



PORTLAND HARBOR RI/FS

**ROUND 2 QUALITY ASSURANCE PROJECT PLAN
ADDENDUM 11: SEDIMENT CHEMICAL MOBILITY
TESTING**

DRAFT

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August 4, 2008

Prepared for
The Lower Willamette Group

Prepared by
Integral Consulting Inc.

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RECOMMENDED FOR INCLUSION IN ADMINISTRATIVE RECORD

SECTION A: PROJECT MANAGEMENT

A1 Title and Approval Sheet

PORTLAND HARBOR RI/FS SEDIMENT CHEMICAL MOBILITY TESTING

Quality Assurance Plan Approvals

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A2.2 List of Acronyms

ACG	analytical concentration goal
ARI	Analytical Resources Inc.
ASTM	American Society for Testing and Materials
CAS	Columbia Analytical Services
CDF	confined disposal facility
CVAA	cold vapor atomic absorption spectrometry
DOC	dissolved organic carbon
ECD	electron capture detector
EET	effluent elutriate test
EPA	U.S. Environmental Protection Agency
FSP	field sampling plan
GPC	gel permeation chromatography
GC/ECD	gas chromatography/electron capture detector
GC/MS	gas chromatography/mass spectrometry
iAOPC	initial areas of potential concern
iCOC	initial contaminants of concern
HRGC/HRMS	high-resolution gas chromatography/high-resolution mass spectrometry
ICP/AES	inductively coupled plasma/atomic emission spectrometry
ICP/MS	inductively coupled plasma/mass spectrometry
MDL	method detection limit
MET	modified elutriate test
MRL	method reporting limit
PAH	polycyclic aromatic hydrocarbon
PARCC	precision, accuracy, representativeness, completeness, and comparability
PCB	polychlorinated biphenyl
PSEP	Puget Sound Estuary Program
QA	quality assurance
QAPP	quality assurance project plan
QC	quality control
RI/FS	remedial investigation and feasibility study
SBLT	sequential batch leachate test
SIM	selected ion monitoring
SM	standard method
SOP	standard operating procedure
SVOC	semivolatile organic compound

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TCLP	toxicity characteristic leaching procedure
TDS	total dissolved solids
TOC	total organic carbon
TSS	total suspended solids
USACE	U.S. Army Corps of Engineers
Vista	Vista Analytical Laboratory
VOC	volatile organic compound
ZHE	zero headspace extractor

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A3 Distribution List

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U.S. EPA QA Manager: Ginna Grepso-Grove
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NOAA: Helen Hillman
U.S. Fish & Wildlife Service: Ted Buerger
Columbia River Inter-Tribal Fish Commission: Patti Howard
Yakama Nation: Sheila Fleming
Confederated Tribes of the Warm Springs Reservation of Oregon: Brian Cunninghame
Confederated Tribes of the Umatilla Indian Reservation: Audie Huber
Confederated Tribes of the Siletz Indians: Tom Downey
Nez Perce Tribe: Erin Madden
Confederated Tribes of the Grand Ronde Community of Oregon: Jeff Baker
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A4 Introduction and Project Organization

A4.1 Introduction

This quality assurance project plan (QAPP) addendum describes procedures that will be used to conduct sediment chemical mobility tests for the remedial investigation and feasibility study (RI/FS) of the Portland Harbor Superfund Site (Site). Specifically, this information will be used in the FS portion of the project to evaluate chemical mobility under various removal and disposal scenarios.

This QAPP addendum supplements the Round 2 QAPP (Integral and Windward 2004). The Round 2 QAPP describes procedures and requirements for the generation of data of documented and acceptable quality that will be used for the RI/FS. This QAPP addendum addresses procedures that will be used for the sediment chemical mobility investigation that are not described in the Round 2 QAPP.

Study objectives, station locations, and sample collection and shipping procedures are described in the Sediment Chemical Mobility Testing Field Sampling Plan (Sediment and Chemical Mobility FSP; Anchor 2008). The Sediment Chemical Mobility FSP and this QAPP addendum provide the procedures to accomplish the following types of activities and data collection (as summarized in Table A4-1):

- Perform Modified Elutriate Test (MET¹), Sequential Batch Leachate Test (SBLT) (USACE 2003), and Toxicity Characteristic Leaching Procedure (TCLP) (40 CFR §261.24) chemical mobility tests on sediments from select initial areas of potential concern (iAOPCs) with elevated concentrations of initial chemicals of concern (iCOCs)
- Complete chemical analysis of MET elutriate, SBLT leachate, and TCLP leachate
- Complete chemical analysis of bulk sediment to evaluate the quality of the sediment collected to perform MET, SBLT, and TCLP
- Complete chemical analysis of surface water used in MET.

Supplemental information to Sections A and B of the Round 2 QAPP is provided in this QAPP addendum. Special training and certification requirements are described in Section A8 of the Round 2 QAPP, and specifications for documents and records are described in Section A9 of the Round 2 QAPP; these items are not addressed further in this QAPP addendum. Supplemental information provided in Section B and referred to in the Round 2 QAPP is summarized in the introduction to Section B.

¹ This test is now called the effluent elutriate test (EET) in the most recent U.S. Army Corps of Engineers (USACE) guidance on these tests (USACE 2003). However, the older term MET is used for convenience given that more people are familiar with that historical name.

Procedures for project assessment and oversight will be completed as described in Section C of the Round 2 QAPP, with the exception that laboratories will only be audited if serious problems are encountered. Analytical Resources, Inc. (ARI) was audited previously in connection with work completed for Round 1 of the Portland Harbor RI/FS. The role of Vista Analytical Laboratory (Vista; formerly known as Alta Analytical Laboratory) has been limited to analysis of polychlorinated biphenyl (PCB) congeners in sediment, and the quality of its data for Round 2 samples was very good. Anchor may audit ARI regarding their specific procedures for MET and SBLT extractions depending on lab performance.

Procedures for data validation will be completed as described in Section D of the Round 2 QAPP. No further supplemental information is required for QAPP Sections C and D.

A4.2 Project and Task Organization

The organizational structure for activities associated with the sediment chemical mobility investigation is provided in Section 3.1 of the Sediment Chemical Mobility FSP (Anchor 2008). Contact information for key project personnel for the sediment chemical mobility investigation is provided in Table A4-2 of this QAPP addendum.

Analyses for dioxins and PCB congeners for all sample types will be completed by Vista, located in El Dorado Hills, CA. MET, SBLT, and TCLP and the remaining chemical analyses, except for free cyanide, will be completed by ARI, located in Tukwila, WA. Analyses for free cyanide for the surface water, MET elutriate and SBLT leachate will be completed by Columbia Analytical Services, Inc. (CAS), located in Kelso, WA. The laboratory project and quality assurance (QA) managers are identified in Table A4-2. Their responsibilities are described in the Round 2 QAPP.

A5 Problem Definition and Background

The Portland Harbor RI/FS began in 2001 and has proceeded through three rounds of data collection, including physical conditions; fish, shellfish, and invertebrate tissue chemistry; surface and subsurface sediment chemistry; sediment toxicity testing; surface water chemistry; transition zone water chemistry and supporting measurements; and stormwater data among others.

The U.S. Environmental Protection Agency (EPA) has requested that sediment chemical mobility testing be conducted to support the FS by evaluating the mobility of chemicals in contaminated material that may potentially be removed from selected iAOPCs. At this stage in the RI/FS, exact locations of potential dredge areas, dredge volumes, and the range of conditions where dredged materials may be disposed of are unknown. The MET, SBLT, and TCLP sediment mobility tests provide the most value at the FS stage of the project given that identification of dredge material and disposal options is preliminary at this time. This information will be used directly in the feasibility evaluation of capping, dredging, containment, and disposal options.

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The objectives of the sediment chemical mobility sampling program are to assess the mobility of chemicals in sediments from iAOPCs where iCOC concentrations are relatively high for the Site. At the FS stage of the project, it is reasonable to assume that areas with higher chemical concentrations will likely have sediment removal as an evaluated option later in the FS. Sampling efforts will target these areas where known sediment chemical concentrations are elevated, and will focus on collecting sediments that would represent a range of chemical concentrations within each selected area. Although areas of higher concentrations are more likely to be subject to removal, disposal, and capping, these technologies will be applied to relatively wide areas and large volumes of sediments. Thus, the sampling should not focus exclusively on just the area represented by a single location of highest concentrations within each iAOPC. EPA has therefore selected additional sampling locations that are “representative” of the site without necessarily being areas of “high” concentrations.

The sampling effort will include collection of sediments that will be subjected to three types of elutriate or leachate production protocols: MET, SBLT, and TCLP. These tests are commonly used to understand potential environmental impacts associated with various remediation and disposal technologies for contaminated sediments (USACE 2003). They are most commonly performed in design phases of work, but can also be conducted for sediment FS reports and other preliminary evaluations.

The MET, SBLT, and TCLP test protocols are intended to provide information about leachate or elutriate production and chemical concentrations during various stages of removal and disposal. The MET protocol is intended to simulate conditions in effluent from a confined disposal facility (CDF) as it is being filled using Site sediments and surface waters. The SBLT protocol is intended to provide information on the leaching characteristics of chemicals in sediments that can be applied to several types of disposal situations and is also useful in evaluating chemical migration in *in-situ* capping scenarios. The TCLP protocol is a standard regulatory procedure for simulating leachate production in an upland landfill. Federal regulations (40 CFR §261.24) use the results of this test to determine whether a material should be classified as a hazardous waste. To support these tests, analysis of subsurface bulk sediment chemistry and surface water chemistry will also be conducted to understand the chemical levels already present in the materials used in the tests.

Elutriate and leachate data will be used in the FS to:

- Predict effluent chemical concentrations for dredged material from a confined disposal facility (MET elutriate)
- Estimate leaching of sediments in various confined disposal scenarios (SBLT leachate)
- Estimate the range of disposal facility sizes for dredged contaminated sediment such as CDFs (MET elutriate)
- Refine evaluations and costs for the use of disposal facilities (MET and SBLT)

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- Refine evaluations and the effectiveness of *in-situ* caps at the Site (SBLT)
- Evaluate sediments for hazardous waste criteria (TCLP).

Bulk sediment data will allow an understanding of the relationship between bulk sediment chemical levels and leachate/elutriate chemical levels. In the case of the SBLT, the paired sediment/leachate data allows the derivation of site-specific partitioning relationships. Surface water chemistry data will be used to understand whether chemicals present in MET elutriates originate from surface water rather than the sediments.

A6 Task Description

The following tasks will be completed for the sediment chemical mobility investigation:

- Subsurface sediment sample collection for chemical analysis and the MET, SBLT, and TCLP protocols
- Surface water collection for chemical analysis and the MET protocol
- Laboratory performance of MET, SBLT, and TCLP protocols
- Laboratory analysis of surface water; subsurface sediment bulk samples; and MET elutriate, SBLT leachate, and TCLP leachate samples
- Data validation and data quality evaluation
- Data management
- Report preparation.

Summaries of field and laboratory tasks and references to detailed descriptions are provided in this section. Procedures for data quality evaluation, data management, and report preparation are described in the Round 2 QAPP.

A6.1 Sample Collection and Processing

Subsurface sediment and surface water samples will be collected for the sediment chemical mobility investigation. For MET and SBLT samples, sediment from four locations will be collected and composited to create a single sample for each selected iAOPC. For TCLP samples, sediment from a single core will be homogenized for testing. Sediment core samples will be collected using a vibracorer equipped with 14-ft core tubes from the locations shown in Figures 2-1 and 2-2 of the Sediment Chemical Mobility FSP for MET/SBLT and TCLP protocols, respectively.

Sediment sampling procedures are described in detail in Section 7.5.1 of the FSP. Surface water samples will be collected using a peristaltic pump as described in Section 7.5.2 of the FSP. A total of 11 sediment samples and 11 surface water samples will be collected for MET and SBLT. Eleven sediment samples will also be collected for TCLP. Sampling procedures are summarized in Section B2 of this QAPP addendum.

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A6.2 Laboratory Mobility Tests, Chemical Analyses, and Deliverables

Sediment chemical mobility tests and analyses for chemical constituents other than free cyanide, PCB congeners, and dioxins will be completed by ARI. CAS will complete the free cyanide analyses for surface water, MET elutriate, and SBLT leachate samples. Vista will complete the PCB congener and dioxin analyses for all sample types. Laboratory procedures for the mobility tests are summarized in Sections 4.0 through 6.0 of the Sediment Chemical Mobility FSP. Standard operating procedures (SOPs) for the mobility tests are provided in Appendices A through C of the FSP.

Bulk sediment samples for the MET/SBLT protocols will be analyzed for inorganic and organic constituents and conventional parameters using methods summarized in Table A6-1. Sediment from selected iAOPCs will be analyzed for dioxins and total cyanide as shown in Table 4-2 of the Sediment Chemical Mobility FSP. Surface water, MET elutriate, and SBLT leachate samples will be analyzed for inorganic and organic constituents and conventional parameters using methods summarized in Table A6-2. Surface water, MET elutriate, and SBLT leachate samples from selected iAOPCs will be analyzed for dioxins and total, amenable, and free cyanide as shown in Table 4-4 of the Sediment Chemical Mobility FSP. All MET elutriate samples will be analyzed for both total and dissolved inorganic and organic constituents. SBLT leachate samples will be analyzed for dissolved inorganic and organic constituents. The MET elutriate and SBLT leachate samples will be filtered for dissolved metals using a 0.45- μm filter. For dissolved organic constituents, the MET elutriate samples will be centrifuged, and the SBLT leachate samples will be centrifuged and filtered using a 1- μm filter with a 4- μm pre-filter.

Bulk sediment samples used for TCLP and the TCLP leachates will be analyzed for inorganic and organic constituents as shown in Tables A6-5 and A6-6, respectively. Analytical methods for TCLP bulk sediments and leachate samples are shown in Tables A6-1 and A6-2, respectively. The sediment and leachate samples will be analyzed for metals (including mercury), semivolatile organic compounds (SVOCs), volatile organic compounds (VOCs), organochlorine pesticides, and chlorinated herbicides. The TCLP leachate samples will be filtered using a 0.6- to 0.8- μm glass fiber filter as specified in EPA method 1311, *Toxicity Characteristic Leaching Procedure* (EPA 2008).

Chemical mobility tests and laboratory analyses will be completed using EPA methods and other established methods as indicated in Tables A6-1 and A6-2. Laboratory data deliverables will be provided as described in Section A9.2 of the Round 2 QAPP. Complete analyte lists with method detection limits (MDLs), and method reporting limits (MRLs) are provided in Table A6-3 for MET and SBLT sediment samples; in Table A6-4 for the surface water, MET elutriate, and SBLT leachate samples; in Table A6-5 for TCLP sediment samples; and in Table A6-6 for TCLP leachate samples.

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A6.3 Project Schedule

Actual start dates for the sediment chemical mobility sampling will be determined following EPA approval of the Sediment Chemical Mobility FSP. Other conditions that may affect the sampling schedule are weather, river flows and stages, and equipment conditions and availability. Currently, it is anticipated that the sediment chemical mobility testing field investigations will begin in the summer of 2008.

Reporting of sediment chemical mobility sediment sampling results is discussed in Section 10.2 of the Sediment Chemical Mobility FSP.

A7 Quality Objectives and Criteria for Measurement Data

A7.1 Data Quality Objectives

Data quality objectives for the Portland Harbor RI/FS are described in Section 7 of the Work Plan (Integral et al. 2004). Analytical concentration goals (ACGs) were established during Round 1 for sediment and during Round 2 for surface water to identify analytical sensitivity levels that would be sufficient to determine ecological and human health risks for the Portland Harbor RI. Method modifications were implemented for water and sediment analyses to increase the sensitivity of various methods to meet ACGs to the extent that this was possible and practical. The project detection and reporting limits are the result of the specific laboratories and methods that were used for the RI work. These project limits are included in Tables A6-3 through A6-6.

ARI will not be able to meet the project detection limits and/or reporting limits for some cases as shown in Tables A6-3 and A6-4, although the ARI limits are generally close to the project limits. In the case of elutriate water, Table A6-4 also shows that ARI's detection and/or reporting limits may be higher than potential water quality criteria that may be used as benchmarks in the FS evaluation of the dredge alternatives. However, in some cases, the project limits are also above these same potential benchmarks. Consequently, it does not appear that ARI's inability to meet these limits will demonstrably limit the use of MET results in the FS evaluations. ARI is the preferred laboratory because they have greater familiarity and experience with running the MET (and SBLT). (It should be noted that MET detection and reporting limits should be compared to freshwater continuous concentration criteria, and not the human-health-consumption-based criteria, because effluent discharges do not represent a long-term impact relevant to the bioaccumulation pathway.)

A7.2 Data Quality Indicators

The overall data quality objective for the sediment chemical mobility investigation is to develop and implement procedures that will ensure the collection of representative data of known and acceptable quality. The QA procedures and measurements that will be used for this project are based on EPA and Puget Sound Estuary Program (PSEP)

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guidance (EPA 1983, 1994, 1999, 2005, 2008; Plumb 1981; PSEP 1986, 1997a,b,c) and on established laboratory methods from other sources.

Quality control (QC) samples and procedures are specified in each method protocol that will be used for this project. Methods are summarized in Table A6-1 for bulk sediment and Table A6-2 for surface water, MET elutriate, and SBLT and TCLP leachate samples. All QC requirements will be completed by each laboratory as described in the protocols and in the Round 2 QAPP. Laboratory control limits for QC samples and procedures are provided in Tables A7-1 through A7-4. QC procedures are described in the laboratory QA manuals (provided previously to EPA) and cited methods (Tables A6-1 and A6-2).

Data validation criteria and procedures are described in Sections D1 and D2 of the Round 2 QAPP. During data validation, low matrix spike or surrogate recoveries will be reviewed for analytes with wide laboratory control limits to evaluate potential bias of the data. Data will be qualified on this basis if a bias is identified. PARCC parameters (i.e., precision, accuracy or bias, representativeness, completeness, comparability) are described in Section A7.2 of the Round 2 QAPP.

Target MRLs for this study are summarized in Tables A6-3 through A6-6. Laboratory methods are described below in Section B4. MDLs have been determined by each laboratory for each analyte, as described in Section A7-2 of the Round 2 QAPP. MDLs are provided in Tables A6-3 through A6-6.

Analyte concentrations for this investigation will be reported to the MDL, as described in Section A7-2 of the Round 2 QAPP. For high-resolution gas chromatography/high-resolution mass spectrometry (HRGC/HRMS) analyses (i.e., chlorinated dioxins/furans and PCB congeners), sample-specific detection limits will be reported as described in EPA methods 1613B and 1668A (EPA 1994, 1999).

SECTION B: DATA GENERATION AND ACQUISITION

Section B of this Round 2 QAPP addendum includes the following supplemental sections:

- B1 Sampling Process Design
- B2 Sampling Methods
- B3 Sample Handling and Custody
- B4 Field and Laboratory Methods
- B5 Quality Control.

The following information is provided in the Round 2 QAPP and is not addressed further in this QAPP addendum:

- B6 Instrument/Equipment Testing, Inspection, and Maintenance
- B7 Instrument/Equipment Calibration and Frequency
- B8 Inspection/Acceptance of Supplies and Consumables
- B9 Non-direct Measurements
- B10 Data Management.

Details regarding field documentation for sediment collection are provided in the Sediment Chemical Mobility FSP (Anchor 2008).

B1 Sampling Process Design

The sediment chemical mobility investigation sampling effort will include the following activities:

- Surface water collection using a peristaltic pump
- Subsurface sediment cores collected using a vibracore sampling device.

The complete sampling design (including station locations, types and numbers of samples that will be collected, the rationale for collection, and the analyses that will be performed), as well as detailed sample collection and handling methods, are described in the Sediment Chemical Mobility FSP.

B2 Sampling Methods

This section outlines sample collection methods, equipment, and sample requirements for surface water and sediment samples. Procedural details and SOPs for field methods are provided in the Sediment Chemical Mobility FSP. Sample containers, sample size requirements, preservation, and holding times are summarized in Table B2-1. Corrective actions are addressed in Sections B2.3 and C1 of the Round 2 QAPP.

B2.1 Sample Collection and Processing Procedures

Samples to be collected for the sediment chemical mobility investigation include 14-ft sediment cores and surface water. Sediment cores will be collected using a vibracorer. Surface water will be collected using a peristaltic pump. Field QA samples are presented in Table B2-2 and will consist of field splits, which will be submitted to the laboratories as blind samples. Detailed descriptions of the sediment collection, homogenization, and characterization procedures are provided in Section 7.0 of the Sediment Chemical Mobility FSP.

B3 Sample Handling and Custody

Sample custody procedures are described in Section B3 of the Round 2 QAPP. Sample preservation and storage requirements and holding times for the sediments are provided in Table B2-1 of this QAPP addendum.

All samples will be stored in a cooler with ice on board the sampling vessel, transported to the field laboratory at the end of the day, and placed in a refrigerator until shipment to the laboratories. All samples will then be transferred to the analytical laboratories in coolers with ice and stored refrigerated at the laboratories (Table B2-1). Sediment remaining after MET and SBLT analysis will be archived frozen (-20°C).

B4 Analytical Methods

Laboratory methods to be used for the surface water, bulk sediment, and elutriate/leachate analyses are consistent with requirements provided in EPA methods and other widely accepted protocols (DEQ 2008; EPA 1983, 1994, 1999, 2005, 2008; Plumb 1981; PSEP 1986, 1997a,b,c). Modifications will be made to these methods, as necessary and technically feasible, to improve MRLs. Analytes and MRLs for surface water, bulk sediment, and elutriate/leachate samples are provided in Tables A6-3 through A6-6. Method modifications will not be sufficient to reduce MRLs to the level of the project reporting limits for several analytes, and project reporting limits will not be attained in these cases when the analyte is not detected.

Sediment chemical mobility testing samples will be analyzed for the constituents as shown in Table A6-3 for bulk MET/SBLT sediments; Table A6-4 for surface water, MET

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elutriates, and SBLT leachates; and Tables A6-5 and A6-6 for TCLP sediments and leachates, respectively.

The laboratory methods for sample preparation and analysis are summarized in Table A6-1 for sediment samples and Table A6-2 for surface water, elutriate, and leachate samples.

Sediment samples will be analyzed as described in the Round 2 QAPP. Modifications or refinements to QAPP procedures are included below.

B4.1 Bulk Sediments – MET/SBLT

Conventional Analyses

Conventional analyses of MET/SBLT sediment samples will include total solids, grain-size distribution, total sulfides, ammonia, total organic carbon (TOC), and specific gravity. In addition, selected sediment samples will be analyzed for total cyanide. EPA and PSEP methods will be used as shown in Table A6-1 and described in the Round 2 QAPP.

Total solids will be determined according to PSEP (1986). These results will be used to calculate analyte concentrations on a dry-weight basis and will also be reported in the database.

For the chemical analysis of sediment samples, grain-size analysis will be completed using PSEP (1986) protocols, as described in the Round 2 QAPP. This procedure includes determination of the silt and clay fractions using the pipette method. The following grain-size intervals will be reported:

Medium gravel	Fine sand	Very fine silt
Fine Gravel	Very fine sand	Clay, phi size 8-9
Very coarse sand	Coarse silt	Clay, phi size >9
Coarse sand	Medium silt	
Medium sand	Fine silt	

Total sulfide analysis will include distillation of the sulfide into a sodium hydroxide trap (PSEP 1986) and analysis by colorimetry using Standard Method (SM) 4500-S2 D-00/F-00.

Ammonia will be analyzed by EPA method 350.1M. The method, originally developed for use in water samples, will be modified for sediment samples by adding an extraction with a potassium chloride solution. Colorimetry will be used to determine ammonia concentrations.

TOC will be analyzed by Plumb (1981). Samples will be pretreated with hydrochloric acid to remove inorganic carbon, dried at 70°C, and analyzed by combustion in an induction furnace.

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Specific gravity will be measured on selected sediment samples in accordance with ASTM D854. The specific gravity of samples is one of the engineering properties used in the evaluation of sediment consolidation.

For selected sediment samples, total cyanide will be analyzed by SM 4500-CN-E. This method, originally developed for use on water samples, will be modified for sediment samples by adding acid distillation by SM 4500-CN-B. Colorimetry will be used to determine the total cyanide concentration in the resulting distillate.

Metals

MET/SBLT sediment samples will be analyzed for metals (aluminum, antimony, arsenic, cadmium, chromium, copper, lead, mercury, nickel, silver and zinc) as described in the Round 2 QAPP, with the following modifications. Analyses for aluminum, copper, and zinc may be completed by EPA method 6010B (inductively-coupled plasma/atomic emission spectrometry [ICP/AES]) rather than EPA method 6020 (inductively-coupled plasma/mass spectrometry [ICP/MS]). Since the concentrations of these analytes are expected to be sufficiently high, they may need to be reported from the ICP/AES analysis for all samples. These metals required multiple dilutions for analysis by ICP/MS due to matrix interferences in previously analyzed Round 2 samples (Integral 2005). For ICP/MS analyses, ARI will use EPA method 200.8 rather than EPA method 6020. Method 200.8 is very similar to EPA method 6020 and will provide data that are comparable in quality to previous data.

Organic Compounds

SVOCs will initially be screened to determine the level of extraction required for each sample. Clean samples will be extracted at an equivalent dry-weight mass of 7.5 g to 0.5 mL final volume for target detection limits between 67 and 670 µg/kg. Samples will be extracted using sonication, with gel permeation chromatography (GPC) cleanup if necessary, and analyzed by gas chromatography/mass spectrometry (GC/MS).

Chlorinated phenols will be extracted using sonication, followed by a diazomethane derivitization of the phenols by EPA method 8041, and analyzed by GC electron-capture detector (ECD).

Phthalate and polycyclic aromatic hydrocarbon (PAH) analyses will be completed by ARI using a series of two analyses. These analyses include the following components:

1. Prescreening of the samples to determine the approximate levels of analytes and matrix interferences.
2. Analysis of phthalates by GC/MS with selected ion monitoring (SIM), if applicable, at an appropriate dilution as determined by the screening.
3. Analysis of PAHs by GC/MS–SIM after silica gel cleanup.

In the absence of significant matrix interference, it may be possible to complete a single analysis for PAHs and phthalates. Sonication extraction will be used for the phthalates

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and PAHs. GPC cleanup will be used for phthalates and PAHs as required, and silica gel cleanup will additionally be used for PAH analyses if necessary.

For pesticide analyses, an equivalent dry-weight mass of 25 g will be extracted and taken to a final volume of 5.0 mL after appropriate cleanup(s). Silica gel and sulfur cleanups will be performed. The extracts will be run against a low-level calibration curve to produce the MRLs listed in Table A6-3. The method will be modified by adding analysis of PCB interference check standards to the pesticide analysis to allow evaluation of the pesticide chromatograms for PCB interference, as requested in recent EPA Region 10 guidance for organochlorine pesticide analysis (EPA 2005). PCB interference check standards will be analyzed within 72 hours of the sample if interference is noted during the laboratory's review of the pesticide chromatograms. The PCB interference check standard will be used to determine which column to use to quantify the pesticides, in the case when PCBs only interfere on one column. The PCB interference check standards will be reviewed during validation to evaluate the laboratory's quantification of pesticide results and to qualify data when interference is present.

For PCB Aroclor analyses, an equivalent dry-weight mass of 25 g will be extracted and taken to a final volume of 1.0 mL after appropriate acid, sulfur, and silica gel cleanups. Multiple acid cleanups are not necessarily expected, but will be employed if necessary. The extracts will be run against a low-level calibration curve to produce the MRLs provided in Table A6-3.

PCB Congeners and Chlorinated Dioxins and Furans

PCB congeners and dioxins/furans in sediment samples will be analyzed using HRGC/HRMS. Dioxins/furans will be analyzed as described in the Round 2 QAPP. PCB congeners will be analyzed by Vista using the methods employed for Rounds 2 and 3 of the RI, as described below.

For PCB congener analysis, the sediment samples will be extracted with toluene by Soxhlet extraction. The cleanup procedures that will be used by the laboratory include back-extraction with sulfuric acid, acidic and basic silica gel column chromatography, and acidic alumina column chromatography. These procedures are expected to provide sufficient cleanup even for samples that contain high levels of interferents such as petroleum hydrocarbons. Vista will use a DB-1 (or equivalent) column rather than the more commonly used SPB-octyl column. This will allow the resolution of PCB 156 and PCB 157, which coelute on the SPB-octyl column. Although PCB 118, a dioxin-like congener, coelutes with PCB 106 on the DB-1 column, PCB 106 is not a significant constituent of any of the Aroclors. Therefore, the concentration of this coelution can be attributed wholly to PCB 118.

Vista will analyze 10 g of sample initially. If PCB 126 is not detected in a sample and if the sample matrix allows, the sample will be re-extracted and reanalyzed using a sample mass of up to 50 g. However, if other coplanar PCB congeners are detected and PCB 126 will not contribute significantly to the sediment's toxicity even if it is present at a level

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below the MDL, the sample will not be reanalyzed using a larger sample mass. A sample size of 10 g is expected to be sufficient to meet project detection limit goals for all of the coplanar PCB congeners except PCB 126. The collocated sediment samples will be analyzed for all 209 PCB congeners.

Detection limits for PCB congeners and chlorinated dioxins and furans are calculated on an individual compound and sample basis and depend on the signal-to-background ratio for the specific labeled isomer. The detection limits listed in Table A6-3 are based on MDL studies completed by Vista for each method and are expected to approximate the sample-specific detection limits for typical samples. Sample-specific detection limits will be reported in the database for coplanar PCB congeners and dioxins/furans when these analytes are not detected.

B4.2 Bulk Sediments - TCLP

Metals

Metals samples will be prepared using digestion with nitric and hydrochloric acids for analysis of metals other than mercury. Analysis for these metals will be completed by EPA method 6010B (ICP/AES) or EPA method 200.8 (ICP/MS). Mercury samples will be digested using sulfuric and nitric acid. Analysis will be performed using cold vapor atomic absorption (CVAA).

Organic Compounds

Volatiles will be analyzed following SW8260C as described in the Round 2 QAPP. Highly contaminated samples may be analyzed using medium-level methods (methanol extraction and analysis).

Semivolatiles will initially be screened to determine the level of extraction required for each sample. Clean samples will be extracted at an equivalent dry-weight mass of 7.5 g to 0.5 mL final volume for target detection limits between 67 and 670 µg/kg. Samples will be extracted using sonication, with GPC cleanup if necessary, and analyzed by GC/MS.

For pesticide analyses, an equivalent dry-weight mass of 12 g will be extracted and taken to a final volume of 4.0 mL after appropriate cleanup(s). Silica gel and sulfur cleanups will be performed to meet target detection limits of 1.7 to 3.3 µg/kg. Extracts will be analyzed by dual column gas chromatography electron capture detector (GC/ECD). The method will be modified by adding analysis of PCB interference check standards to the pesticide analysis to allow evaluation of the pesticide chromatograms for PCB interference, as requested in recent EPA Region 10 guidance for organochlorine pesticide analysis (EPA 2005).

For PCB Aroclor analyses, an equivalent dry-weight mass of 12 g will be extracted and taken to a final volume of 4.0 mL after appropriate acid, sulfur, and silica gel cleanup to meet the target reporting limit of 33 µg/kg. Extracts will be analyzed by dual column GC/ECD.

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Herbicide samples will be extracted using methylene chloride/acetone and methylene chloride/hexane mixtures and an equivalent dry-weight mass of 15 g of sample. Herbicide esters are hydrolyzed to the acid form using a potassium hydroxide solution and back extracted with methylene chloride/acetone and methylene chloride/hexane mixtures, with a final extract volume of 50 mL. Extracts will be analyzed by dual column GC/ECD.

B4.3 Surface Water, MET Elutriates, SBLT Leachates

MET Elutriates

Elutriate samples extracted from the MET will be divided, and both total and dissolved fractions of all metals and organic compounds will be analyzed. Dissolved metals will be analyzed in samples that have been passed through a 0.45- μm filter. To avoid the effects of adsorption onto the filter surface, dissolved organics will be analyzed in unfiltered samples that are instead prepared by centrifugation, consistent with recommended MET procedures.

SBLT Leachates

SBLT procedures call for analysis of dissolved metals and dissolved and colloidal organic constituents. Leachates will be separated from the sediments by centrifugation. In accordance with the USACE (2003) protocol, metals are filtered through a 0.45- μm filter. To avoid adsorption onto the filter surface, organics are pre-filtered at 4 μm and then filtered through a 1- μm glass fiber filter.

Conventional Analyses

Conventional analyses of surface water, MET elutriate, and SBLT leachate samples will include total suspended solids (TSS), total dissolved solids (TDS), TOC, dissolved organic carbon (DOC), total sulfides, and ammonia. In addition, select surface water, MET elutriate, and SBLT leachate samples will be analyzed for total, amenable, and free cyanide. EPA methods will be used as shown in Table A6-2. Conventional analyses will be conducted on the total fraction of MET elutriates and SBLT leachates.

TSS and TDS will be determined gravimetrically according to EPA methods 160.2 and 160.1, respectively. The laboratory will use a 0.45- μm filter for these analyses.

TOC and DOC will be analyzed by EPA method 415.1. Organic carbon in the samples will be oxidized, and the evolved CO_2 will be analyzed using an infrared detector. Samples for DOC will be filtered in the field. Samples will be pretreated with sulfuric acid to remove inorganic carbon.

Total sulfides will be analyzed according to EPA method 376.2. The method is based upon the reaction of sulfide present in a sample with N,N-dimethyl-p-phenylenediamine and ferric chloride to form methylene blue. The color associated with ferric chloride can be removed by addition of di-ammonium hydrogen phosphate. Methylene blue color formation is directly proportional to the concentration of sulfide present in the sample

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Ammonia will be analyzed according to EPA method 350.1M, using a continuous-flow analytical instrument. Within the instrument, alkaline phenol and hypochlorite react with ammonia in the sample to form indenophenol blue, which is proportional to the ammonia concentration. The blue color formed is intensified with sodium nitroprusside.

Total cyanide will be determined by acid distillation using SM 4500-CN-C followed by colorimetric analysis in accordance with SM 4500-CN-E. For the determination of amenable cyanide, the sample will undergo a pre-chlorination step in accordance with SM 4500-CN-G, followed by acid distillation (SM 4500-CN-C) and colorimetric analysis (SM 4500-CN-E). Free cyanide will be determined by microdiffusion followed by colorimetric analysis following ASTM D4282 procedures.

Metals

Three methods may be used to analyze surface water, elutriate, and leachate samples for total and dissolved metals (Table A6-4). Digestion with nitric and hydrochloric acids will be used to prepare samples for analysis of metals other than mercury. Analysis for these metals will be completed by EPA method 6010B (ICP/AES) or EPA method 200.8 (ICP/MS). Samples for analysis of arsenic will be concentrated by a factor of 10 prior to analysis as described above, if necessary.

Mercury samples will be digested using sulfuric acid and nitric acid. Analysis will be completed by CVAA.

Organic Compounds

Organochlorine pesticides will be extracted from samples using separatory funnel extraction procedures. Samples will be analyzed by dual column GC/ECD. Silica gel cleanup may be performed on the sample extracts. Pesticide detections will be confirmed at ARI by reverse search of the GC/MS data when concentrations are sufficiently high.

Separatory funnel extraction using ultra-clean hexane will be used for PCB Aroclor extraction. One liter of sample will be extracted. Acid cleanup will be performed on the sample extract followed by silica gel and sulfur cleanup. Samples will be analyzed for PCB Aroclors by dual column GC/ECD. The surrogate compounds, tetrachloro-*m*-xylene and decachlorobiphenyl, will be added to every sample and QC sample and to continuing calibration standards.

Sample extractions for phthalates and PAHs will be completed using continuous liquid-liquid or separatory funnel extraction. Analyses for phthalates and PAHs will be performed separately using SIM. Analyses for PAHs will be completed after silica gel cleanup if necessary.

PCB Congeners and Chlorinated Dioxins and Furans

Analyses for PCB congeners and chlorinated dioxins and furans will be completed by Vista. These analyses will be completed by HRGC/HRMS using isotope dilution methodology with multiple ¹³C labeled surrogate standards.

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Cleanup procedures for chlorinated dioxins and furans will include sulfuric acid cleanup and silica/acid alumina and florisil column cleanup. Additional cleanup procedures will be used, as necessary, to remove analytical interferences. Detection limits for PCB congeners and chlorinated dioxins and furans are calculated on an individual compound and sample basis and depend on the signal-to-background ratio for the specific labeled isomer. The detection limits listed in Table A6-4 are based on MDL studies completed by Vista for each method and are expected to approximate the sample-specific detection limits for typical samples. Sample-specific detection limits will be reported in the database for PCB congeners and dioxins/furans when these analytes are not detected.

B4.4 TCLP Leachates

The TCLP leachate for metals and extractable organics will be created following EPA method 1311. Samples will be evaluated for choice of the proper buffered solution, and enough volume will be created to complete all the analyses listed.

The TCLP leachate for volatile analysis will be created by zero headspace extraction (ZHE).

Metals

Three methods may be used to analyze TCLP leachate samples for total metals (Table A6-2). Digestion with nitric and hydrochloric acids will be used to prepare samples for analysis of metals other than mercury. Analysis for these metals will be completed by EPA method 6010B (ICP/AES) or EPA method 200.8 (ICP/MS), or both.

Mercury samples will be digested using sulfuric acid and nitric acid. Analysis will be completed by CVAA.

Organic Compounds

Sample volumes will be reduced relative to standard volumes for all organic analyses except VOCs to minimize the volume of TCLP leachate needed for analysis. However, the leachate volume prepared and extracted for each analysis will be sufficient to meet the TCLP limits.

Herbicides will be extracted from samples using separatory funnel extraction procedures. Esters of the phenoxy acid herbicides in the sample will be hydrolyzed to the acid form by the addition of sodium hydroxide. The sample will then be acidified and the acid herbicides extracted using methylene chloride/acetone and methylene chloride/hexane mixtures. The acids will then be converted to their methyl esters using diazomethane. Extracts will be analyzed by dual column GC/ECD.

Organochlorine pesticides will be extracted from samples using separatory funnel extraction procedures. Samples will be analyzed by GC/ECD. Silica gel cleanup may be performed on the sample extracts. Pesticide detections will be confirmed at ARI by reverse search of the GC/MS data when concentrations are sufficiently high.

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Semivolatile sample preparation will be completed using continuous liquid-liquid extraction or separatory funnel extraction. Tentatively identified compounds will not be reported. Analyses will be completed by GC/MS for target TCLP compounds only.

Volatiles leachates created using ZHE will be analyzed using purge-and-trap GC/MS for target TCLP compounds only.

B5 Quality Control

QC samples will be prepared in the field and at the laboratories to monitor the bias and precision of the sample collection and analysis procedures.

Field QC samples for this study will include the collection and analysis of field splits (i.e., an additional subsample of a given homogenized sample). Field splits will be collected for sediment samples, as shown in Table B2-2. Field splits for surface water and sediment will be analyzed, but MET, SBLT, and TCLP will not be completed on the field splits.

Laboratory QC samples and control limits applicable for the analysis of sediment, surface water, MET elutriates, SBLT leachates, and TCLP leachates are presented in Tables A7-1 through and A7-4. Elutriate/leachate volumes may be restricted; therefore, at a minimum, ARI will prepare a preparation blank (per USACE methods), and ARI, CAS, and Vista will analyze method blanks and laboratory control sample/laboratory control sample duplicates at a frequency of one for every 20 samples analyzed or per analytical batch, whichever is more frequent. If sufficient elutriate/leachate volume is present, matrix spikes/matrix spike duplicates will be included for organic analyses; and matrix spikes and matrix duplicates will be included for the metals analyses and conventional parameters, as appropriate.

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