position, then the manual should be consulted for sources of error and the reference position verified. All DGPS units have a design positional accuracy of 10 meters. The GPS satellites are owned and controlled by the U.S. Department of Defense (DOD), which has the ability to degrade the accuracy of the GPS signal available to non-military users for purposes of national defense. During periods of selective availability (SA), the accuracy of the DGPS may vary by ±100 meters; however, SA was turned off by the U.S. Government on May 1, 2000. The calibration of each DGPS unit is checked by the field team prior to each day of use vs. the reference location defined in the WP/FSP.

The measurement points on the lead line used to determine sampling depths will be verified prior to the survey. The refractometer will be calibrated using MilliQ deionized water at the start of each sampling day. There is no calibration procedure associated with the ammonia Hach®Kit.

Calibration information will be recorded in the field logbook. Should any of the field equipment become inoperable, it will be removed from service and tagged to indicate that repair, recalibration, or replacement is needed. Backup equipment will be available and will be calibrated prior to use in the field.

A.3.7.2 Laboratory Equipment and Instruments

Laboratory equipment and instrument must be calibrated prior to use as defined in the laboratory SOPs. These requirements are summarized in Tables A-12 and A-13. Certified calibration standards used for instrument calibration will be obtained from commercial vendors for both inorganic and organic compounds and analytes. Where possible, standards will be traceable to National Institute of Standards and Technology (NIST). Stock solutions for spiking solutions, surrogate compounds, and other inorganic compound mixes will be made from reagent-grade chemicals or as specified in the SOPs. All analytical stock solutions will be prepared using Class-A volumetric ware. The receipt, preparation, and use of standards, and the related documentation, will be performed according to laboratory SOPs. All new calibration or spiking solutions will be analyzed against a previously-accepted standard to verify that the concentration of each COPEC is within 15% of the verified stock. Once accepted, an initial calibration check (IC) standard is run and quantified with each initial calibration. The resulting concentrations for the IC must be within 15% of the certified values.

Prior to analysis, instrument calibration must be verified through the analysis of a check solution prepared from a source (or at least a lot) independent of that used to prepare the calibration standards. Calibration check solutions must include all COPECs. GC/MS instruments must include a passing tune at the beginning of each instrument run (e.g., each new sequence).

A.3.8 Inspection/Acceptance of Supplies and Consumables

Supplies and consumables will be inspected and tested prior to use to ensure that they conform to the required level of quality. Any defective material will be tagged and removed from the work area. Each laboratory must maintain an inventory of all chemicals, reagents, and solvents.

Certified clean containers (ESS, SPEC, or equivalent) will be used as sample containers. Certificates of analysis provided with the containers will be retained by the laboratory or field sample custodian, depending upon whether containers are shipped from the lab or drop-shipped directly to the field by the

supplier. The containers will be inspected prior to use. Appropriate packing materials will be available for packing samples to avoid breakage during transport. Sample containers or field sampling utensils that are cleaned by the laboratory will be assigned a cleaning lot number. The cleaning log number will be recorded on the sample collection form.

A.3.9 Non-direct Measurements

Historical data were reviewed as Task 1 for this task order to identify data gaps and the need for additional investigation. A summary of the review will be incorporated into the risk assessment problem formulation and conceptual site model.

A.3.10 Data Management

Data generated in support of the Thames River RSC Pilot Study will be tracked and reviewed by the appropriate project team leader.

Data management (*e.g.*, paper flow; data tracking, data entry, etc.) and data assessment (*e.g.*, verification, validation, and Data Quality Assessment (DQA)) activities require adequate QC procedures to ensure that the SOPs are followed, resulting in records and reports that are accurate and appropriate, and corrective action as necessary to resolve non-conformance. QC procedures include peer review of each step and management review of a certain percentage of the data. Each laboratory must document its data management procedures in a SOP. Data verification and review is described in Section A.5.0.

A.3.10.1 Field Data

Preprinted labels (Section A.3.3.2) that include a unique sample identification number and prompt for required sample-specific information will be provided to the field team. A separate label is attached to each sample container and the sample ID recorded on the field log. This provides a unique link between the field records and each sample.

Sample collection information is hand recorded in field logbooks, then keyed into spreadsheets. Data entry into the electronic data deliverable (EDD) will be verified.

A.3.10.2 Laboratory Data

Data management at the laboratory begins with the receipt of samples. Samples are logged in and assigned unique identification numbers that are used to identify samples throughout storage, processing, analysis, and reporting.

For organics analysis, a Laboratory Information Management System (LIMS) houses all data for samples from the arrival of the sample in the laboratory to the final delivery of data to the client. This system is used to track samples from arrival through analysis to reporting. The LIMS software is a two-fold system. SQL Server 2000 is used as the back end of the system, all data is stored in a SQL Server 2000 database. Data is entered and manipulated on the end user level with an application developed in Microsoft Access 2000. With the exception of the database administrator and the database developer, all access to the database is accomplished using Microsoft Access 2000.

For trace metals analysis, a combination of hand-recorded and electronically-captured data are generated. Hand-recorded data include sample processing and spiking procedures. Hand-recorded data are transcribed to spreadsheets using established formats. (The raw data are maintained in the project files and the transcribed data are 100% verified). Electronically captured data may include sample weights and

instrument outputs. Once the analyst verifies the instrument output, data are exported to Excel spreadsheets for final reduction.

A.3.10.2 Electronic Data Deliverable

Laboratory deliverable will be as Microsoft Access database files that conform to the requirements of the Navy Environmental Data Transfer Standard (NEDTS). NEDTS requirements are defined at http://thadium.spawar.navy.mil/nedts. Fields that must be included in the spreadsheets for the Pilot Study are defined in Table A-14. Details are provided in Battelle Navy SOP 003 and 005.

A.4.0 ASSESSMENTS/OVERSIGHT

This section presents the internal and external checks (assessments) that will be used to assure that the elements of this QAPP have been implemented so that the quality of the data generated is adequate and satisfies the DQOs. Assessments also verify that when needed, corrective action is timely and effective. Assessment activities will include inspection, peer review, data audits, and data quality assessment.

A.4.1 Assessment and Response Actions

The following subsections identify planned assessment and oversight activities for this project. The Project QA Manager, and/or the Project Manager may identify additional assessment activities to be performed during the course of the Thames River RSC Pilot Study, based upon findings of the planned assessment activities described below. These individuals are authorized to stop work for cause if data quality or staff safety is threatened.

A.4.1.1 Assessment Actions

Audits evaluate the performance vs. the requirements of the QAPP and identify problems warranting correction. Auditors will be independent of the activities audited and will be selected by the Project QA Manager. Auditors will have the technical expertise and required to conduct a meaningful audit.

A.4.1.1.1 Assessment of Field Activities

Field audits are conducted during field sampling activities. No field audit is planned for the Thames River RSC Pilot Study.

A.4.1.1.2 Assessment of Laboratory Operations

Laboratory performance audits have been conducted for NFESC at BDO, BSL, and STL analytical laboratories. The purpose of a performance audit is to assure that the laboratory is capable of producing data of known and acceptable quality. The laboratory audits included reviews of the laboratory quality system, SOPs, and analytical procedures. The performance audits also include analysis of blind performance evaluation samples provided by the Navy to assess technical competence in the intended analyses. Navy certification of BDO, BSL, and the Severn-Trent Laboratories has been granted.

Each laboratory must have an internal audit program to monitor the degree of adherence to its own quality system. The internal audit programs include systems audits, performance evaluations, data audits, and laboratory inspections. The Quality System at each laboratory must be defined in a QA manual and/or SOPs.

The Project Laboratory Manager will communicate with each analytical laboratory on a regular basis. This will allow assessment of progress in meeting schedules, DQOs, and MQOs. This will ensure early identification of problems requiring corrective action. Problems and corrective actions will be reported to the Project Manager. The Project Laboratory Manager will be responsible for working directly with the laboratory to assure the prompt resolution of any problems identified.

A.4.1.2 Response Actions (Corrective Action)

An effective Quality System requires prompt and thorough correction of non-conformance conditions that can affect quality. Rapid and effective corrective action minimizes the possibility of questionable data or documentation.

Two types of corrective actions exist: immediate and long-term. Immediate corrective actions may include correction of documentation deficiencies or repair of inaccurate instrumentation. Often, the source of the problem is obvious and can be corrected at the time it is observed. Long-term corrective actions are designed to eliminate the sources of problems. Examples of long-term corrective actions are correction of systematic errors in analysis and correction of procedures producing questionable results. Corrections can be made through additional personnel training, instrument replacement, or procedural improvements. Long-term corrective action results in improvements to the system.

A.4.1.2.1 Field Procedures

Field non-conformance conditions are defined as occurrences or measurements that are either unexpected or that do not meet QAPP requirements and which will effect data quality if corrective action is not implemented.

A.4.1.2.2 Laboratory Procedures

The internal laboratory corrective action procedures and a description of non-conformance situations requiring corrective action are contained in the laboratory QA plan and SOPs. At a minimum, corrective action and notification of the Project Laboratory Manager will be implemented if: (1) control chart warning or control limits are exceeded, (2) QC requirements are not met, and (3) sample holding times are exceeded. Non-conformance situations will be reported to the appropriate laboratory manager within two working days after they are identified. Where corrective action is not feasible, appropriate qualifiers will be added to data.

A.4.1.2.3 Corrective Action

Corrective action procedures will depend on the severity of the non-conformance condition. In cases in which immediate and complete corrective action is implemented, the corrective action will be documented in the log notebook. Non-conformance conditions that could impact data quality must be reported to the Project Manager. If warranted, a follow-up audit will be conducted to verify that the corrective action is adequate and permanently corrects the problem. The Project Manager can require that technical activities be limited or discontinued until the corrective action is complete and the problem eliminated.

Quality issues and corrective actions will be documented at each laboratory. Documentation will include definition of the problem, the intended short and long-term corrective action, and the dates that these actions were implemented and verified by QA.

A.4.2 Reports to Management

The results of Quality Assurance audits and inspections will be reported to both laboratory line management and the Project Manager.

A.5.0 DATA VALIDATION AND USABILITY

This section of the QAPP provides a description of the data review activities that will occur after the data collection phase of the project is completed. The requirements and methods for data review, verification, and validation, as well as the process for reconciling data generated with the DQOs are described. Implementation of these methods will determine whether or not the data conform to the specified criteria, thus satisfying the project objectives.

A.5.1 Data Review, Validation, and Oversight

Data review includes data verification, validation, and oversight, as well as reconciliation of the data quality with user requirements. The data verification process includes the initial review of the data packages to ensure that the analytical results are accurate, complete, traceable, and performed according to the QAPP. Data validation is the process of reviewing data and accepting, qualifying, or rejecting data on the basis of sound criteria using established EPA guidelines. Final technical data review of the confirmation (quantitative) data occurs after independent data validation has been completed. It provides an indication of overall trends in data quality and usability.

A.5.1.1 Data Verification

Analytical data, including supporting QC results, will be assembled in data packages by sample delivery group as defined in Section A.2.6.5. Each analytical laboratory is responsible for reviewing each data package prior to release for validation. At a minimum, the following reviews must be performed:

- 100% review by the analyst;
- 100% review by a technical supervisor or qualified;
- Final administrative review that verifies that previous reviews were documented properly and that the data package is complete; and,
- QA office review of a minimum of 10% of all data packages for technical completeness and accuracy. This review is part of the oversight program and does not have to be completed in "real time."

Implementation of these procedures is defined in laboratory SOPs. These reviews must ensure the following:

- All data for project samples are reported accurately and completely;
- Sample analysis was conducted in accordance with required laboratory procedures and analytical methods specified in the QAPP and WP/FSP;
- Criteria for data quality have been met or deviations are documented in the package narrative and data flags have been appropriately applied;
- Each data set is appropriately reviewed; and,
- All project requirements have been met.

The verification of data generated by the SSC SD laboratory is the responsibility of the Navy.

A.5.1.2 Data Validation

Data validation is conducted to assess the compliance of chemistry data with the MQOs defined in the QAPP, and compliance with the requirements of the analytical methods. For this study, data validation will be performed on the quantitative data generated by BDO (organics compounds), BSL (trace metals), and STL (TOC and grain size). The results of the RSC analyses will not be validated. The validation strategy applied to this project is that 20% of the data will receive Level-IV validation and the remaining 80% of the data will receive Level-III validation. Laboratory Data Consultants (Carlsbad, CA) is the data validation firm.

The data generated by BDO and BSL use low-level (NOAA Status and Trends) analytical methods that are appropriate for the assessment of ecological risk. The National Functional Guidelines (NFG) (EPA 1994a, 1994b, and 1994c) which are typically applied to the validation of data generated according to EPA Methods may not apply to the low-level data because the requirements of the methods may vary. The data assessment criteria are defined in the QAPP, the WP/FSP, and the laboratory analytical SOPs. Where conflicts exist, the requirements of the QAPP and the related SOPs over-ride the NFG.

- Level-III data validation assumes that reported data values are correct as reported. Data quality is assessed by verifying that the criteria defined in the QAPP for each compound class have been achieved (Table A-8). Thirty samples will receive Level III validation.
- Level-IV data validation is based on the assessment of raw data packages, which include all data required for a full review and assessment of compound selection, integration, interference assessment, and re-quantification (*e.g.,* spectra and chromatograms). Level-IV data validation includes re-quantification of reported QC and field sample values using the raw data files. In addition, instrument performance, calibration methods, and calibration standards are reviewed to ensure that the detection limits and data values are accurate and appropriate. Seven samples will receive Level IV validation.

During data validation, the laboratory data are assessed against prescriptive requirements. Evaluation of laboratory performance against prescription requirements is assessed through compliance with the method requirements and the acceptability of QC sample results that are independent of sample matrix (*e.g.,* instrument performance checks, calibration criteria).

The data validation report will include a narrative that details all QC exceedances and explains qualifications of data results. Final data qualifiers will be applied by the validators directly in the EDD. The validation assessment parameters are listed in Table A-15.

A.5.2 Data Quality Assessment Reconciliation with Planning Objectives

Data Quality Assessment (DQA) is a data analysis and interpretation process involving scientific and statistical evaluation of data to determine if data are sufficient to support their intended use (usually stated in terms of supporting a specific decision). To implement the DQA process, the data analyst will work closely with a multidisciplinary team potentially including the Leaders, chemist, statistician, risk assessor and other relevant scientists.

The proposed RSC Pilot Study will result in the estimates of several important metals (Cu, Pb, Zn, Cr, and As) using XRF methods, and total PCBs, and total PAHs using immunoassay methods. In addition, 37 samples will be split, and sent to a traditional fixed laboratory for confirmation purposes using NOAA National Status and Trends Methods. Upon receipt of the analytical data, the data analyst will assemble

the data set, including field information such as sample coordinates and descriptions of the associated field measurements. Any laboratory validation reports will also be reviewed. The DQA will begin with some exploratory data analysis steps, including graphical analysis of the data using box plots, scatter plots, q-q plots, histograms, and other tools. Potential outliers identified through this process may trigger focused validation to determine if the suspect data points resulted from errors in the data generation process, or whether they appear to be valid measurements and should be used. Unless this process uncovers a specific explanation supporting their removal, they will be kept and used. All such decisions to accept or reject data will be carefully documented.

The immediate use of these RSC data will be to evaluate the spatial patterns of contamination. This will be accomplished using a variety of spatial plots including bubble plots, and geostatistical plots involving smoothing algorithms (e.g., Kriging or other geostatistical tools). To determine sample sizes, the variability of concentrations observed in the pilot study will provide an important input to design calculations. Data will be used to determine the appropriateness of stratifying the study area, to maximize the utility of the follow-on study. As soon as the fixed lab data are available, regression analysis will be performed to evaluate how well the RSC methods correlate with fixed lab measurements, whether the detection limits associated with these methods were adequate, and whether corrections (for example to remove positive bias) are supported.

Confirmatory laboratory data will also be used for a screen using ecological benchmark values, during the screening refinement evaluation and in a screening level ecological risk assessment. Provided that the sampling design was adequate to support the stated decision, the evaluation of data adequacy to support that decision may terminate after the initial exploratory analysis, and the site should move forward in the decision-making process. This determination will be made based on the observed chemistry values, the variability of these measurements, and a determination of the uncertainty associated with the types of comparisons that are being made with the data.

The investigators will use the elements of the DQA process defined by EPA that are relevant to data use, or its equivalent, to assess data adequacy to support a statistically based decision. The first two steps of this formal DQA process, review of the sampling design and preliminary data review, are described above. The remaining three steps are summarized below:

- Data analyst will work with the project team to ensure that the most appropriate statistical tests will be used. Additional or alternate tests may be considered at this time. Underlying assumptions that must hold for the proposed statistical procedures will be evaluated for this data set. Also, the data analyst will consult with the appropriate scientists and site experts to ensure that the comparisons implied by the statistical test are appropriate.
- Data analyst will use the site data to generate estimates of total study error based on the data collected and to perform the appropriate statistical tests at a significance level consistent with the decision-makers' desire to control decision errors.
- If an adequate level of confidence was achieved at the chemical concentrations actually observed, this observation supports the case that data are sufficient to support the proposed decision.

Results of DQA will be documented in adequate detail for the decision-maker and peer reviewers to evaluate the effect of these results on decision-making.

Table A–1. Cross-walk Between EPA QA/R-5 and the Requirements of EPA Region I.

Required USEPA QA/R-5 Elements	QAPP Location	Required USEPA-NE QAPP Elements and Corresponding US EPA-NE QAPP Sections	USEPA-NE QAPP Worksheet No.	Required Information
		Project Management and Objectives		
A1	Page A-1	1.0 Title and Approval Page	1	- Title and Approval Page
A2	Page A-3 Page A-3 All A.2.6.1 Table A-1	2.0 Table of Contents and Document Format 2.1 Table of Contents 2.2 Document Control Format 2.3 Document Control Numbering System 2.4 EPA-NE QAPP Worksheet #2	2	- Table of Contents - USEPA-NE QAPP Worksheet
A3	Page A-2	3.0 Distribution List and Project Personnel Sign-off Sheet	3 4	- Distribution List - Project Personnel Sign-off Sheet
A4, A8	A.2.1 Figure A-1 Figure A-1 A.6.1.1 A.2.1 A.2.5	4.0 Project Organization 4.1 Project Organization Chart 4.2 Communication Pathways 4.2.1 Modifications to Approved QAPP 4.3 Personnel Responsibilities and Qualifications 4.4 Special Training Requirements/Certification	5a 5b 6 7	- Organization Chart - Communication Pathways - Personnel Responsibilities and Qualifications Table - Special Personnel Training Requirements Table
A5	A.2.3 A.1.0 A.2.2	5.0 Project Planning/Project Definition 5.1 Project Planning Meetings 5.2 Problem Definition/Site History and Background	8a 8b	- Project Planning Meeting Documentation - Project Scoping Meeting Attendance Sheet with Agenda - Problem Definition/Site History and Background - USEPA-NE DQO Summary Form - Site Maps (historical and present)
A6	A.2.3 A.2.3 A.1.0	6.0 Project Description and schedule 6.1 Project Overview 6.2 Project Schedule	9a 9b 9c 9d 10	- Project Description - Contaminants of Concern and Other Target Analytes Table - Field and Quality Control Sample Summary Table - Analytical Support Laboratories - System Designs - Project Schedule Timeline, products, and deliverable table
A7	A.2.4 A.2.4 A.2.4.2	7.0 Project Quality Objectives and Measurement Performance Criteria 7.1 Project Quality Objectives 7.2 Measurement Performance Criteria	11 11	- Measurement Performance Criteria - Concentration Levels
		Measurement/Data Acquisition		
B1	A.3.1 A.2.3	8.0 Sampling Process Design 8.1 Sampling Design Rationale	 12a 12b	- Sampling Rationale - Sampling Locations, Sampling and Analytical Method/SOP Requirements Table - Sample Location Map

Table A–1. Cross-walk Between EPA QA/R-5 and the Requirements of EPA Region I.

Required USEPA QA/R-5 Elements	QAPP Location	Required USEPA-NE QAPP Elements and Corresponding US EPA-NE QAPP Sections	USEPA-NE QAPP Worksheet No.	Required Information
B2, B6 B7, B8	A.3.2 A.3.2 A.2.6.1.1 A.3.3 A.3.7 A.3.6 A.3.8	9.0 Sampling Procedures and Requirements 9.1 Sampling Procedures 9.2 Sampling SOP Modifications 9.3 Cleaning and Decontamination of Equipment/Sample Containers 9.4 Field Equipment Calibration 9.5 Field Equipment Maintenance, Testing and Inspection Requirements 9.6 Inspection and Acceptance Requirements for Supplies/Sample Containers	13 12b 14 15	- Sampling SOPs - Project Sampling SOP Reference Table - Sampling Container, Volumes and Preservation Table - Sampling Equipment Calibration Table - Cleaning and Decontamination SOPs - Field Equipment Maintenance, Testing and Inspection Table.
B3	A.3.3 A.2.6.2 A.2.6.2 A.2.6.2 A.3.3 A.3.3.2	10.0 Sample Handling, Tracking, and Custody Requirements 10.1 Sample Collection Documentation 10.1.1 Field Notes 10.1.2 Field Documentation Management System 10.2 Sample Handling and Tracking System 10.3 Sample Custody	16	- Sample Handling, Tracking and Custody SOPs - Sample Handling Flow Diagram - Sample Container Label (Sample Tag) - Chain-of-Custody and Seal
B4, B6 B7, B8	A.3.4.1 A.3.2 A.2.6.1.1 A.3.7.1 A.3.6.1 A.3.8	11.0 Field Analytical Method Requirements 11.1 Field Analytical Methods and SOPs 11.2 Field Analytical Method/SOP Modifications 11.3 Field Analytical Instrument Calibration 11.4 Field Analytical Instrument/Equipment Maintenance, Testing and Inspection Requirements 11.5 Field Analytical Inspection and Acceptance Requirements for Supplies	17 18 19	- Field Analytical Methods/SOPs - Field Analytical Method/SOP Reference Table - Field Analytical Instrument Calibration Table - Field Analytical Instrument/Equipment Maintenance, Testing and Inspection Table
B4, B6 B7, B8	A.3.4 A.3.4.3 A.2.6.1.1 A.3.7.2 A.3.6.2 A.3.8	12.0 Fixed Laboratory Analytical Method Requirements 12.1 Fixed Laboratory Analytical Methods and SOPs 12.2 Fixed Laboratory Analytical Method/SOP Modifications 12.3 Fixed Laboratory Instrument Calibration 12.4 Fixed Laboratory Instrument/Equipment Maintenance, Testing and Inspection Requirements 12.5 Fixed Laboratory Inspection and Acceptance Requirements for Supplies	20 21	- Fixed Laboratory Analytical Methods/SOPs - Fixed Laboratory Analytical Method/SOP Reference Table - Fixed Laboratory Instrument Maintenance and Calibration Table

Table A–1. Cross-walk Between EPA QA/R-5 and the Requirements of EPA Region I.

Required USEPA QA/R-5 Elements	QAPP Location	Required USEPA-NE QAPP Elements and Corresponding US EPA-NE QAPP Sections	USEPA-NE QAPP Worksheet No.	Required Information
B5	A.3.5 A.3.51 A.3.5.2 A.3.5.1 A.3.5.2 A.3.5.1 A.2.4.2	13.0 Quality Control Requirement 13.1 Sampling Quality Control 13.2 Analytical Fixed Laboratory Quality Control 13.2.1 Field Analytical QC 12.2.2 Fixed Laboratory QC 13..2.3 QC sets for field 13.2.4 Acceptance Criteria	 22a 22b 23a 23b 24a 24b	**Sampling** - Field Sampling QC Table - Field Sampling QC Table (Cont) **Analytical** - Field Analytical QC Sample Table - Field Analytical QC Sample Table (Cont) - Fixed Laboratory Analytical QC Sample Table - Fixed Laboratory Analytical QC Sample Table (Cont)
B9	A.3.10.2	14.0 Data Acquisition Requirements	25	- Non-direct Measurement Criteria and Limitations Table
A9, B10	A.2.6 A.2.6 A.2.6.2 A.2.6.6 A.2.6.5 A.2.6.6 A.3.10.2 A.3.10 A.2.6.1.2 A3.10	15.0 Documentation, Records, and Data management 15.1 Project Documentation and Records 15.2 Field Analysis Data Package Deliverables 15.3 Fixed Laboratory Data Package Deliverables 15.4 Data Reporting Formats 15.5 Data Handling and Management 15.6 Data Tracking and Control	26	- Project Documentation and Records Table - Data Management SOPs
Assessment/Oversight				
C1	A.4.1 A.4.1 A.4.1.2 A.4.1.2.3	16.0 Assessments and Response Actions 16.1 Planned Assessments 16.2 Assessment Findings and Corrective Action Response 16.3 Additional QAPP Non-Conformances	27a 27b 27c	- Assessment and Response Actions - Project Assessment Table - Project Assessment Plan - Audit Checklists
C2	A.4.2	17.0 QA Management Reports	28	- QA Management Reports Table
Data Validation and Usability				
D1	A.5.0	18.0 Verification and Validation Requirements		- Validation Criteria Documents
D2	A.5.0	19.0 Verification and Validation Procedures	29a 29b 29c	- Data Evaluation Process - Data Validation Summary Table - Data Validation Modifications
D3	A.5.2	20.0 Data Usability/Reconciliation with Project Quality Objectives	30	- Data Usability Assessments

Table A–2. COPECs, Detection Limits, and Benchmarks for Thames River RSC Pilot Study.

Analytes	Laboratory Values for Sediment Analysis		Benchmark Source/Citation
	Method Detection Limit	Reporting Limit	
TRACE METALS (mg/Kg)			
Arsenic (EPA 200.8)/XRF[1]	0.07/10	0.22/20	8.20 (ER-L; Long *et al.*, 1995)
Barium (EPA 200.7)	0.02	0.06	NA
Cadmium (EPA 200.8)	0.02	0.06	1.2 ER-L (Long *et al.*, 1995)
Chromium (EPA 200.7)/XRF[1]	0.5/50	1.6/100	81 ER-L (Long *et al.*, 1995)
Copper (EPA 200.7)/XRF[1]	0.2/10	0.64/20	34 ER-L (Long *et al.*, 1995)
Lead (EPA 200.8)/XRF[1]	0.2/5	0.64/10	46.7 ER-L (Long *et al.*, 1995)
Mercury (EPA 245.5 MOD)	0.002	0.006	0.15 ER-L (Long *et al.*, 1995)
Nickel (EPA 200.7)	0.04	0.13	20.9 ER-L (Long *et al.*, 1995)
Selenium (EPA 270.3)	0.1	0.32	NA
Silver (EPA 200.9)	0.03	0.10	1.0 (ER-L; Long *et al.*, 1995)
Zinc (EPA 200.7)/XRF[1]	0.1/10	0.32/20	150 ER-L (Long *et al.*, 1995)
ORGANIC COMPOUNDS - PAHs (µg/Kg)			
Naphthalene	1.486	0.6	160 ER-L (Long *et al.*, 1995)
2-Methylnaphthalene	0.037	0.6	70 ER-L (Long *et al.*, 1995)
1-Methylnaphthalene	0.023	0.6	NA
Biphenyl	0.017	0.6	NA
2, 6-Dimethylnaphthalene	0.033	0.6	NA
Acenaphthylene	0.026	0.6	44 ER-L (Long *et al.*, 1995)
Acenaphthene	0.016	0.6	16 ER-L (Long *et al.*, 1995)
1, 6, 7-Trimethylnaphthalene	0.026	0.6	NA
Fluorene	0.023	0.6	19 ER-L (Long *et al.*, 1995)
Phenanthrene	0.030	0.6	240 ER-L (Long *et al.*, 1995)
Anthracene	0.024	0.6	85.3 ER-L (Long *et al.*, 1995)
1-Methylphenanthrenes	0.028	0.6	NA
Fluoranthene	0.025	0.6	600 ER-L (Long *et al.*, 1995)
Pyrene	0.020	0.6	665 ER-L (Long *et al.*, 1995)
Benzo(a)anthracene	0.017	0.6	261 ER-L (Long *et al.*, 1995)
Chrysene	0.013	0.6	384 ER-L (Long *et al.*, 1995)
Benzo(b)fluoranthene	0.018	0.6	NA
Benzo(k)fluoranthene	0.013	0.6	240 ER-L (Persaud *et al.*, 1993)
Benzo(e)pyrene	0.012	0.6	NA
Benzo(a)pyrene	0.021	0.6	430 ER-L (Long *et al.*, 1995)
Perylene	0.015	0.6	NA
Indeno(1,2,3-c,d)pyrene	0.010	0.6	78 (Freshwater ER-L, based on 14-day *H. azteca* test. EPA 1996c)
Dibenzo(a,h)anthracene	0.018	0.6	63.4 ER-L (Long *et al.*, 1995)
Benzo(g,h,i)perylene	0.013	0.6	290 Freshwater ER-L based on 14-day *C. riparius* test. EPA 1996c)
Total PAH (immunoassay)[1]	200	400	
ORGANIC COMPOUNDS - Pesticides (µg/Kg)			
4,4'-DDD	0.045	0.4	2 (ER-L; Long and Morgan, 1991)
2,4'-DDD	0.045	0.4	NA
4,4'-DDE	0.041	0.4	2.20 ER-L (Long *et al.*, 1995)
2,4'-DDE	0.061	0.4	NA
4,4'-DDT	0.042	0.4	1 (ER-L; Long and Morgan, 1991)
2,4'-DDT	0.057	0.4	NA

Table A–2. COPECs, Detection Limits, and Benchmarks for Thames River RSC Pilot Study. (con't)

Analytes	Laboratory Values for Sediment Analysis		Benchmark Source/Citation
	Method Detection Limit	Reporting Limit	
ORGANIC COMPOUNDS - Pesticides (µg/Kg)			
Aldrin	0.029	0.4	NA
Cis-Chlordane	0.033	0.4	0.50 (ER-L; Long and Morgan, 1991)
Dieldrin	0.044	0.4[2]	0.02 (ER-L; Long and Morgan, 1991)
Endrin	0.040	0.4[2]	0.02 (ER-L; Long and Morgan, 1991)
Gamma-BHC (Lindane)	0.018	0.4	NA
Heptaclor	0.037	0.4	NA
Heptaclor epoxide	0.035	0.4	NA
Hexachlorobenzene	0.041	0.4	NA
Mirex	0.037	0.4	NA
Trans-nonachlor	0.037	0.4	NA
PCB COMPOUNDS - Pesticides (µg/Kg)			
PCB 8	0.075	0.4	NA
PCB 18	0.036	0.4	NA
PCB 28	0.052	0.4	NA
PCB 44	0.046	0.4	NA
PCB 52	0.049	0.4	NA
PCB 66	0.054	0.4	NA
PCB 77	0.076	0.4	NA
PCB 101	0.047	0.4	NA
PCB 105	0.041	0.4	NA
PCB 118	0.053	0.4	NA
PCB 126	0.066	0.4	NA
PCB 128	0.087	0.4	NA
PCB 138	0.044	0.4	NA
PCB 153	0.066	0.4	NA
PCB 170	0.044	0.4	NA
PCB 180	0.047	0.4	NA
PCB 187	0.043	0.4	NA
PCB 195	0.040	0.4	NA
PCB 206	0.040	0.4	NA
PCB 209	0.043	0.4	NA
Total PCBs (immunoassay)[1]	50	100	22.7 (ER-L; Long *et al.* 1995)

[1]This compound is analyzed using rapid sediment characterization techniques.

[2]The reporting limits for two COPECs (dieldrin and endrin) are higher than the ER-L benchmarks. The actual reporting limits can be lowered by increasing the sample size and decreasing the pre-injection volume of the sample. Detected values that are less than the reporting limit will be qualified as estimates and used with caution during any assessment. See Section A.2.4.2 for a discussion of how COPECs with reporting limits that are greater than the benchmark will be treated.

Table A–3. Critical and Non-Critical Measurements for the Thames River Rapid Sediment Characterization Pilot Study.

Critical	Non-Critical
Field Measurement	
• Latitude • Longitude • Water depth	• Salinity • Porewater ammonia
Physical Characterization	
• Grain size • Sediment total organic carbon (TOC) • Percent moisture	
Chemical Analysis	
• Trace metal screening by XRF • Total PCBs screening by immunoassay • Total PAH screening by immunoassay • COPEC Analysis: Low-level trace metals Low-level PAHs Low-level Pesticides Low-level PCB congeners	

Table A–4. Standard Operating Procedures.

Battelle Duxbury Operations	
(3) Facilities And Equipment	
3-092	Operation and Maintenance of Hewlett-Packard 5970B, 5972A, and 5973A Gas Chromatograph/ Mass Selective Detector (GC/MSD) using Hewlett-Packard Software
3-116	Operation and Maintenance of Gas Chromatographs
(5) Laboratory and Field Procedures	
3-051	Use of Salinity Refractometers
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
5-169	Collection and At-Sea Processing of Benthic Grab Samples
5-192	Soil/Sediment Extraction for Trace Level Semi-Volatile Organic Contaminant Analysis
5-210	Packaging and Shipping of Samples
(6) Documentation, Records, And Reports	
6-010	Sample Receipt, Custody and Handling
(7) Data Processing	
7-029	Preparation, Analysis, and Reporting Quality Control Data in the Chemistry Laboratory
BDO HPS SOP 001	Performance of Analytical Chemistry Work According to the Requirements of the Naval Facilities Engineering Service Center
BDO HPS SOP 003	EDD for Analytical Laboratories
Battelle Sequim Laboratory	
(A) Administrative	
MSL-A-002	Sample Chain of Custody
(I) Inorganic chemistry	
MSL-I-013	Total Mercury in Aqueous Samples by CVAF
MSL-I-016	Total Mercury in Tissues and Sediment by CVAA
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-028	Navy Sample Analysis Plan
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
Severn-Trent Laboratories-Burlington (Grain Size and TOC)	
LM-WC-TOC Lloyd	Determination of Total Organic Carbon in Soils and Sediments: Lloyd Kahn Method
LM-SL-D422	Method: ASTM D422 Particle Size Analysis of Soils
SSC SD SOPS	
SSC-SD	X-Ray Fluorescence technique (XRF) (EPA Method 6200, EPA, 1998)

Table A–5. Sample Containers, Sample Size, Preservative Requirements, and Holding Time for Analytical Samples.

Parameter	Method Container[1]	Minimum Sample Size	No. of Containers	Sample Preservative	Holding Time[2] x/y
SEDIMENT - Organic Compound Analysis					
Polycyclic aromatic hydrocarbons	G	100 g	1	4°±2°C	14 days/40 days
Organochlorine pesticides Polychlorinated biphenyls (PCBs)	G	(100 g)[3]	(1)[3]	4°±2°C	14 days/40 days
SEDIMENT - Inorganic Compound Analysis					
Metals –GFAA, ICP-AES, ICP-MS, or HGAA-FIAS	G	(30 g)[3]	1	4°±2°C	6 months
Hg	G	(30 g)[3]	(1)	4°±2°C	28 days
SEDIMENT - Physical Characterization					
Total organic carbon (TOC)	G	1.0 g	(1)	4°±2°C	14 days
Grain size	G	(500 g)[4]	1	4°±2°C	None
Percent moisture	G	(10 g)[3,4]	(1)	4°±2°C	14 days
Rapid Sediment Screening					
Rapid sediment screening samples (Total PCB, Total PAH, and metals)	G	20 g total[5]	1	4°±2°C	14 days/40 days

[1] Container Types: G = Amber glass with Teflon-lined lid.
[2] "x" days/"y" days refers to the maximum number of days from sampling to extraction/the maximum number of days from extraction to analysis.
[3] One 16 oz jar of sample will be collected and split at Battelle Duxbury for these analyses (See Section A.3.3.1).
[4] One 16 oz jar of sample will be collected and shipped directly from Battelle Duxbury to STL Burlington for these analyses (See Section A.3.3.1).
[5] One 4 oz jar of sample will be shipped or hand-carried directly from the field to SSC SD for these analyses (See Section A.3.3.1).

Table A–6. Methods for Laboratory Analysis.

Parameter (Units)	EPA Base Method	Lab	Laboratory SOP	Description[1]
SEDIMENT				
PAHs (ng/g)	Lauenstein and Cantillo (1993)[2]	BDO	5-157	ST GC/MS SIM
Pesticides (ng/g) PCBs (Congeners) (ng/g)	Lauenstein and Cantillo (1993)[2]	BDO	5-128	ST GC/ECD
Metals (μg/g)	EPA Method 200.9[2], and EPA 7000 series	BSL	MSL-I-029	AD GFAA
Metals (μg/g)	EPA 270.3, and EPA 7000 series; Mod	BSL	MSL-I-030	AD HGAA-FIAS
Metals (μg/g)	EPA 200.7[2], and EPA 6010B; Mod	BSL	MSL-I-027	AD ICP-AES
Metals (μg/g)	EPA 200.8[2] and 6020	BSL	MSL-I-022	AD ICP-MS
Hg (μg/g)	EPA 245.5[2] Mod	BSL	MSL-I-016	AD CVAA
Physical Characterization				
Percent moisture (%)	Lauenstein and Cantillo (1993)	BDO	5-192	Drying Oven
Percent moisture (%)	Lauenstein and Cantillo (1993)	BSL	MSL-C-003	Freeze Drying
TOC (mg/g)	Kahn (1988)	STL-Burlington	LM-LS-D422	Elemental Analyzer
Percent moisture (%)	EPA 3550	STL-Burlington	SAC-GC-0007	Drying Oven
Grain size (%)	ASTM D-422	STL-Burlington	LM-SL-D422	Sieve Hydrometer
Rapid Sediment Screening				
Total PCBs (ppb)	EPA 4020 Mod	SSC SD	NA	Methanol extraction with modified dilution for 0.1 to 1.0 ppm range
Trace metals (ppm)	EPA 6200	SSC SD	NA	Sediment homogenization and direct analysis of sediment using "soils" method (SSC-SD).
Total PAH (ppb)	EPA 4035	SSC SD	NA	Methanol extraction and analysis by EPA Method 4035

[1]Description: ST=Shaker table extraction; AD=Acid digestion; US=Ultrasonic extraction.
[2]Low-level NOAA Status and Trends methods are selected to achieve the required study detection limits.
[3]Modified for low-level detection limits.

Table A–7. Definitions, Requirements, and Frequency for Typical Quality Control Samples.

QC Sample	Definition	Frequency
	FIELD QUALITY CONTROL	
Field Duplicate (DU)	Field duplicates are two samples of the same matrix, which are collected, to the extent possible, at the same time, from the same location, using the same techniques. Field duplicates must be handled, containerized, preserved, stored, and transported in the same manner. Pier 1 (two), Zone 4 (two), and Zone 7 (three).	A total of 7 collocated duplicate samples (10 %) will be collected for RSC analysis.
	LABORATORY QUALITY CONTROL	
Method or Procedural Blank (MB)	A combination of solvents, surrogates, and all reagents used during sample processing, processed concurrently with the field samples. Monitors purity of reagents and laboratory contamination[2].	1/sample batch[1] A processing batch MB must be analyzed with each sequence.
Laboratory Control Sample (LCS)	A LCS sample is a matrix-specific sample that is prepared with each processing batch. It is spiked with the analytes of interest and processed identically to the field samples[2].	1/sample batch
Matrix Spike (MS)[3]	A field sample spiked with the analytes of interest at 10 X the MDL, processed concurrently with the field samples; monitors effectiveness of method on sample matrix. An MS must be processed for each distinct matrix[2].	1/sample batch
Duplicate Sample (QADU) or Matrix Spike Duplicate (MSD)[3]	Second aliquot of a field sample processed and analyzed to monitor precision; each sample set should contain a duplicate. The duplicate may be a second matrix spike sample.	1/sample batch
Recovery Internal Standards (RIS)	All field and QC samples are spiked with recovery internal standards just prior to analysis; used to quantify surrogates to monitor extraction efficiency on a per sample basis[2].	Each organic compounds sample
Surrogate Internal Standards (SIS)	All field and QC samples are spiked with a known amount of surrogates just prior to extraction; recoveries are calculated to quantify extraction efficiency[2].	Each organic compounds sample
Standard Reference Material (SRM)	An external reference sample containing a certified level of target analytes; serves as a monitor of accuracy. Extracted and analyzed with samples of a like matrix.	1/batch
Performance Evaluation (PE) or Performance Test (PT)	Blind sample of unknown composition that is analyzed as a routine sample. The PE is provided by either a government or commercial agency. Results are submitted to the supplier who determines whether the results fall within a statistically acceptable range[2].	2/year
Independent Instrument Check (IC)	Direct spike of target analytes into solvent where the spike source is independent of that used to prepare the calibration standards to assess instrument performance.	1/analytical run if an independent source is available
Instrument (Solvent) Blank (IB)	An injection of straight solvent to assess sample carry-over in GCs (not GC/MSs)[2].	1/10 samples
Reagent or Solvent Purity Checks	All reagents are lot-checked prior to use[2].	Per lot purchase

[1]A batch is defined as 20 field samples processed simultaneously and sharing the same QC samples.
[2]Applies only to confirmatory analyses.
[3]Non-Navy samples may not be substituted to meet this requirement.

Table A–8. Measurement Quality Criteria.[1,2]

QC Parameter	Acceptance Criteria	Corrective Action[3]
Accuracy		
Field(Equipment) Blank	Organic compounds, metals: <RL	Review data and assess results for evidence of field-related contamination. Flag all data that are >RL.
Instrument Solvent Blank	Organic compounds, metals, XRF: < lowest calibration standard	Review data and analysis for possible sources of contamination. Reanalyze and/or document corrective action. Data must be flagged.
Method (Procedural) Blank	Organic compounds, metals, immunoassays for Total PCB, Total PAH, and XRF: <RL	Perform corrective action as above and re-process (extract, digest) sample batch. If batch cannot be re-processed; flag all data that are >RL.
•*SRM*	Organic compounds: Average PD ≤30%; ≤35% for each analyte. Metals: ≤20% PD. Evaluated for analytes >10 x the MDL. XRF (PACS-1 and/or PACS-2): ≤20% PD Immunoassay (Aroclor 1254 and Phenanthrene): ±20% Recovery **Comment: Determined vs. certified range. Certified value must be 10xMDL to be used for MQC for the selected analyte.**	Review data to assess impact of matrix. Reanalyze sample and/or document corrective action. If other QC data are acceptable then flag associated data if sample is not reanalyzed.
•*Matrix Spike*	Organic compounds: 40 - 120% recovery (Concentration in spike must be >5 ×background levels to be used for data quality assessment) Metals: 70 - 130% recovery	Review data to assess impact of matrix. If other QC data are acceptable and no spiking error occurred, then flag associated data. If QC data are not affected by matrix failure or spiking errors occurred, then re-process MS. If not possible, then notify client and flag associated data.
•*Surrogate Spike (SIS)*	Organic compounds: 40 - 120% recovery	Review data. Discuss with Laboratory Manager (LL). Reanalyze, re-extract, and/or document corrective action and deviations.
•*Laboratory Control Sample (LCS)*	Organic compounds: 40 - 120% recovery Metals: 70 - 130% recovery Immunoassay (Aroclor 1254 and Phenanthrene): ±20% Recovery	Perform corrective action. Re-analyze and/or re-process sample batch. Batch data associated with failed LCS (LCS data outside control limits) cannot be reported. If batch cannot be re-processed: notify client, flag data, discuss impact in report narrative.
•*Instrument Check*	Organic compounds: 85 - 115% recovery	Perform corrective action. Re-analyze and/or re-process sample batch. Data outside control limits cannot be reported. If batch cannot be re-processed, notify client, flag data, discuss impact in report narrative.
Precision: *Laboratory Duplicates*	Organic compounds (MSD): <30% RPD. **Concentration detected must be >5 times RL to be used for data quality assessment** Metals: <30% RPD. **Evaluated for analytes >10 x MDL.** XRF: <20% RPD Immunoassay Extraction duplicate <30% RPD Immunoassay Assay duplicate <30% RPD	Review data to assess impact of matrix. If other QC data are acceptable, then flag associated data. If QC data are not affected by matrix failure, then re-process duplicate. If not possible, then notify client and flag associated data.
	PHYSICAL CHARACTERIZATION	
Accuracy	TOC: Method Blank <Reporting Limit LCS: % Recovery within control limits MS: 75 – 125 % Recovery ICV/CCV: ± 10% true value	Review data. Discuss with LL. Reanalyze and/or document corrective action and deviations. Flag data.
Precision: *Duplicates*	TOC: ± 20% RPD Grain size: Visual inspection of graphical presentation of results.	Review data. Discuss with LL. Reanalyze and/or document corrective action and deviations. Flag data.

[1]See abbreviation definitions
[2]Individual parameters included in the compound classes "Organic compounds," and "Metals" are defined in Table A-4.
[3] Data qualifiers for quality control sample exceedences are defined in Table A-9.

Table A–9. Calculation of Quality Control Assessment Statistics.

Percent Recovery

The percent recovery is a measurement of accuracy, where one value is compared with a known/certified value. The formula for calculating this value is:

$$Percent\ \ Recovery = \frac{amount\ \ detected}{amount\ \ expected} x\ 100$$

Percent Difference

The percent difference (PD) is a measurement of precision as an indication of how a measured value is difference from a "real" value. It is used when one value is known or certified, and the other is measured. The formula for calculating PD is:

$$Percent\ \ Difference = \frac{X_2 - X_1}{X_1} x\ 100$$

where: X_1 = known value (*e.g.*, SRM certified value)
X_2 = determined value (*e.g.*, SRM concentration determined by analyst)

Relative Percent Difference

The relative percent difference (RPD) is a measurement of ***precision***; it is a comparison of two similar samples (matrix spike/matrix spike duplicate pair, field sample duplicates). The formula for calculating RPD is:

$$RPD = \left| \frac{2\ x\ (X_1 - X_2)}{(X_1 + X_2)} \right| x\ 100$$

where: X_1 is concentration or percent recovery in sample 1
X_2 is concentration or percent recovery in sample 2

Note: Report the absolute value of the result -- the RPD is always positive.

Relative Standard Deviation

The relative standard deviation (RSD) is a measurement of ***precision***; it is a comparison of three or more similar samples (*e.g.*, field sample triplicates, initial calibration, MDLs). The formula for calculating RSD is:

$$\%RSD = \frac{Standard\ Deviation\ of\ All\ Samples}{Average\ of\ All\ Samples}\ x\ 100$$

Table A–10. Navy Environmental Data Transfer Standard (NEDTS) Data Qualifiers.

	Data Qualifiers
A	Indicates that the Tentatively Identified Compound (TIC) is a suspected aldol condensation product
B	Blank contamination: The analyte was detected above the reporting limit in an associated blank. *For this study, blank contamination indicates that the analyte was found in both a sample and the associated blank. The "B" will be reported on the result associated with the field samples, not the blank*
C	Presence confirmed by GC/MS (Pesticides only)
D	Dilution run. Initial run outside linear range of instrument
E	Estimate, result outside linear range of instrument. GC/MS only
J	Estimated value: The analyte was positively identified but is less than the sample-specific reporting limit; the quantitation is an estimation. (Other values may be estimated due to matrix interference).
R	Rejected
S	Reported value determined by Method of Standard Additions (MSA)
U	Undetected at the method detection limit: The associated data value is the method detection limit, adjusted by any dilution factor used in the analysis (i.e., the sample-specific MDL).
W	Post-digestion spike out of control limits
X	Indicates manual modification of result or EPA qualifier
	Quality Control Qualifiers
Q	One or more quality control criteria (for example, LCS recovery, surrogate spike recovery) failed. Data usability should be carefully assessed by an individual experienced in data review who represents the data user or the user's agent.
*	Duplicate analysis not within control limits
+	Correlation coefficient for the MSA is less than 0.995

Table A–11. Maintenance Procedures for Field and Laboratory Equipment.[1]

Equipment	Activity	Frequency
Field Equipment		
Magellan Promark	none	Not applicable
Surveyor Tape	none	Not applicable
Refractometer	Rinse with MilliQ water	Initially, finally, and between samples.
Laboratory Equipment		
Deionized water system	Replace seals Replace cartridges	As needed for leaks and to maintain resistivity > 18 mOhms
MilliQ deionized water system	Replace seals Replace cartridges	Every 6 months or as needed for leaks and to maintain resistivity > 18 mOhms
Cahn balances	Clean	As needed
Electronic balances	Clean	As needed
Freezers/refrigerators	Clean Defrost	As needed
Ovens	Clean	As needed
Glass thermometers	Store in protective case	Always except when in use
Digital thermometer	Avoid bending thermocouples	Always
Conductivity meter	Remove batteries when inactive Replace batteries Clean electrodes	At least annually

[1]See Attachment A for STL-Burlington equipment maintenance procedures and frequency.

Table A–12. Maintenance Procedures for Analytical Instruments.

Equipment	Activity	Frequency
GC/MS Maintenance (SOP 3-092)		
Rough pumps Turbomolecular pump Diffusion pumps	Routine service (service contract) Check fluid levels	Six months Weekly
Foreline traps Helium gas traps	Inspect trap pellets for color change Replace adsorbent pellets	Routinely 6-12 months, as needed
Injection port septum	Replace	As needed to maintain EPC pressure
Injection port liners	Replace	Approximately every 30-40 samples
Precolumn	Replace	As needed to improve peak shape, resolution, or sensitivity
Calibration vial (PFTBA)	Refill	4 months or as needed
Back grills of the MS	Vacuum dust	6 months or as needed
Ion source	Clean	As indicated when usage-dependent surface deposits degrade ion source function
GC Maintenance (SOP 3-116)		
Injection port	Replace	Weekly (~50 injections) or as needed
Injection port liner	Replace	Weekly or as needed
Injection port	Clean	Monthly or as needed
Column	Clip	As needed to maintain performance
Precolumn	Replace	As needed when chromatographic degradation is observed
Gas cylinders	Replace	When PSI is < 300
Autosampler rinse vial	Fill	Prior to analysis
Autosampler syringe	Replace/align	As needed
Ferrule	Replace	As needed for leaks
Gas drying/purification traps	Replace	Annually or as needed
Column, detector	Bakeout	As needed
ICP-AES Maintenance (MSL-I-027)		
Pump tubing	Check and replace	Daily
Diluent bottle	Check and refill	Daily
Torch	Check and clean or replace	Weekly
ICP-MS Maintenance (MSL-I-022)		
Argon supply	Check and record; replace as needed	Daily
Vacuum	Check and record	Daily
Cooling chiller	Check and record temperature	Daily
Nebulizer flow	Check and adjust	Daily or as needed
Sensitivity and stability	Check and record	Daily

Table A–12. Maintenance of Analytical Instruments (continued).

Equipment	Activity	Frequency
Auto sampler tubing	Change	As needed
Cones	Clean or change	As needed
GFAA Maintenance (MSL-I-029)		
Graphite furnace tube	Check and replace (~500 burns)	Daily and as needed
Contact cylinders	Check and replace as needed (10,000 burns)	Daily and as needed
Windows	Clean	Whenever tubes are changed or as needed
Water recirculator fluid level	Check and refill	Daily
CVAA Maintenance (MSL-I-016)		
Soda lime	Check and change	Checked daily, changed weekly
Reagents (SnCl,3% HNO_3, rinse water)	Check and change	Checked daily, changed weekly
Carbon trap	Check and change	Checked daily, changed bimonthly
Filters	Check and change	Checked daily, changed bimonthly
Sample injection syringe	Check and change	Checked weekly, changed as needed
Tubing	Check and change	Checked weekly, changed as needed
Connectors	Check and change	Checked weekly, changed as needed
Lamp	Check and change	Checked weekly, changed as needed
Autosampler arm	Lubricate	Bimonthly
HGAA- FIAS Maintenance (MSL-I-030)		
Autosampler alignment	Check and change	Checked daily, changed as needed
Autosampler cleaning/waste resevoirs	Check and change	Checked daily, changed as needed
Fumehood Operation	Check	Checked daily
Argon airflow to manifold	Check and optimize	Checked daily, changed as needed
Lamp alignment and gain setting	Check and change	Checked daily, changed as needed
All flow tubing	Check and change	Checked daily, changed as needed
Reagents (carrier and reductant solutions)	Check and change	Checked weekly, changed as needed
STL-Burlington Instruments for TOC and Grain Size		
	See Attachment A	
SSC SD Instruments for XRF Screen		
Energy	Check calibration	Daily
Cu-stability	Check stability	Bi-monthly
SSC SD Instruments for Immunoassay Screen		
Lamp	Check linearity	Daily with standard series

Table A–13. Calibration Procedures for Field and Laboratory Equipment.

Measurement	Instrument	Calibration Procedure (Accuracy Requirement)	Frequency
Field Equipment			
Latitude/longitude	Magellan Promark	Verify vs. benchmark (± 10m - See Section A.3.7.1) Check vs. position with historical data (± 10 m - See Section A.3.7.1)	Prior to the survey Daily
Salinity	Refractometer	Verify "zero" reading using MilliQ deionized water	Prior to each survey day.
Ammonia	Hach®Kit	None	Not applicable
Water depth	Surveyor Tape	Lead line or bar-check (± 0.1m)	Prior to the survey
Laboratory Equipment			
Grain size	Analytical balance	Calibrate vs. NIST-traceable standards	Annually by outside contractor; daily using weights that bracket the expected sample range
Percent moisture	Analytical balance	NIST traceable standards	Annually and daily

Table A–14. Calibration of Laboratory Instruments.

Instrument	Standard Sources	Initial Calibration			Calibration Verification		
		No. Standard	Criteria	Frequency	Standards and Conc. Range	General Criteria	Frequency
GC/ECD SOP 5-128	Supelco, Inc. Accu-Standard, Inc. Ultra Scientific Chem Service, Inc.	≥5 for pesticides and PCB Congeners	Quadratic r ≥0.99 (3-point) r≥0.995 (5-point) Average Response Factor <25% RSD in initial for each target compound	Initially and after failure of continuing calibration	1 Mid-level calibration standard	≤25% from true check standard concentration	Every 10-12 samples (24 hours)
GC/MS SOP 5-157		≥3	Average Response Factor[1] ≤25% RSD for each analyte and average RSD for all analytes ≤ 15%		1 Mid-level calibration standard	≤25% from initial calibration average RF for each analyte and average difference for all analytes ≤ 15%	Every 12 hours
GFAA MSL-I-029	High Purity Standard	≥3 (plus blank)	r > 0.995		1 ICV 1 CCV	≤15% from true standard concentration	Every 10 samples
HGAA-FIAS MSL-I-030		≥3 (plus blank)	r > 0.995		1 ICV 1 CCV	≤15% from true standard concentration	Every 10 samples
ICP/AES MSL-I-027		≥3 (plus blank)	r > 0.995		1 ICV 1 CCV	≤15% from true value	Every 10 samples
ICP/MS MSL-I-022		≥3 (plus blank)	r > 0.995		1 ICV 1 CCV	≤15% from true value	Every 10 samples
FIAS		≥5 (plus blank)	r > 0.995	Daily	1 IPR 1 OPR	≤21% from true value	Every 10 samples
TOC		See Attachment A					
XRF EPA 6200 Mod	CRNC and NIST	5	r>0.98	Initially and after failure of continuing calibration	NIST #2704, 2709, 2710, 2711, CNRC, PAC-1	± 20% from true value	Every 20 samples
Rapid Assay EPA 4020 Mod	SDI	≥3 (plus blank)	r>0.98		SDI Aroclor 1254 At 3 ppb	± 20% from true value	Every 20 samples
Rapid Assay EPA 4035	SDI	≥3 (plus blank)	r>0.98		SDI Phenanthrene at 25 ppb	± 20% from true value	Every 20 samples

[1]A PFTBA tune will be performed prior to initiation of a new sample sequence.

Table A–15. Essential Electronic Data Deliverable (EDD) Parameters.

FIELD RECORDS	LABORATORY RECORDS
Study ID	Lab Sample ID
Survey ID	Lab
Sample ID	Lab Batch
Client Sample ID	Lab Receipt Date
Field Sample Type	Analysis Date
Field QC Code	Analysis Method
Associated Sample ID	Method Citation
Station ID	Extraction Method
Station Type	Extraction Date
Station Type 2	Clean-up Method
Facility	Instrument Run ID
Area Name	Sample Type
Media	Rep
Gear Type	SubRep
Sample Collector	Fraction
Date Start	Sample Weight/Volume
Depth Top	Sample Weight/Volume Unit
Depth Bottom	Percent Moisture
Depth Unit	Parameter Class
Water Depth	Parameter Code
Water Depth Unit	Parameter Name
Field Sample Weight/Volume	Lab Qualifier
Field Sample Weight/Volume Unit	Result
Latitude	Result Unit
Longitude	MDL
X Y Datum	RDL
	Validation (Final) Qualifier
	Dilution
	Validation Level
	Validation Date

Table A–16. Level III Data Validation Assessment Parameters.

Criteria	PAH (GC/MS)	Pesticides/ PCBs (GC)	Trace Metals	Wet Chemistry
Holding times	X	X	X	X
Instrument tunes	X			
Initial and continuing calibrations	X	X	X	X
Blanks	X (5X/10X rule)	X	X	X
LCS/ Laboratory set limits	X		X	X
Surrogates	X	X		
MS/MSD	X	X	X	X
Duplicates			X	X
Internal standard area performance	X			
Target compound retention times		X		
Instrument performance		X		
Interference with compound quantification		X		

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ATTACHMENT A

Analytical Documentation
Severn-Trent Laboratory – Burlington

METHOD: ASTM D422
STANDARD OPERATING PROCEDURE
FOR PARTICLE SIZE ANALYSIS OF SOILS
Applicable Matrix or Matrices: Soil, Sediment, Sludge
Standard Compound List and Reporting Limits: NA

Approvals and Signatures

Laboratory Director:	Christopher A. Ouellette	Date: 5-12-00
QA Manager:	Kim B. Watson	Date: 5-12-00
Inorganics Technical Director:	Kristine A. Dusablon	Date: 5-12-00
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1.0 SCOPE AND APPLICATION

1.1 This method determines the particle size distribution in soil. Particles greater than 75um (gravels to fine sands) are determined by sieving and particles less than 75um (silts and clays) are determined by sedimentation using an hydrometer.

1.2 Minimum quantity of sample depends on subsequent analyses to be performed. Typical range is 150 to 350 grams of dry soil. Larger amounts (from 500 to 5000 grams) are specified for particle size analysis of soils with appreciable gravel component.

1.3 This preparation is amenable to samples containing sand, silt, clay and gravel.

2.0 SUMMARY OF METHOD

2.1 Soils for particle size analysis are prepared according to ASTM D421 or D2217. The soils are sieved in two steps. The particles greater than 2.00mm (retained on the No. 10 sieve) are sieved after the soil has been prepared. A portion of the soil passing the No. 10 sieve is prepared for hydrometer measurements. Seven hydrometer readings are made over a 24 hour time frame. The soil in the hydrometer is rinsed on a No. 200 (75 um) sieve and dried for sieve analysis of material less than 2.00mm (No. 10 sieve). Calculations are made to determine the percent finer of soil for each sieve and hydrometer reading. These calculations are dependent on percent solid, which is determined during the drying process, and the specific gravity that is assumed to be 2.65 (unless separate analysis is requested for specific gravity).

3.0 DEFINITIONS

N/A

4.0 INTERFERENCES

N/A

5.0 SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

5.2 STL Burlington maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. Material Safety Data Sheets (MSDS) are made available to all personnel involved in the chemical analysis. STL Burlington also has a written environmental health and safety plan.

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5.3 Please note chemicals that have the potential to be highly toxic or hazardous, the appropriate MSDS must be reviewed by the employee before handling the chemical.

6.0 EQUIPMENT AND SUPPLIES

6.1 Balance sensitive to 0.01 grams

6.2 Mixer and dispersion cup

6.3 1000 ml sedimentation cylinder

6.4 Soil test hydrometer meeting specification E 100

6.5 Mortar and rubber tipped pestle for breaking up soil aggregates

6.6 Sieves of the following size:

3.0 in (75.00mm)	No. 20 (850.0um)
2.0 in (50.00mm)	No. 40 (425um)
1.5 in (37.50mm)	No. 60 (250.0um)
1.0 in (25.00mm)	No. 80 (180.0um)
3/4 in (19.00mm)	No. 100 (150.0um)
3/8 in (9.50mm)	No. 200 (75.0um)
No. 4 (4.75mm)	
No. 10 (2.00mm)	

6.7 Oven with temperature range of 60° C to 110° C

6.8 Thermometer accurate to 0.5° C

6.9 Timer with second hand and capable of counting up to 25 hours

6.10 Mixing utensils, metal and bristle brushes for sample recovery.

6.11 Rototap machine

7.0 REAGENTS AND STANDARDS

7.1 Sodium Hexametaphosphate (dispersion reagent)

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8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1 Typical sample of 150 to 350 grams is used for analysis. Larger amounts (from 500 to 5000 grams) are specified for particle size analysis of soils with appreciable gravel component. The sample container must remained sealed to maintain natural water content.

8.2 There are no holding time requirements.

9.0 QUALITY CONTROL

9.1 Check balance daily with Class S weight, yearly manufacturer calibration.

9.2 Oven temperature is checked daily prior to start of work.

9.3 Thermometer is checked against similar or more accurate temperature device.

9.4 Duplicate samples are recommended every 20 samples.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Sieves calibrated twice a year using the National Bureau of Standard, Certificate of Calibration, standard reference materials 1017a, 1018a and 1019a calibrated glass beads.

10.2 Hydrometers are calibrated twice a year, and checked prior to each use.

10.3 Thermometer calibrated against NIST certified thermometer.

11.0 PROCEDURE

11.1 Large Sieve (dry): The soil retained on the No. 10 sieve is used in this step. Rinse the particles on a No. 10 sieve and then place the material in an oven until dry.

Large Sieve (wet): Take the equivalent of 200 grams of dry soil (use the percent solid table). Place soil on a No. 10 sieve and wash the soil. Take the soil retained on the No. 10 sieve and place in an oven until dry.

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11.1.1 Record the weights of the sieves greater than No. 10. Take the dry soil and pour into the sieve stack. Place the sieve stack on the Rototap machine and shake sample for ten minutes.

11.1.2 Weigh and record the contents of each sieve.

11.1.3 Record the maximum particle size. Determine the hardness of the particles by dropping a hammer on the particle from a height or approximately one foot. Record the hardness as hard, soft or brittle. Save the soil particles.

11.2 Hydroscopic Moisture (dry prep. only): The soil passing the No. 10 sieve is used in this step. Take a small tin, label it and record the weight. Place approximately 10 to 15 grams of soil in the tin. Place the tin in the oven at 110°C for at least 16 hours. Remove the tin and record the weight.

11.3 Hydrometer Test: The soil passing the No. 10 sieve is used in this step.

11.3.1 Sample Preparation:

- Dry Prep: Tare a 250 ml beaker. Place and record approximately 50 grams for silt or clay particles or 100 grams for same particles into the beaker. Add 125 ml of a 40g/l sodium Hexametaphosphate solution to sample and allow to soak overnight

  Wet Prep: Tare a 500 ml beaker. Place and record the dry equivalent (use the percent solid table) of approximately 50 grams for silt or clay particles into the beaker. Add 125 ml of a 40g/l sodium Hexametaphosphate solution to sample.

- Dry Prep: Rinse the sample with DI water into a dispersion cup. Fill the cup to the halfway mark with DI water and place cup on the blender. Mix sample for approximately one minute. Pour content of cup into a 1000 ml sedimentation cylinder. Rinse cup with DI water to wash all the sample into cylinder. Fill the cylinder to the 1000 ml line and cover with a sheet of paraffin wax.

  Wet Prep: Rinse the sample with DI water into a dispersion cup. Fill the cup to the halfway mark with DI water and place cup on the blender. Mix sample for approximately five minutes. Pour content of cup through a No. 10 sieve into a 1000 ml flask. Rinse cup with DI water to wash all the sample into the flask. Fill the flask to the 1000

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ml line and cover the flask with a sheet of paraffin wax. Take the material on the No. 10 sieve, dry it in the oven and record the weight.

11.3.2 After preparing up to 12 flasks, begin setup for hydrometer readings. The following paperwork is needed: hydrometer data sheet, hydrometer reading table, and temperature table if conversion from Fahrenheit to Celsius is necessary. Initiate timer to indicate the elapsed time, counting up from zero. Check readings of hydrometer and temperature probe in a DI water rinse bath. Get the rubber stopper to shake flask and prepare staging and test areas.

11.3.3 Initiate timer to indicate the elapsed time. The hydrometer reading table is used to perform activities as indicated (shake, place or read) for each 1000 ml cylinder.

A reading consists of inserting the hydrometer gently into the cylinder, (after the cylinder has been shaken for 1 minute), about 20 seconds before the actual reading. Read the hydrometer to the nearest 0.0005 at the top of the meniscus. Remove the hydrometer and insert a temperature sensor into the cylinder to the depth to which the hydrometer reached. Read the temperature meter to the nearest 0.1°C and remove the temperature sensor. The hydrometers and temperature sensor are rinsed in a DI bath between each reading.

After each cylinder is read, the hydrometer reading, temperature, and time (from table) is entered on the hydrometer data sheet at the corresponding cylinder (test) number and time portion on the data sheet; deviations from the table schedule are noted on the sheet. The readings are taken at 2, 5 and 15 minute and at 30, 60, 240 and 1440 minutes.

11.4 Small Sieve: Soils from the hydrometer test are rinsed on the No. 200 sieve. The soil retained on the No. 200 sieve is placed in an oven and dried over night.

11.4.1 Record the weights of the sieves used between No. 10 and No. 200. Take the dry soil and pour into the sieve stack. Place the sieve stack on the Rototap machine and shake sample for ten minutes.

11.4.2 Weigh and record the contents of each sieve.

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12.0 CALCULATIONS

12.1 Percent Solids (PS) and Hydroscopic Moisture Correction Factor (HMCF)

12.1.1 HMCF is used for air dried samples (dry prep.)

*HMCF = (pan and baked sample - pan)/(pan and dry sample -pan)*100*

12.1.2 Wet Method:

*PS = (pan and dry sample- pan)/(pan and wet sample - pan)*100*

Dry Method:

*PS = HMCF * (pan and dry sample -pan)/(pan and wet sample - pan)* 100*

12.2 Sample Used (SU):

12.2.1 Wet Method:

SU = (pan and wet sample - pan) PS*

Note: for hydrometer SU, subtract the dry weight of any material retained on the No. 10 sieve.

12.2.2 Dry Method:

*SU = ((pan and dry sample - pan) - (pan and non-soil material - pan)) * HMCF*

12.3 Sieve Analysis (Percent Finer = PF)

12.3.1 Large Sieves:

- 3 inch: PF = 100-100* (Sieve and Sample (3 inch) - Sieve (3 inch))/SU
- 2 inch: PF = PF (3 inch) - 100*(Sieve and Sample (2 inch) - Sieve (2 inch))/SU and so on through the #10 Sieve.

12.3.2 Small Sieves

- #20: PF = PF(#10) - 100*(mass passing #10/sample mass (Hyd))*(sieve and sample (#20) - sieve(#20))/sample used
- #40: PF = PF (#20) - 100*(mass passing #10/sample mass (Hyd))*(sieve and sample (#40) - sieve (#40))/sample used and so on up through #10 sieve.

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12.4 Hydrometer Analysis

12.4.1 Particle size, Micron

*1000*sqrt [930*viscosity/980*(SG-1))*(effective depth/time)]*

Viscosity at sample temperature, poises

- Effective Depth, cm = 16.29-264.5*(actual Hydrometer reading - 1) above equation for effective depth based on equation found with table 2 in method, in which 16.29 = 0.5*(14.0-67.0/27.8)+10.5 and 264.5 = (10.5-2.3)/0.031
- Time, minutes = Time of hydrometer reading from beginning of sedimentation

Sqrt - square root
SG - Specific Gravity of soil
Viscosity - is the resistance of a liquid to flow

12.4.2 Percent Finer (PF):

PF = Constant(actual hydrometer reading - hydrometer correction factor - 1)*

Where:
Constant = (100,00/W)*SG/(SG-1)
W = (Total sample used *sample used for hydrometer analysis*HMCF)/Amount of total sample passing #10 sieve
Hydrometer Correction = slope*sample temperature + Intercept
Slope = ((low temp. reading -1)-(high temp. reading -1)/(low temp. - high temp.))
Intercept = (low temp. reading -1) - (low temp. * slope)

13.0 METHOD PERFORMANCE

N/A

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has

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established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

14.2 The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

14.3 For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction", available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington, D.C. 20036; (202) 872-4477.

15.0 DATA ASSESSMENT AND CRITERIA AND CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

15.1 Data is initially reviewed by the analyst in the lab. Following this, the data is secondarily reviewed by QC personnel before being put into its final data package form (where the data is thirdly reviewed before being sent to the client).

15.2 Data that is out of control is marked as such and slated for re-analysis. Any corrective action undertaken is documented on a corrective action form (detailing the client information, problem, investigation findings and solution). This form is kept together with the project.

16.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

16.1 Generally, any data that is out of control is considered unusable. There are, however, cases in which laboratory supervisor will be made aware of the issue and, if the data is used, it will be thoroughly narrative noted.

17.0 WASTE MANAGEMENT

17.1 The USEPA requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. Excess reagents, samples, and

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method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water and land by minimizing and controlling all releases from hoods, and bench operations, complying with the letter and spirit of any waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel", available from the American Chemical Society at the address listed in Section 14.3.

18.0 REFERENCES

18.1 Annual Book of ASTM Standards, volume 04.08 Soil and Rock (I): D 420 - D4914, Section 4, Construction edition; American Society for Testing and Materials, Philadelphia, Pa., 1994.

19.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION FORMS

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DETERMINATION OF TOTAL ORGANIC CARBON IN SEDIMENT
LLOYD KAHN METHOD
STANDARD OPERATING PROCEDURE MODIFIED

Applicable matrix or matrices: Soil

Standard Compound List and Reporting Limits: Total Organic Carbon, 100 mg/Kg

Approvals and Signatures

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1.0 SCOPE AND APPLICATION

1.1 This method describes protocols for the determination of organic carbon in sediments.

1.2 The detection limit may vary with sample type however the typical reporting limit is 100 mg/Kg as received.

1.3 Data are reported on a mg/Kg dry weight basis and represent the average

2.0 SUMMARY OF METHOD

2.1 Inorganic carbon from carbonates and bicarbonates is removed by treatment with phosphoric acid.

2.2 The organic compounds are decomposed by pyrolysis in the presence of oxygen.

2.3 In the Carlo Erba Model EA1108 CHN Analyzer, the sample is pyrolyzed in a inductive type furnace, and the resultant carbon dioxide is chromatographically separated and analyzed by a differential thermal conductivity detector.

2.4 Water content is determined on a separate portion of sediment.

3.0 DEFINITIONS

3.1 Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV): A solution of one or more method analytes, surrogates, internal standards, or other test substances used to evaluate the performance of the instrument system with respect to a defined set or criteria.

3.2 Initial Calibration Blanks (ICB) and Continuing Calibration Blank (CCB): A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes, internal standards, or surrogate analytes.

3.3 Calibration Standard (Curve): A solution prepared from the primary dilution standards solution or stock standard solutions and the internal standards and surrogate analytes. The solutions are used to calibrate the instrument response with respect to analytes concentration.

3.4 Matrix Spike (MS) and Duplicate (DP): A duplicate sample is a sample brought through the whole sample preparation and analytical process. The duplicate sample is used to document the precision of a method in a given sample matrix.. A matrix spike sample is an aliquot of an environmental sample to which known qualities of the method analytes are added in the laboratory. The laboratory fortified matrix is analytes exactly like a sample, and its purpose is to determine whether the sample matrix

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contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.

3.5 Laboratory Control Sample (LCS): A solution of method analytes of known concentrations that is used to fortify sample matrix. The LCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

3.6 Method Prep Blank (PB): An Analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method prep blank should be carried through the complete sample preparation and analytical procedure. The method prep blank is used to document contamination resulting from the analytical process. For the method prep blank to be acceptable for use with the accompanying samples, the concentration of any analyte of concern in the blank should not be higher than the reporting limit.

3.7 Method Detection Limit (MDL) Instrument Detection Limit (IDL): The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

4.0 INTERFERENCES

4.1 Volatile organics in the sediments may be lost in the decarbonation step resulting in a low bias.

4.2 Bacterial decomposition and volatilization of the organic compounds are minimized by maintaining the sample at 4 °C, analyzing within the specified holding time, and analyzing the wet sample.

5.0 SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

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5.2 STL maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in these methods. Material Safety Data Sheets (MSDS) are made available to all personnel involved in the chemical analysis. STL also has an ongoing safety plan.

5.3 The following chemicals have the potential to be highly toxic or hazardous, consult MSDS.

- Phosphoric Acid
- Hydrochloric Acid

6.0 EQUIPMENT AND SUPPLIES

6.1 Drying oven maintained at 103 °to 105 °C.

6.2 Carlo Erba Elemental Analyzer model 1108.

6.3 A permanent recording device is present to document the analysis.

7.0 REAGENTS AND STANDARDS

7.1 Acetanilide and Sulfanilamide crystals purchased commercially.

7.2 Phosphoric acid solution, 1:19 by volume.

7.3 Hydrochloric Acid.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

Collect sediments in glass jars with Teflon or aluminum foil. Cool and maintain at 4 °C. Analyze within 14 days.

9.0 QUALITY CONTROL

9.1 An ICV/CCV (Acetanilide) must be analyzed every 10 drops and be within 90-110% of the true value.

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9.2 An ICB/CCB must be analyzed every 10 drops and be less than the detection limit.

9.3 In January of each year, a well mixed, homogeneous sample is prepared and a series of 15 determinations is analyzed. The standard deviation of this series is determined and posted with the instrument. (See 9.4)

9.4 For each prep batch, a commercially purchased soil LCS must be prepared in quadruplicate. Calculate and highlight the standard deviation. If the standard deviation for the LCS exceeds the standard deviation limit (as determined in Section 9.3), identify error and rerun all environmental samples in that batch.

9.5 The average of the triplicate results will be reported for each sample unless there is an obvious problem or error associated with a single result. In that event, the average of the remaining two results will be reported. Unless specifically requested, results of a single determination will not be reported.

9.6 Samples will be spiked with an NIST certified carbon source. The spike material will be added prior to the removal of the inorganic carbon. All spiked samples must be prepared in duplicate and spike recovery should be 75-125%.

10.0 CALIBRATION AND STANDARDIZATION

Follow Carlo Erba SOP for calibration procedure using sulfanilamide.

11.0 PROCEDURE

11.1 Thoroughly mix and transfer 5-10 mg to a tared tin capsule.

11.2 Weigh sample to nearest microgram. Record weight in Run Log.

11.3 Place tin capsule/sample into a sample holder (one of two flat bars drilled with 15 numbered wells). Record well number in Run Log.

11.4 Weigh all samples to be prepared, filling both sample holders.

NOTE: All samples are to be prepared in triplicate. Additionally, a soil

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LCS must also be prepared in quadruplicate for quality control requirements (See Section 9.4)

11.5 Add 1-2 drops 1:19 H_3PO_4 to each capsule. Check for effervescence. Add H_3PO_4 until all signs of effervescence are gone.

NOTE: This procedure will convert inorganic carbonates and bicarbonates to carbon dioxide and eliminate it from the sample.

11.6 Maintain at approximately 103 °C for 1 hour.

11.7 Crimp top of tin capsule and place into autosampler of Carlo Erba CHNS-O Elemental Analyzer.

11.8 Enter sample ID's and weights in Sample Table Menu. Refer to Appendix B for sample table entry and actual analysis.

11.9 Analyze the residue according to the instrument manufacturer's instructions.

11.10 Determine percent residue on a separate sample aliquot.

11.11 A summary page must be presented for each sample indicating the average percent carbon. Convert this result to mg/Kg as received by multiplying by 10,000. The Percent Relative Standard Deviation (% R.S.D.) also appears on the summary page, giving a measure of the results' range. The smaller this value, the closer the triplicate results, although it should be remembered that low results, with very little scatter may still yield a high % R.S.D. The % R.S.D. for the LCS is monitored by control chart as an indication "ideal" scatter.

12.0 CALCULATIONS

12.1 1% OF 1,000,000 $^{mg}/_{Kg}$ = 10,000 $^{mg}/_{Kg}$

$$X\%\,Carbon = \frac{X\,10{,}000\,mg\,/\,Kg}{1\%} = X\,mg\,/\,Kg\,wet\,carbon$$

12.2 Matrix Spike:

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- $\frac{A}{B} = C$

- $D \times E = F$

- $\frac{F \times 100}{G} = H$

- $\frac{H \times 10{,}000}{1\%}\ {}^{mg}/_{Kg} = I$

- $\frac{C - J}{I} \times 100 = \%\ \text{Recovery}$

where:
A = $^{mg}/_{Kg}$ wet carbon
B = % Solid
C = $^{mg}/_{Kg}$ dry carbon
D = True value of spike
E = Spike wt. Added
F = Spike Added
G = Sample wt. (mg)
H = % spike added
I = $^{mg}/_{Kg}$ of dry spike added
J = $^{mg}/_{Kg}$ dry carbon from parent sample

13.0 METHOD PERFORMANCE

N/A

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source,

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the Agency recommends recycling as the next best option.

14.2 The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

14.3 For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction", available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington, D.C. 20036; (202) 872-4477.

15.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA AND CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

N/A

16.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

N/A

17.0 WASTE MANAGEMENT

The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. Excess Reagents, samples, and method process wastes should be characterized and dispose of in an acceptable manner. The Agency urges laboratories to protect the air, water and land by minimizing and controlling all release from hoods and bench operations, complying with the letter and spirit of any waste regulations, particular the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel", available from the American Chemical Society at the address listed in Section 14.3.

18.0 REFERENCES

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18.1 Standard Method for the Examination of Water and Wastewater, 19th edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C., 1995.

18.2 Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.

18.3 Lloyd Kahn, Quality Assurance Specialist, U.S. Environmental Protection Agency, Region II, Determination of Total Organic Carbon in Sediment, July 27, 1988.

19.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

N/A

20.0 MODIFICATIONS

20.1 1:19 Phosphoric Acid

20.2 5-10 mg Sample weight.

20.3 Heat 1 hour at 103°C.

20.4 Three results averaged.

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Appendix A
Marine Sediments High in Organic Carbon

1.0 SAMPLE PREPARATION

1.1 Record weight of beaker (100ml), then transfer approximately 5 grams of a thoroughly mixed sample.

1.2 If the sample is to be spiked, weigh the beaker to the nearest 0.1 mg. Record the weight. Likewise, determine and record the weight of the added sample. Now add 0.4-0.5 grams of NIST 1632b Trace Elements in Coal (80.11% Carbon) to the sample. Record this weight as well. Evenly distribute the spike over the sample when adding, rather than make one big pile and use a glass stirring rod to mix the spike in with the sample (make sure not to use that stirring rod with any other sample!).

1.3 Talc-free latex gloves should be worn from this point on to minimize the risk of acid burns. Add several drops of 1:1 HCL to each sample and stir each with its own glass stirring rod. Samples containing large concentrations of inorganic carbon may effervesce to the point of overflowing the beaker, so take care to add the acid in small aliquots and stir vigorously. Should the sample "boil over" it must be re-prepared from step 1.

1.4 Continue to add 1:1 HLC in small aliquots until there is no further reaction.

1.5 Very carefully rinse the stirring rod and beaker walls with DIH_2O. Use a fine-tipped squirt bottle and use only what is needed to bring all of the sample to the bottom of the beaker. Remember, you are "adding water to acid" so take all necessary precautions to avoid splashing!

1.6 Dry the treated samples on hot plate in the hood.

1.7 Weigh beaker with residue and record. NOTE: Samples are hydroscopic and will absorb water if they are exposed to air for too long.

1.8 Once thoroughly dried, scrape the sample residue from the beaker and grind it to a powder using the pink mortar and pestle reserved for this purpose (it has a much smoother surface than most other mortars, thus reducing contamination to or carryover). Transfer the ground sample to a clean, dry 40 mL vial to await analysis.

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NOTE: Depending on the nature of the sample, it may be quite difficult to completely remove the dried residue from the beaker or to grind it to a homogenous powder. Where difficulties are encountered, make a note on the preparation worksheet. Also, it is comparatively more important to remove and grind all of a spiked sample, otherwise percent recoveries may be effected.

1.9 The samples are now ready for analysis by the Carlo Erba Elemental Analyzer (see Appendix B), it needs no further treatement and the instrument results will be "dry weight".

2.0 CALCULATIONS

2.1 1% of 1,000,000 mg/kg = 10,000 mg/kg

2.2 $\frac{\text{x \% carbon x 10,000 mg/kg carbon}}{1\%}$

2.3 Value to be recorded = x mg/kg carbon x dry/wet ratio = mg/kg.

2.4 Matrix Spike

2.4.1 Sample (actual) where c = carbon, r = residue, w = wet wt., d = dry wt.

$$X\ {}^{mgc}/_{Kgr}\ X\ (\text{dry wet ratio})\ {}^{Kgr}/_{Kgw} = {}^{mgc}/_{Kgw}$$

$$X\ {}^{mgc}/_{Kgw}\ X\ \frac{100Kgw}{(\%\ \text{solid})}\ {}^{mgc}/_{kgd} = \text{actual reported value}$$

2.4.2 Spike (theoretical) where: c = carbon, r = residue, w = wet, d = dry wt.

(spike added mg) (true value of spike) = x mgc

$$\frac{X\ {}^{mgc}}{Y\ {}^{mgw}}\ X\ \frac{100\ mg_w}{(\%\ \text{solid})\ {}^{mgd}} = z\ {}^{mgc}/_{mgd}$$

where: x = spike added (wet)
z = sample wt. (mg)

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xy = spike theoretical value

2.4.3 $\% \text{ Recovery} = \frac{\text{actual - parent}}{\text{theoretical}} \times 100$

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Appendix B
Operating the Carlo Erba Elemental Analyzer
For Carbon and Sulfur Analysis in Soil.

1.0 INSTRUMENT SET-UP

1.1 The power switch is the white handle behind the right side of the machine. This is not usually touched, except in times of malfunction. The instrument is generally left on.

1.2 Left furnace temperature dial should read 102. Press "FURN STBY" button so that it is in the out position. ("Out is On , In is Off").

1.3 Turn on UHP Oxygen at the wall. Turn carrier gas knob from "st by" to flow. Turn purge knob from off to on. Pressure gauges should read, from left to right: 95, 60, 90, 350 kPa.

1.4 When the LED temperature readout is 1020, press FIL OFF button so that it is in the out position. ("Out is On , In is Off").

1.5 Turn the autosampler tray until the number 1 is behind the post, in front of the autosampler.

1.6 Turn on the computer monitor. The main menu of the EAGER200 program should be shown. If not, make it so.

1.7 The Carlo Erba is calibrated with each new column (Refer to Section 3: Changing the Column and Section 4: Calibration), and that calibration is used until either quality control or the column fails. Typically the column holds for approximately 200 drops. The instrument is left in stand-by between analytical batches.

2.0 LOADING SAMPLES

2.1 Make sure that previous analysis has been saved by clicking mouse on "save" box. The date of the last analysis {i.e., CHNS1120.EAD) should appear in the highlighted box. If it does not, enter date of previous analysis and click on confirm. If the date does appear, click on confirm anyway and confirm the overwrite statement.

2.2 Change date in "METHOD IN USE" box to current date.

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2.3 Click mouse on "EDIT" box of sample table menu. If sample table menu is not on bottom of screen, click on the sample table box itself to bring it there.

2.4 On the sample table grid, go up and click on the "edit row" box on top left of screen. Then click on "delete samples table" box. The table should now be empty except for the word "bypass" in the type column.

2.5 Click mouse on the little square to the right of the number one. A box displaying "first sample/last sample" will appear. Select "first sample" (an "F" will appear in the little box). Enter "Blank" in the sample box and enter the filename, ie C112401 (C: Carbon, 1124: MMDD, 01: Position in Sample Queue; use "S" for Sulfur Analysis). Under sample type, enter "UNK" (Unknown), and enter the default weight of 30 mg. Repeat this for position number two.

2.6 Crush an empty tin capsule, pressing into a small cube. Use the two forceps reserved for this analysis. Place into position one on the sample tray. Repeat for the second position.

2.7 Place a tin capsule on the balance and tare. Remove the capsule and add a few crystals of Acetanilide (LCS for TOC; for sulfur analysis use 2,5 Bis (5-tert-butyl-benzoxazol-2yl)thiophene (BBOT)). Replace the capsule on the balance and record the weight of the acetanilide. This weight should be between 0.3 and 0.6 mg.

2.8 Enter data for the LCS in position three, again selecting "unknown" for sample type (you only use the other options during calibration). Pay attention to the filename; the date must be correct and the sample position must match the number at the far left of the row. Errors here may result in a sample file being "lost" in the computer, or worse, another file may be overwritten.

2.9 Crush the capsule by pinching the top closed, then folding it over. Press into a small cube using the forceps and place in position three.

2.10 Repeat this procedure for position four.

2.11 Load prepared samples into autosample tray and enter sample ID's, filenames and sample weights in the sample table. Remember that every 10 drops a series of two blanks and two LCS's must be analyzed. Remember to enter "last sample" at the end.

Note: When crushing the tin capsules of prepared samples, be aware that the phosphoric acid/heat (used in Organic Carbon analysis only) can make the tin

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brittle and easy to tear. Rather than use excessive force or pressure to produce the smallest package possible, concentrate on making a smooth, intact cube with nothing "sticking out" which might hang up the capsule in the sample tray or catch as the capsule drops into the combustion column. Should a capsule tear while compacting it, immediately place it into a new capsule and continue. Of course, should any sample be lost as the result of a tear or any other circumstance, that sample must be discarded and a new one prepared.

2.12 Once all data has been entered in the sample table, click the mouse on "update" and then "confirm" the changes to the sample table. When the main menu screen re-appears, click on "save", the save screen will appear. Check that "method in use" is in fact the method you want (if not, make it so), and "confirm" to save the method to disk. Now you won't lose the data in the event of a power failure. Now "print" the sample table for the run log.

2.13 Click on "run" in upper left of the screen, then click "start analysis". You should hear a click sound and a "whoosh" as the instrument begins its first cycle. Click on "run" again, then on "monitor detector". The detector screen will appear and a curser will be seen to draw the chromatogram.

2.14 To leave the monitor detector screen, click on the strange box in the extreme upper left corner and then on " quit monitor detector". The main menu screen will return.

2.15 Each run takes approximately 14 minutes, so after about 45 minutes, check the printout. The two blanks will have been run and must be <0.01% (<100 mg/Kg) to be acceptable. The first Acetanilide will have been analyzed and must be 71.09%C ± 7.1%; or for sulfur, BBOT range: 7.44% ± 0.74%. If the opening QC samples fail, abort the run (click on "run" then click on "abort analysis"). If the column has over 100 drops it may need to be changed, but if the column is fairly new it is worth preparing a new set of blanks and LCS and trying again. Remember to change the LCS weights in the sample table ("update" and "save"), before re-starting! Should this second set fail, abort the run, put the instrument in stand-by (See 2.18) and then turn down the furnace temperature in preparation for repacking the column.

2.16 When the run has finished, check that all QC is acceptable. Check that all sample "carbon areas" are below the area posted for the highest standard (any that exceed this limit must be reanalyzed using less sample).

2.17 "Save" the method, which now includes the processed chromatograms. If this is not done, a power failure (or turning off the computer) will wipe out all your data. Avoid this at all costs.

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2.18 If another batch of samples is not ready to load, or if the Carlo Erba will be idle for more than 8 hours, put the instrument in standby: push "FURNSTBY" and "FILOFF" buttons in; turn the carrier gas to "STBY", the purge to "OFF" and turn the UHP Oxygen off at the wall.

3.0 CHANGING THE COLUMN

A typical column will last for approximately 200 drops; however; a column may fail at any time. Samples high in sulfur especially will shorten a column's usefull life. The surest sign that a column needs replacing is failing blanks. Also, many small "noise peaks" may be observed in the last third of a run. To change the column:

3.1 Turn off the gases to the instrument.

3.2 Depress the "FURN STBY" and "FIL OFF" buttons to the "in" position. (Out is on, In is off)

3.3 Turn the dials in the upper left corner of the instrument panel for the left furnace temperature to 032. In a few hours, the temperature LED readout will read 253. Do not rush this. Replacing the column when the furnace is too hot will crack the new column.

3.4 Unscrew the autosampler from the top of the column.

3.5 Remove the panel covering the furnace and unscrew the bottom of the column.

3.6 Wiggle the nut and O-ring until they come off the column bottom.

3.7 Lift the column out of the furnace by the upper O-ring.

CAUTION: DO NOT TOUCH. 253°C is very hot!!!

3.8 Pack a new column using a clean (but not necessarily new) quartz column, and following "Figure 19" shown on the next page. Do not pack the quartz wool too tightly; it is there to keep the tungsten trioxide and copper wire separated and retained in the column. Packing too tightly will only serve to restrict the flow of gas through the system and may result in retention time shifts and missed peak identification.

3.9 Set temperature back to 102 and press "FURN STBY" to the out position.

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3.10 Once thoroughly cooled, the old column may be reamed out and used again. Before re-using, check carefully that there are no cracks or chips in the column (discard if any are found).

3.11 Calibrate the new column. Refer to Section 4.

4.0 CALIBRATION

4.1 To calibrate the Carlo Erba or carbon and sulfur analysis, start a new method as described in Section 2.1-2.4.

4.2 Positions one and two are used to establish an instrument baseline. Enter"Bypass" under sample type. Note that you cannot enter a weight for a bypass analysis.

4.3 Next enter two blanks. Select "Blank" under sample type, and place a crushed tin capsule in autosampler positions 3 and 4. Note that you cannot enter a weight for blank analysis either, but a tin capsule must be analyzed.

4.4 The standard curve itself follows. Entering "Standard" under sample type will prompt a dialog box listing all standards that the Carlo Erba is programmed to recognize. Select sulfanilamide and enter the weight. Crush the tin capsule. For the first standard, use 0.2-0.5 mg, for the second 1.0-1.5 mg and for the third, use 2.0-2.5 mg Sulfanilamide. Place the crushed capsules in their proper place in the autosampler tray.

4.5 Follow this calibration series with two blanks and two LCS drops. Remember to designate these as unknowns and enter weights as usual. An example of a calibration series is shown on the next page.

4.6 Mark first and last samples and analyze this entire series as full described in Section 2.

4.7 When analysis is complete, check that the "unknown" blanks and LCS's meet QC criteria. Check also that the "average K-factor" (shown in the third sulfanilamide standard printout) falls within the acceptable limits. If any of these criteria fail, the column must be recalibrated.

4.8 Again, refering to the third standard (position 9), highlight the area units obtained for both carbon and sulfur. Record and post these values by the Carlo Erba. Any sample which exceeds these limits must be repeated

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using a lower weight.

4.9 As a further measure of the linearity of the instrument, plot "weight of sulfanilamide" against "area response" for the three standards and determine the regression coefficient. This must be 0.995 or better. If the regression coefficient is <0.995, the column must be recalibrated. Record the regression coefficient at the bottom of the page (third standard again).

4.10 Initial and date the first page of the calibration series.

4.11 A copy of this calibration series, including the blank and LCS analyses, must accompany each set of data completed. The original is kept with the Carlo Erba, and is kept on file once the instrument is calibrated again. Remember to make all copies as dark as possible or they may be difficult to read.

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APPENDIX B

Response to Comments on the Thames River Rapid Sediment Characterization Pilot Study Draft Work Plan/Field Sampling Plan

U.S. Environmental Protection Agency, Region I
Response to May 14, 2003 Comments
Thames River Rapid Sediment Characterization Pilot Study
Final Work Plan/Field Sampling Plan

GENERAL COMMENTS

1. **The 1998 Ecological Risk Assessment (ERA) recommended a data gap evaluation for Zones 4 and 7. This Work Plan appears to fill the sediment sampling and analyses gaps identified in the RI. Data are to be combined with results of the RI to determine risk in Zones 4 and 7. The proposed numbers of sediment samples for the Rapid Sediment Characterization (RSC) should meet the Data Quality Objectives (DQO) for these Zones, as well as for Pier 1, as stated in Tables 1, 2, and 3. The spatial coverage and sample sizes for the RSC (n=31 for Zone 7, n=15 for Zone 4, and n=12 for Pier 1, plus previously collected data) should meet the DQO of characterizing the spatial extent of COPC. Similarly, the sample sizes for confirmatory samples (n=15 for Zone 7 and n=11 for Zone 4) should meet the DQO of supporting the SLERA for each of these Zones.**

   Response: This comment is acknowledged.

2. **The DQO related to the chemical analyses may not be met, however, as detailed in some of the following comments. Further, the spatial coverage of confirmatory samples cannot be reviewed at present, as locations will be determined later.**

   While the number of RSC samples may be sufficient to characterize the spatial extent of selected COPC, the RSC method presents a limitation. The rationale for the focus on copper, lead, and zinc as the selected trace metals is not clear. The 1998 ERA identified arsenic, chromium, mercury, and selenium as well as other inorganic COPC for Zone 4 and Zone 7. In Zone 4, chromium exceeded ER-Ms. At Pier 1, mercury, copper, lead, nickel, and zinc exceeded ER-Ms. In addition, for both Zone 4 and 7, the AVS/SEM evaluation in the ERA suggested that copper, lead, and zinc may be below levels of concern. Unless evidence can be provided showing that high concentrations of copper, lead, and zinc can be used to accurately track high concentrations of the other inorganic COPC, this screening method could miss areas with high concentrations of the other inorganic COPCs. This limitation should be addressed in the Work Plan.

   Response: The RSC method for inorganic COPCs (i.e., XRF) is most reliable for the measurement of selected metals including copper, lead and zinc. The Navy also has successfully utilized XRF at the Quantico Marine Corps Base to measure both chromium (>50 ppm) and arsenic (>10 ppm), when these constituents are present at levels of concern. An evaluation of historical data collected at NSB New London indicates that the assumption that high concentrations of copper, lead and zinc will co-occur with high concentrations of other inorganic COPCs (arsenic, chromium, nickel, and mercury) is reasonably well met.

Figure 1 shows the relationship between copper and arsenic concentrations in sediment at NSB New London. Using the combined Pier 1, Zone 4, and Zone 7 data, copper is a satisfactory predictor for arsenic (r = 0.52), with copper accounting for 52% of the observed concentration variability of arsenic. When considering the more recent Pier 1 data alone, the correlation between copper and arsenic improves (r = .78).

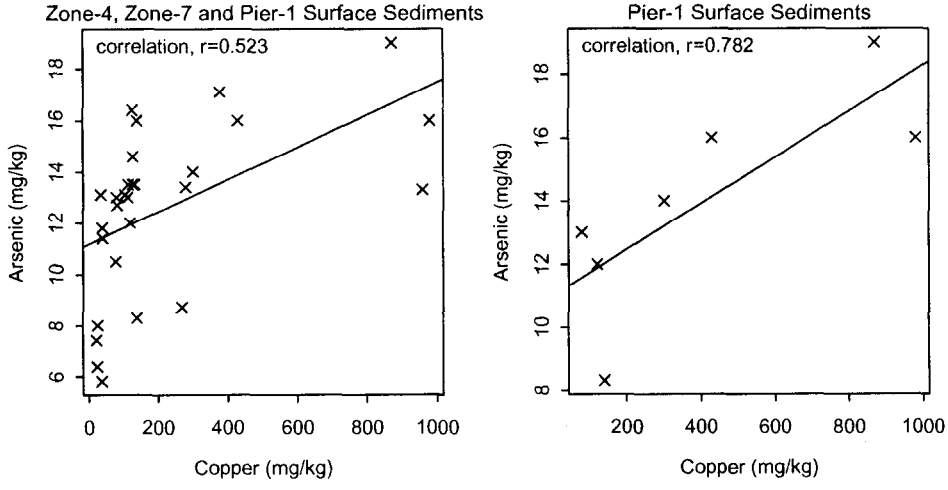

Figure 1. Arsenic vs Copper

Figure 2 presents historical data for chromium and lead in sediments. The combined data from Zones 4 and 7 and Pier 1 show that lead is positively correlated with chromium. In Zone 7 alone, where the higher concentrations of chromium were observed, the correlation with lead is strong (r = 0.96).

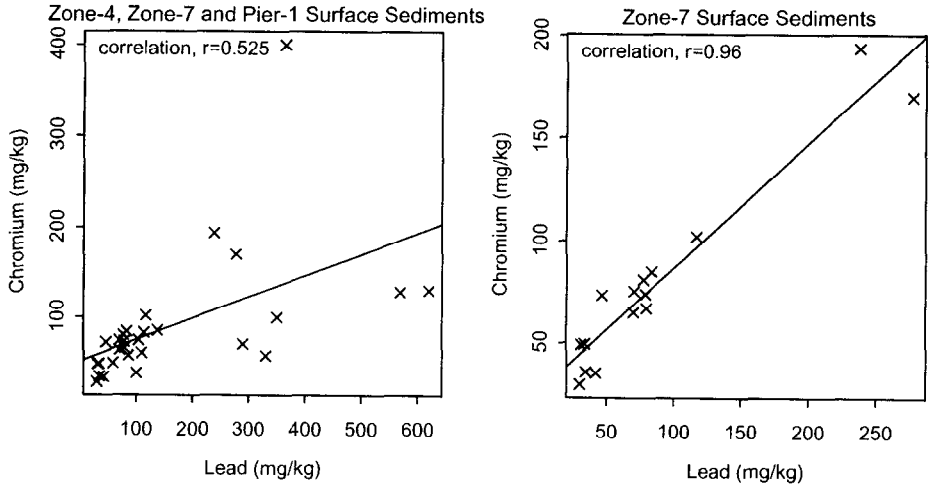

Figure 2. Chromium vs Lead

Figure 3 examines the relationship between nickel and lead. Nickel is strongly correlated with both the combined data set from all three areas, as well as the more recent data from Pier 1 alone (r > 0.9).

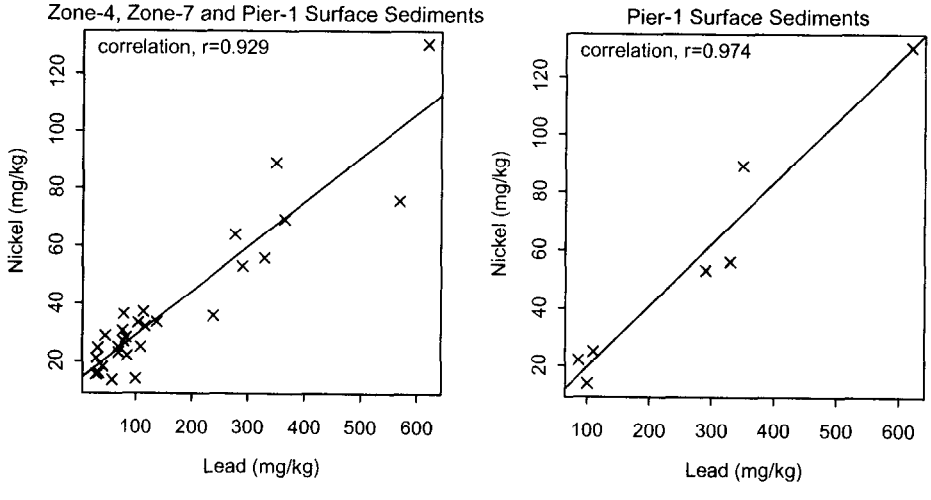

Figure 3. Nickel vs Lead

Figure 4 presents the relationship between copper and mercury. Copper is strongly correlated with mercury when considering both the combined data from all three areas (r = 0.87), as well as in individual areas, such as Zone 7 where some of the higher mercury values were observed (r = 0.97).

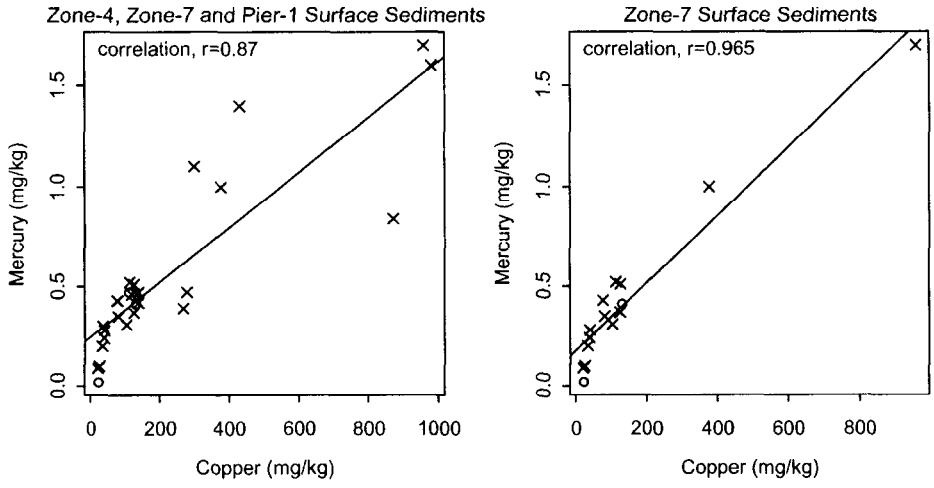

Figure 4. Mercury vs Copper

Copper, lead, and zinc do not appear to be good predictors of selenium concentrations. However, based on an evaluation of the historical data for these sites, it is not clear that selenium is present at concentrations of concern. Selenium was not detected in samples from Zone 7, and in Zone 4 the highest measured value was 0.9 ppm, which is slightly elevated above the ERL of 0.7 ppm. The lack of correlation between selenium and other metals may be a reflection of low selenium concentrations, and/or the relatively high analytical detection limits associated with the historical selenium data.

The assumption that high concentrations of copper, lead and zinc are expected to co-occur with high concentrations of arsenic, chromium, nickel, and mercury will be clearly stated in the Work Plan/Field Sampling Plan. Additionally, an attempt will be made to measure arsenic and chromium concentrations using XRF in the Pilot Study.

3. **Step 3 of the DQOs for all the sites indicates that confirmatory samples will be selected based on RSC results and will span the range of RSC concentrations. As these confirmatory sample results are to be used in a SLERA for Zone 7 and Zone 4, the bias should be toward the more highly contaminated RSC results. It is understood that a greater proportion of confirmatory samples will be analyzed from locations within the upper range of RSC results for Zones 7 and 4 (8 to 10 of 15 in Zone 7 and 7 of 11 in Zone 4), with fewer confirmatory samples analyzed at locations with lower RSC results. The most conservative approach for the SLERA would be to collect confirmatory**

samples only at locations with the highest RSC results. Because the sample sizes in these zones is sufficient, and because the need for developing a correlation between RSC results and fixed lab results at lower concentrations, as well, is recognized, the approach is acceptable for Zones 7 and 4.

Response: This comment is acknowledged.

At Pier 1, however, because the sample size is smaller (n=3), confirmatory sampling should focus on areas with the highest RSC results rather than over a range of results.

Response: At Pier 1, the Pilot Study is designed to support the development of a more comprehensive work plan for a baseline risk assessment by identifying the full suite of site related COPCs, and estimating the variability of these COPCs in sediment. Therefore, it is important that the confirmatory samples represent the full range of COPC variability. The Navy recognizes EPA's concern that by analyzing a small number of confirmatory samples, it is possible that locations with elevated chemical concentrations could be missed. However, given the recent study in Pier 1 indicating PCBs and numerous metals are present at levels above screening benchmarks, it is unlikely that the Pilot Study will conclude that Pier 1 does not require further study.

4. **The number of samples (n = 4 for each area) at the reference areas should provide an adequate comparison for the NSBNL sediment results. The spatial coverage of samples at the reference locations versus study site locations might lessen comparability, however. Paragraph 2 of Section 4.0 states that "Samples from the reference stations will be collected along two transects at similar distances from the shore as the sample stations in Zones 4 and 7 to maximize data comparability." Figure 4, that provides a rough picture of the reference sample locations, indicates that reference samples will be collected from approximately 100 to 700 feet from shore. Figures 2 and 3 show sample locations for Zones 7 and 4 to be much closer to shore, with several locations just off shore. If these figures are accurate, the spatial configuration of sample locations is not entirely comparable. Please address.**

   Response: The Navy will consider placing the reference area sample locations closer to shore, if the depth of the channel is comparable to the depth at NSB New London. The reference area samples were placed further out in the channel to ensure that some reference area sample locations were taken from the deeper water, recognizing that the river in this location may not be as deep as the maintained dredged areas adjacent to and between the piers at NSB New London .

5. **Please include a flowchart/diagram be added to the Work Plan/Field Sampling Plan that clearly presents the samples and locations to be collected. This flowchart should also clearly define the type of analysis planned for the specific samples. As written, it is difficult to understand which samples will be analyzed using RSC (and what analytes will be detected) and which samples will be submitted to the laboratory for analysis. To aid the clarity of the document, please include the intended use of the analytical results (*i.e.*, what results are planned for use in the ERA).**

Response: All sample locations will be analyzed for the suite of XRF metals (Pb, Cu, Zn, Cr, and As), and the two immunoassays (total PAHs and total PCBs). Following the completion of these analyses a subset will be selected for fixed laboratory analysis. The intended use of the RSC results is to maximize the probability that if contaminants are present at levels of potential concern, that the fixed laboratory analyses will provide quantitative measurements of these contaminants to support a screening level ecological risk evaluation, as well as to support the refinement of the screening process (Step 3a) through a comparison to Reference Area locations and conservative food chain exposure modeling. The RSC data will be further used to estimate spatial variability of COPCs, which in turn will be used to support further survey design sample size estimates, where addition data collection is necessary. The intended use of the fixed lab confirmation samples is to support a SLERA, to identify an initial list of COPCs, and then to support a quantitative estimate of site COPC concentration and comparison to Reference Area concentration to assist in narrowing the COPC list to those elevated above reference. In addition, confirmation samples alone, or in combination with RSC results will be used to calculate EPCs to support a conservative food chain model to look at potential adverse impacts to upper trophic levels.

A flow chart will be added to the Work Plan/Field Sampling Plan to clarify the overall Pilot Study approach. The flow chart will include the following elements:

- Identification of samples for RSC analysis and the associated analytes;
- Intended use of RSC data;
- Criteria for selecting samples for confirmatory laboratory analysis;
- Identification of samples for confirmatory laboratory analysis and associated analytes; and
- Intended use of confirmatory laboratory data, including the SLERA for Zones 4 and 7.

6. **The RSC methods taken from the Navy's Space and Naval Warfare Systems Center (SSC SD) are not referenced. It is understood that X-ray fluorescence (XRF) will be used to screen for copper, lead, and zinc, and immunoassay techniques will be used for total PAHs and PCBs. The detailed methods are neither discussed nor cited in this Work Plan. The methods should be presented: detection limits, sample volume, *etc.***

   The following comparison between RSC detection limits (taken from *The Use of Rapid Sediment Characterization Tools (RSC) in Sediment Assessment EPA Technical Support Project Meeting May 09, 2001Presented by: Victoria J Kirtay Environmental Sciences Division Space and Naval Warfare Systems Center San Diego*) with NOAA benchmarks shows that, for the selected chemicals, the detection limits are generally adequate to detect concentrations above ecological benchmarks. If the detection limits are at the high end of the ranges for copper, lead, and PCBs, however, elevated concentrations may go undetected. These limitations should be discussed in the Work Plan.

Chemical	ER-L	ER-M	XRF Detection Limit (mg/Kg)	Immunoassay Detection Limit Inorganics (ug/kg)
Copper	34	270	20-100	
Lead	47	218	10-50	
Zinc	150	410	20-100	
Organics				
Total PAH	4022	44972		50-500
Total PCB	23	180		50-500

Response: The methods to be employed for RSC include a modified EPA Method 4020 for PCBs, a modified EPA Method 4035 for PAHs, and a modified EPA SW846 Method 6200 for XRF metals. These methods are specified in the Pilot Study QAPP in Section A.3.4.3.1, and in Table A-6. The range of detection limits contained in Victoria Kirtay's table reflects the capabilities of bench-top instruments (at the low end of the detection limit range) and field deployed measurements (at the high end of the detection limit range). Bench-top instruments will be used for all Pilot Study analyses; therefore, the lower detection limits will apply. Section A.3.4.3.1 of the QAPP will be updated to include the RSC detection limits.

The Navy has extensive experience utilizing these RSC methods, and has succeeded in routinely measuring PCBs at 50-70 ppb, unless very high levels of PAHs are present. PAH interference is not anticipated at NSB New London based on the historical data. In addition, the total PAH immunoassay method has historically met the 4 ppm ecological screening benchmark for total PAHs. Other supporting information for the RSC methods is provided in the Pilot Study QAPP tables as follows: Table A-5 defines the sample size and handling requirements; Table A-6 defines the analytical methods, and Table A-8 defines the measurement quality criteria.

7. **For appropriate risk determinations to be made at these locations, some data may be needed that are not proposed at this time. These include AVS/SEM analyses as well as toxicity tests and benthic surveys to complement the chemical data in a triad approach. While the concentrations of COPC in sediments may not merit collection of these data, it should be recognized that they may need to be collected in a separate effort in the future.**

Response: This comment is acknowledged. If COPC concentrations in sediment are found to exceed conservative screening benchmarks, are above reference area concentrations, and/or are predicted to pose a potential risk to upper trophic levels, then a more detailed study will be designed to evaluate multiple lines of evidence (potentially including benthic surveys, toxicity tests, bioaccumulation tests, and/or AVS/SEM analyses). This study will be developed based on an ecological risk assessment scoping exercise and with input from regulatory agencies.

8. **One of the DQOs for Zone 7 is to determine if the previous dredging effectively removed all or some of the contaminated sediments near Pier 17 and Pier 15. According to Figure 2, two sediment samples are proposed within the dredged area north of Pier 17 with one on the edge of the area, and one sediment sample is proposed within the dredged area south of Pier 17 with one on the edge of the area. All three samples near the Pier 15 dredged area appear to be within the dredge outline. If the figures are accurate, moving the edge samples into the dredged areas would better ensure that this DQO is addressed. Please consider repositioning these samples.**

   Response: These sample locations will be repositioned to ensure that the areas within the dredged footprint will be adequately represented.

9. **The EPA Region I Immunoassay Guidelines for Planning Environmental Projects was not referenced. It is not clear if it was used in the development of this Work Plan/Field Sampling Plan. All intended uses of immunoassay test kits should be conducted in accordance with EPA protocols specified in the guidelines. Particular concern may be the use of immunoassay test kits for determination of PCBs in sediment that may contain greater than 30% moisture.**

   Response: The EPA Region I Immunoassay Guidelines will be referenced in the Pilot Study Work Plan/Field Sampling Plan. The Pilot Study will be implemented using the best available technology for RSC analyses. As such, the SSC SD analysts will follow the Region I guidance, EPA standard operating procedures, and the instructions of the test kit manufacturers. The recommended protocol for dewatering samples prior to analysis will be followed (*i.e.*, moisture content will be reduced to below 30% to ensure that a sufficient extract for PCB and PAH analysis is obtained).

10. **According to the EPA publication Guidance for the Data Quality Objectives Process, EPA QA/G-4, Step 6 of the Data Quality Objectives process should include acceptable limits for decision errors relative to consequences. Tables 1, 2 and 3 do not include acceptable limits for decision errors. Please specify acceptable limits on decision errors as described in the guidance.**

    Response: The intent of Step 6 is to evaluate the potential consequences for making an incorrect decision based on data, and to establish acceptable limits for these decision errors. Specific error tolerances are used as a design tool, in conjunction with estimates of the anticipated total study variability, to determine the number of samples required to achieve the desired limits on decision error rates.

    To support the SLERA, qualitative goals were used to design the Pilot Study, and conservative techniques will be employed to ensure that a problem is not overlooked, while also obtaining a representative sample of the area by utilizing a systematic grid sampling plan. The conservative approach includes biasing the selection of samples for confirmatory laboratory analysis towards those with elevated RSC results, and the use of a conservative decision rule that uses the maximum observed concentration compared to conservative screening benchmarks to identify COPCs. By employing these two techniques, the

probability of incorrectly determining no unacceptable potential ecological risk (false negative) will be limited.

To support the refinement (ERA Step 3a), a statistical comparison between the distribution of chemical concentrations in Zone 4/Zone 7 and the reference area will be performed. Two decision errors are possible. A false positive error would result in a determination that a significant difference between a Zone and the reference area exists, when in truth the concentration distributions are not different. Four tests will be employed to look at differences in distributions. A false positive limit of 0.10 (10%) will be used. A false negative error would result in failing to determine that a significant difference in concentration distributions exist, when in truth a meaningful difference exists. Specifications regarding both the acceptable probability of a false negative and the size of a distribution shift that is meaningful need to be developed. Based on the historically observed variability, it is expected that shifts of between 1.5 and 2 times the reference area values will be observable, with good power (between 75% and 95% of the time). Accordingly, the false negative error rate at a delta of 1.5 is set at 25%, and for a delta of 2 it is set at 10%.

For Pier 1, it is anticipated that a more detailed study will be required. Therefore, a qualitative approach to the Pilot Study sample design was adopted. The primary objective of the Pilot Study at Pier 1 is to generate data that will support the design of the detailed study. This includes identification of all site-related COPCs, better definition of the spatial extent of contamination, and the calculation of the spatial variability of these COPCs within the Pier 1 area. If sampling is concentrated on only the more highly contaminated portions of this zone, then the total variability might be underestimated, which may in turn result in calculating too few samples for the more detailed study. This potential error was addressed by proposing a systematic sampling grid that extends beyond the most highly contaminated area.

11. From the available literature on the RSC method, it appears that the XRF portion of the method has been formalized for only three metals (copper, lead, and zinc). Zone 4 of NSB-New London has historically exceeded chromium benchmarks in sediment, yet the screening approach will not identify areas of high or low chromium, possibly resulting in false negative results. I therefore recommend that more samples be included for fixed-laboratory analysis for metals to reduce uncertainty in this area.

Response: The Navy has successfully measured both chromium and arsenic using XRF at other sites, including the Quantico Marine Corps Base where these constituents are present at levels of concern. The Pilot Study Work Plan/Field Sampling Plan will be amended to include the measurement of chromium and arsenic by XRF. If chromium is present at levels of concern, then Zone 4 will be taken forward to a supplemental baseline ERA and additional data will be collected. Measuring for chromium and arsenic using XRF will be used to ensure contamination/hotspots of metals (arsenic and chromium) are not missed for quantitative analysis, provide better spatial coverage in regards to these metals, and provide a better estimate of variability for these metals.

12. It is not clear from the description of the RSC method exactly how the samples will be indexed from high to low contamination in order to determine which samples proceed with fixed-laboratory analysis. If a sample is high in metals but low in total PCBs, will it rank before or after a sample with high PCB and low metals when samples are selected for fixed-laboratory analysis? Please more clearly explain the methodology for ranking samples.

Response: Samples will be ranked based on Effects Range – Median (ER-M) Quotients (ERM-Qs). Given that one objective of confirmatory laboratory analysis is to represent the full range of chemical concentrations, samples will be selected that span the range of ERM-Qs. If either metals or organics are contributing to higher ERM-Qs, then both types of samples will be included for confirmatory analysis. Finally, spatial representation will be considered in the selection of samples for confirmatory laboratory analysis. A flow diagram presenting these criteria will be developed and included in the Pilot Study Work Plan/Field Sampling Plan (as noted in response to General Comment #5 above).

13. I assume that the screening-level assessment based on fixed-laboratory analysis will be consistent with EPA ecological risk assessment guidance, where the highest detected concentration within each area will be compared with the more conservative benchmark (*e.g.*, ER-L). Any chemical that exceeds the benchmark in any sample will be retained as a COPC. This approach is not explicitly described in the Work Plan. Please confirm that this approach will be used.

Response: The SLERA approach will be consistent with the process described above. The maximum observed COPC concentration in each Zone will be compared to the corresponding conservative screening benchmarks. In addition, all bioaccumulative chemicals that are detected will be carried forward for further evaluation (even if present below screening benchmarks) during the COPC refinement (ERA Step 3a), as well as all constituents for which there are no available benchmarks. Lastly, as discussed in the work plan, a project technical team meeting is proposed to scope the effort for the screening level risk assessment.

EPA REGION 1 SPECIFIC COMMENTS

ATTACHMENT A

Page	Comment
p. 2, §2.0	**The third paragraph states, "Confirmation samples will be selected based on two criteria: they will represent specific areas of interest, and will span the range of measured RSC concentrations." Please explain what is meant by, "specific areas of interest." While the selection of samples based on detected RSC concentrations is explained in more detail in the DQO tables, this first decision criterion is not explained. Please enhance the consistency between the DQO tables and Section 2.0 regarding the explanation of the selection of confirmatory samples.** Response: The statement on page 2 regarding "specific areas of interest" refers to the intent to obtain samples that represent the Zones 4, Zone 7, and Pier 1 study areas. In selecting confirmatory samples for chemical analysis spatial representation will be considered. The Pilot Study Work Plan/Field Sampling Plan will be revised to more clearly explain the selection criteria for laboratory confirmation samples. Additionally, please see the Navy's response provided to General Comment #5 and #12 above).
p. 4, §4.1	**The first sentence of the second paragraph states that 9 surface sediment samples are proposed for Pier 1. Figure 3, however, shows 12 sample locations at Pier 1, P1-47 through P1-58. Further, Step 7 of the DQO table for Pier 1 indicates a sample size of 12. Please confirm the number of samples proposed for Pier 1.** Response: Figure 3 is correct (12 samples) and the Work Plan text will be revised accordingly.
p. 6, §4.1.3	**Please indicate the field duplicate precision criteria within the text of the Work Plan or QAPP. According to the Region I guidance, duplicates must also be analyzed for the immunoassay test method.** Response: Ten percent of all samples will be collected as collocated field duplicates (Table A-7 in QAPP). However, precision criteria for field duplicates will not be defined. The purpose of field duplicates is to obtain estimates of the total measurement, sample collection, and small-scale (proximal) variability at Pier 1, Zone 4, Zone 7, and the reference area. EPA's recent *Peer Review Draft Guidance on Data Quality Indicators* (EPA QA/G-5i, 2002) discusses components of total study variability and the use of collocated samples and other replicates for assessment purposes. Collocated samples are useful for two general purposes: 1) to determine the relative importance of the combined components of (within sampling unit) variability measured by use of collocated samples versus

the larger scale variability (between sampling unit) captured by field samples; and 2) to support the design of future studies. This latter use is facilitated by simultaneous collection of analytical replicates. Analytical replicates and replicate measures for XRF will be conducted to isolate and assist in controlling variability with the measurement procedures, for which measurement quality criteria are specified in the QAPP (Table A-8 in QAPP). By utilizing both the analytical replicates and collocated samples, and assuming an additive linear model, it is possible to estimate how large a contribution the sample collection and small scale variability components are, and to determine if the design strategy needs to explicitly account for these components.

p. 13, Table 1 Please clarify the intent of the Subordinate Objective presented in Step 2: "Do the chemical constituents measured by RSC methods co-occur with other constituents measured in fixed laboratory confirmation samples?" The use of RSC should not include the objective of identification of "co-occurring" contaminants for use in an ecological risk assessment. Please explain the intent of this subordinate objective.

Response: Please refer to the response to General Comment #2 above. The sample design is based on the assumption of co-occurrence of COPCs with constituents measured by RSC methods. The intent of this subordinate objective is to confirm the validity of this assumption. Table 1 will be revised to clarify this point.

p. A-13, §A.2.4.1 A potential data quality concern is presented in the bulleted list on this page of the QAPP. This data quality concern involves maintaining an acceptable level of data quality to allow for statistically valid evaluation or pooling of the data. Please clearly explain the manner in which this data quality concern is being addressed. (Information related to pooling of data could be addressed in the flowchart/diagram suggested earlier.)

Response: Data collected during the Pilot Study will be used to conduct an initial SLERA, and to support the ERA refinement (Step 3a) through a statistical comparison to the reference area, and application of a conservative but realistic food chain model. To maintain acceptable data quality to support these evaluations, several steps were taken in the design of the study. To minimize potential impacts to data quality at the planning stage, the DQO planning process was employed, followed by the development of a Quality Assurance Project Plan. The potential concern defined in bullet three *maintaining an acceptable level of data quality to allow for statistically valid evaluation of pooling of the data* is addressed by establishing measurement quality objectives that define accuracy and precision goals (Table A-8), the means that will be used to assess achievement of those goals (Table A-7), and the corrective action that will be applied if the goals are not met (Table A-8). In addition, independent validation of the

confirmatory laboratory analytical data will ensure that only data that meet predefined quality criteria will be pooled for statistical evaluation.

First, the use of RSC methods to select a subset of stations for fixed laboratory analysis will reduce the probability of failing to obtain measurements from locations where the higher concentrations of COPCs are observed. Second, by employing a conservative screen that compares the maximum observed concentration in an area as measured by the fixed laboratory to conservative screening benchmarks, the probability of failing to identify a COPC is reduced. To control the probability that analytical methods under- or over-estimate the true concentrations, the QAPP describes a comprehensive QA/QC plan that will be employed to control measurement quality. The other key issue related to supporting statistically valid evaluations is addressed through the design of the study. By employing a systematic grid, samples should be representative of the study areas, and meet the assumption of randomness. The planned statistical comparisons to the reference area will be facilitated by collecting the number of samples specified for fixed laboratory analysis in Zones 4 and 7. Sample sizes were selected, in part, by a desire to ensure that these comparisons will be able to detect meaningful differences, if they exist, with adequate statistical power.

Data will be pooled in two ways. First, data across all three areas will be evaluated together when looking at the correlations between RSC and fixed laboratory values for the RSC constituents. Second, data will be pooled area by area to develop exposure point concentrations to support the food chain model and allow comparisons to pooled reference data.

p. A-16, §A.2.6.1 **This section states that version control is maintained and documented through the document header blocks, which identify the document version number and effective dates. This version control information is not found in the Draft version of the QAPP.**

Response: The text in this paragraph will be updated to state that version control is maintained and documented through the document header blocks, which identify the document, version (not version number), and effective date. Versions of the QAPP prepared for review, include preliminary draft and draft versions, are not assigned a version number. The final QAPP will be identified as "Version 1" with the effective date. If the final QAPP is subsequently revised the header block will identify the updated document as "Version 2."

p. A-48, Table A-8 **Please specify the acceptance criteria that will be applied to field duplicate samples. In addition, under the Corrective Action column, please specify the statements "Flag all data...." The added information should state the specific flag that should be applied to the data based on the specific QC criteria.**

Response: As discussed above, precision criteria will not be defined for field duplicates. Footnote 3 was inadvertently omitted from this draft version of the QAPP. The QAPP will be updated to include a footnote that states *data qualifiers for quality control sample exceedences are defined in Table A-8.*

Health & Safety Plan

Page	Comment
p. 8, Table 5.1	**Please correct the second sentence under the column header "Methods to Ensure Worker Safety."**
Response:	This sentence will be corrected.
p. 9, Table 5.2	**Please ensure that all metals are included in this table which lists the toxicological properties of chemical compounds potentially present in sediments. According to Table 1 of the Field Sampling Plan, arsenic and mercury were previously detected in surface sediment grab samples for Zone 7. Copper and zinc were also found to be present in sediments at Pier I and Zone 4. Information concerning exposure to these metals in sediment should also be included in Table 5.2.**
Response:	Table 5-2 will be revised as indicated:

Table 5-2: Toxicological Properties of Chemical Compounds Potentially Present in Naval Submarine Base New London Sediments

Class/Compounds (examples)	Principal Routes of Entry	Acute Exposure Effects/Symptoms	Chronic Exposure Effects/Symptoms
ORGANIC COMPOUNDS			
Aromatic Hydrocarbons			
Benzene	Inh, Ing, Skin	Central nervous system (CNS) depression; skin, eyes & upper respiratory tract irritation	Carcinogen, blood change leukemogenic
Ethylbenzene	Inh, Ing, Skin	Skin, eyes, nose & throat irritation	Skin rash
N-hexane	Inh, Ing, Skin	CNS depression; eyes & nose irritation	Skin irritation peripheral neuropathy
Toluene	Inh, Ing, Skin	CNS depression; skin, eyes, and respiratory tract irritation	Dermatitis
Xylene	Inh, Ing, Skin	Dizziness; nose, throat, skin, and eye irritation; olfactory changes; irritant; poison; distortion; hallucination; central nervous system (CNS) effects	Cardiac arrhythmia
Polychlorinated biphenyls	Inh, Ing, Skin	Chloracne, rashes	Liver disorders, dermatitis, carcinogen
Petroleum Distillates			
Gasoline, Diesel	Inh, Skin, Ing.	Anesthesia, dizziness, headache, nausea, vomiting, sleepiness, fatigue, disorientation, depression, unconsciousness, respiratory tract irritation, sore throat, cough	Dermatitis, headache, mood shifts, CNS effects, fatigue

Table 5-2 (continued)

Class/Compounds (examples)	Principal Routes of Entry	Acute Exposure Effects/Symptoms	Chronic Exposure Effects/Symptoms
Semivolatile Organic Compounds			
Polynuclear Aromatic Hydrocarbons (as PAHs)	Skin, Inh, Ing	Irritant to skin, vomiting, photosensitization, headache	As a class overall, can be considered mutagenic and tumorigenic with several compounds known carcinogens; also causes liver damage
INORGANIC COMPOUNDS			
Metals			
Arsenic	Skin, Inh, Ing	Irritates eyes, skin, and respiratory tract. May cause effects on gastrointestinal tract, cardiovascular system and central nervous system.	Skin dermatitis, sensitization, and pigmentation disorders, hyperkeratosis, perforation of nasal septum, neuropathy, liver impairment, and anemia. Cancer (skin, lung, lymphatic)
Chromium (VI)	Skin, Inh, Ing	Skin, respiratory tract irritation, dermatitis, skin ulceration	Carcinogen, lung and skin effects, nasal septum perforation
Chromium (III)	Skin, Inh, Ing	Skin, respiratory tract irritation	Lung disease
Copper	Inh, Inj	Metal fume fever, nausea vomiting, skin redness	Skin sensitization
Lead	Inh, Ing	GI distress, kidney failure	Neuropathy, CNS anemia

Table 5-2 (continued)

Class/Compounds (examples)	Principal Routes of Entry	Acute Exposure Effects/Symptoms	Chronic Exposure Effects/Symptoms
Mercury	Ihl, Skin, Ing	Skin irritant. Inhalation of vapors may cause pneumonitis. May affect CNS and kidneys. Effect may be delayed.	Affects central nervous system and kidneys, resulting in irritability, emotional instability, tremor, mental and memory disturbances, speech disorders. May cause inflammation and discoloration of the gums.
Nickel	Skin, Inh, Ing	Skin, nasal irritation, respiratory tract irritation	Carcinogen, lung, GI system disease
Zinc	Inh, Ing	Metal fume fever, skin irritation	GI System effects, dermatitis

Notes:

Inh = Inhalation
Ing = Ingestion
Skin = Skin absorption

p. 12, §5.2.5 **The second paragraph of this section should specify that the occupational exposure action level is for lead. Also, the correct units for this action level are micrograms per cubic meter, not micrograms per milligram.**

Response: The text will revised to include as follows: "The occupational airborne exposure action level to lead as established by OSHA is 30 $\mu g/m^3$ average over an 8 hour period."

DRAFT
National Oceanic and Atmospheric Administration
Hazardous Materials Response and Assessment Division
Response to April 2, 2003 Comments
Final Thames River Rapid Sediment Characterization Pilot Study

1. **NOAA is unclear what the screening level risk assessment will be based on. We assume only the sediment data collected from this effort. If there are other measures, please make note of them in the text.**

   Response: NOAA is correct in assuming that only sediment data collected as part of the Pilot Study will be used for the SLERA.

2. **Section 4.1, Page 4 makes note that the sediment collected at each station will be divided into three containers. One for Rapid Sediment Characterization (RSC) and the other two sent to Battelle. One set of the Battelle samples are for a full suite of chemical analysis. But NOAA is unclear what the other Battelle set is for. Because toxicity testing is a distinct future possibility, this set of sediment samples could be used for that purpose but it is likely unwise to hold sediment for such a lengthy time.**

   Response: The Navy is not planning on storing sediments for toxicity testing. Sediment in 4-oz containers will be analyzed at SPAWAR using RSC techniques. Sediment in one 16-oz container will be homogenized and aliquotted for analysis of organic compounds and trace metals. Sediment in a second 16-oz container will be analyzed for TOC and grain size. Sampling handling procedures are defined in the QAPP (Section A.3.3.1 and Table A-5).

3. **Table 2 (Page 15) discusses the past data sediment collected from Zone 4. The text states that the benzo(a)pyrene and chromium were measured at concentrations exceeding the ER-M. NOAA's review of this data from May of 1998 showed elevated sediment concentrations in several more chemicals. NOAA reports that "The highest concentrations of chromium, copper, lead, and zinc were detected in sediments of the Thames River adjacent to Zone 4 with the concentrations of all these trace elements exceeding their respective ER-M concentrations." In addition, the total PAH concentration was greater than the ER-M in Zone 4.**

   Response: NOAA's review is correct. The summary of the problem statement in Table 2 will be revised to more fully describe the constituents found at elevated levels in Zone 4.