LWG
LOWER WILLAMETTE GROUP

Portland Harbor RI/FS

ROUND 2
QUALITY ASSURANCE PROJECT PLAN

June 24, 2004

Prepared for:
The Lower Willamette Group

Prepared by:
Integral Consulting, Inc.
Windward Environmental

SECTION A: PROJECT MANAGEMENT

A1 TITLE AND APPROVAL SHEET

PORTLAND HARBOR RI/FS
ROUND 2

Quality Assurance Plan Approvals

U.S. EPA Project Manager: Chip Humphrey __________________ Date: ________

U.S. EPA Project QA Manager Ginna Grepo-Grove __________________ Date: ________

CERCLA Project Coordinator: Keith Pine __________________ Date: ________

Integral Chemistry QA Manager: Maja Tritt __________________ Date: ________

Windward Bioassay QA Manager: Helle Anderson __________________ Date: ________

CAS Project Manager: Abbie Spielman __________________ Date: ________

CAS Laboratory QA Manager: Lee Wolf __________________ Date: ________

NEA Project Manager: Robert Wagner __________________ Date: ________

NEA Laboratory QA Manager: William Kotas __________________ Date: ________

STL Project Manager: Katie Downie __________________ Date: ________

STL Laboratory QA Manager: Terri Howard __________________ Date: ________

NAS Laboratory Project Manager: Gerald Irissarri __________________ Date: ________

NAS Laboratory QA Manager: Linda Nemeth __________________ Date: ________

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AAS	atomic absorption spectrometry
ACG	analytical concentration goals
AG	amber glass
AOC	Administrative Order on Consent
ASTM	American Society for Testing and Materials
CAS	Columbia Analytical Services
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
CLP	contract laboratory program
COC	chain-of-custody
CSM	conceptual site model
CVAA	cold vapor atomic absorption spectrometry
DQO	data quality objective
EDD	electronic data deliverable
EPA	U.S. Environmental Protection Agency
EQuIS	Environmental Quality Information System
ERA	ecological risk assessment
FSP	field sampling plan
GC/ECD	gas chromatography/electron capture detector
GC/FID	gas chromatography/flame ionization detection
GC/FPD	gas chromatography/flame photometric detection
GC/MS	gas chromatography/mass spectrometry
GFAA	graphite furnace atomic absorption
GPC	gel permeation chromatography
HAZWOPER	Hazardous Waste Operations and Emergency Response
HDPE	high density polyethylene
HHRA	human health risk assessment
HRGC/ HRMS	high resolution gas chromatography/high resolution mass spectrometry
HSP	health and safety plan
ICP	inductively coupled plasma
ICP/MS	inductively coupled plasma/mass spectrometry
ISA	initial study area
LIMS	laboratory information management system
LWG	Lower Willamette Group
µg/L	micrograms per liter
µg/kg	micrograms per kilogram
MDL	method detection limit
mg/kg	milligrams per kilogram
mg/L	milligrams per liter
MRL	method reporting limit
MS	matrix spike
MSD	matrix spike duplicate
NAS	Northwest Aquatic Sciences

NEA	Northeast Analytical
OSHA	U.S. Occupational Safety and Health Administration
PARCC	precision, accuracy, representativeness, completeness, and comparability
PCB	polychlorinated biphenyl
PCP	pentachlorophenol
PSEP	Puget Sound Estuary Program
PTFE	polytetrafluoroethylene
QA/QC	quality assurance/quality control
QAPP	quality assurance project plan
RI/FS	remedial investigation/feasibility study
RM	river mile
RPD	relative percent difference
ROD	record of decision
RPD	relative percent difference
RSD	relative standard deviation
SEA	Striplin Environmental Associates
SIM	selected ion monitoring
SOP	standard operating procedure
STL	Severn Trent Laboratories
SVOC	semivolatile organic compound
TBT	tributyltin
TDS	total dissolved solids
TIC	tentatively identified compound
TPH	total petroleum hydrocarbons
TOC	total organic carbon
TSA	technical systems audit
TSS	total suspended solids
VOC	volatile organic compound
WMG	wide mouth glass

A3 DISTRIBUTION LIST

U.S. EPA Project Officer:	Chip Humphrey
U.S. EPA Project Manager:	Tara Martich
U.S. EPA Oregon Operations Office:	Eric Blischke
U.S. EPA Project QA Manager:	Ginna Grepo-Grove
Oregon Department of Environmental Quality:	Jim Anderson
NOAA:	Helen Hillman
US Fish & Wildlife Service:	Ted Buerger
Oregon Department of Human Services	Dave Stone
Oregon Department of Fish & Wildlife:	Rick Kepler
Columbia River Inter-Tribal Fish Commission:	Patti Howard
Yakama Nation:	Paul Ward
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:	Rick Eichstaedt
Confederated Tribes of the Grand Ronde Community of Oregon:	Rod Thompson
Environment International:	Valerie Lee
Port of Portland:	Jim McKenna
Port of Portland:	David Ashton
Northwest Natural:	Bob Wyatt
Anchor Environmental LLC:	Libby Smith
Integral Project Manager:	Keith Pine
Integral Field Sampling and Analysis Coordinator:	Gene Revelas
Integral Chemistry QA Manager:	Maja Tritt
CAS Project Manager:	Abbie Spielman
CAS Laboratory QA Manager:	Lee Wolf
NEA Project Manager	Robert Wagner
NEA Laboratory QA Manager:	William Kotas
STL Project Manager:	Katie Downie
STL Laboratory QA Manager:	Terri Howard
NAS Project Manager:	Dick Caldwell
NAS Laboratory QA Manager:	Linda Nemeth
NAS Laboratory Project Manager	Gerald Irissarri

A4 INTRODUCTION AND PROJECT ORGANIZATION

A4.1 Introduction

This quality assurance project plan (QAPP) describes quality assurance/quality control (QA/QC) procedures that will be used to complete Round 2 of the remedial investigation and feasibility study (RI/FS) for the Portland Harbor Superfund Site (Site) in Portland, Oregon. The technical approach to the RI/FS is described in the Portland Harbor Programmatic Work Plan (Work Plan; Integral et al. 2004). Round 2 will focus on determining the distribution of chemicals in sediments in the initial study area (ISA), evaluating potential effects of sources on the river system, and supporting the human health and ecological risk assessments.

Field activities for Round 2 will include collection of surface sediment, sediment cores, shorebird area and beach sediment (beach sediment), surface water, and groundwater. The sampling design, rationale, and details regarding the various field activities are provided in the following field sampling plans (FSPs):

- *Portland Harbor RI/FS Round 2 Field Sampling Plan, Sediment Sampling and Benthic Toxicity Testing* (Integral and Windward 2004)
- *Portland Harbor RI/FS Round 2 Field Sampling Plan, Shorebird Area and Beach Sediment Sampling* (Integral et al. 2004)
- *Portland Harbor RI/FS Round 2 Field Sampling Plan, Surface Water Sampling* (Integral 2004).

The FSP for surface water sampling is under revision. An addendum to this QAPP will be prepared for surface water when the field sampling program requirements have been established.

The Round 2 sampling program for groundwater is under development. A field sampling plan for groundwater and an addendum to this QAPP will be prepared when the sampling program has been developed.

An additional component of the Round 2 investigations is a natural attenuation study that will involve collection of sediment cores and analysis of radionuclides to date the sediments. The natural attenuation study and related QA/QC procedures are described in Anchor and Texas A&M (2004).

A4.2 PROJECT AND TASK ORGANIZATION

This section presents the organizational structure for activities associated with the Round 2 investigation, including project management and oversight, field work, sample analysis, and data management. The organizational structure for Round 2 activities is illustrated in Figure A4-1. Contact information is provided in Table A4-1.

Project responsibilities are described below. Additional information is provided in Section 9.1 of the Work Plan (Integral et al. 2004).

A4.2.1 EPA Organization and Responsibilities

EPA is the lead agency for all activities related to site assessment for the Portland Harbor RI/FS. EPA will oversee Lower Willamette Group (LWG) activities associated with the Portland Harbor RI/FS and will coordinate all Trustee, Tribe, and State input regarding the development of technical and decision documents. The site and project managers for EPA are Chip Humphrey, Eric Blischke, and Tara Martich. EPA technical staff with significant involvement in this RI/FS include Dana Davoli (human health risk assessment), Joe Goulet (ecological risk assessment), Rene Fuentes (hydrogeologist), and Ginna Grepo-Grove (EPA QA manager). Contact information for these individuals is provided in Table A4-1.

A4.2.2 LWG Organization and Responsibilities

The LWG is comprised of ATOFINA Chemicals, Inc.; Chevron U.S.A. Inc; City of Portland; Gunderson, Inc.; Northwest Natural Gas; Oregon Steel Mills, Inc.; Port of Portland; Time Oil Co.; ConocoPhillips Corporation; and Union Pacific Railroad. The LWG is responsible for conducting the RI/FS according to the Work Plan, Administrative Order on Consent (AOC), and referenced EPA guidance. Jim McKenna of the Port of Portland and Bob Wyatt of Northwest Natural Gas co-chair the LWG. All official contact with the LWG should be through either Mr. McKenna or Mr. Wyatt (see Table A4-1).

A4.2.3 LWG Team Organization and Responsibilities

Contractors retained by the LWG will undertake Round 2 sampling, analysis, and reporting activities. The LWG consultant team is responsible for implementation of these tasks at the direction and oversight of the LWG.

CERCLA Project Coordinator—Keith Pine (Integral) is the CERCLA Project Coordinator, responsible for managing the Portland Harbor RI and coordinating the overall RI/FS efforts. In this role, he will oversee the RI technical work, participate in agency negotiations, and coordinate RI/FS activities with the LWG consultant team and other technical consultants. Mr. Pine will work closely with the Sampling and Analysis Coordinator to ensure that the objectives of the Round 2 field investigation are achieved. In the event that changes in the FSP or QAPP are needed, he will discuss proposed changes with LWG and EPA’s project managers or other designated EPA staff. Changes to the FSP and QAPP will not be made without prior approval from the EPA Project Manager unless conditions in the field or laboratory require immediate response.

Sampling and Analysis Coordinator—Gene Revelas (Integral) is the Sampling and Analysis Coordinator, responsible for all facets of the sampling and analysis programs. He will report directly to the CERCLA Project Coordinator. His specific responsibilities will include the following:

- Coordinate the field and laboratory analyses
- Communicate with Windward's QA/QC coordinator regarding field activities related to sediment sampling for toxicity testing
- Ensure adherence to the schedule by tracking sampling, laboratory analysis, validation, and data management tasks
- Provide solutions to problems if they occur
- Inform the CERCLA Project Coordinator of any decisions that involve changes to the FSP and QAPP.

Field Coordinator—Ian Stupakoff (Integral) is the Field Coordinator for the sediment, beach sediment, and surface water sampling efforts. He will be responsible for the following activities:

- Oversee planning and coordination for all sampling efforts
- Oversee all aspects of sampling activities to ensure that the appropriate sampling, quality assurance, and documentation procedures are used
- Oversee the establishment and operation of the field laboratory (i.e., for sample processing) and equipment facility near the study site.

Mr. Stupakoff will work closely with the Sampling and Analysis Coordinator and will be immediately notified if problems occur in the field. If changes to the FSP or QAPP are warranted, he will immediately notify the Sampling and Analysis Coordinator.

Field Task Leaders—Due to the magnitude and length of the Round 2 sampling program, the Field Coordinator will be assisted in his role by field task leaders. Joe Thompson (Integral) will serve as Field Task Leader for the surface sediment and beach sampling programs, and Susan FitzGerald (Integral) will serve as Field Task Leader for the subsurface sampling program. The field task leader will have the following responsibilities:

- Ensure that all activities adhere to the FSP and QAPP
- Inform the field coordinator of any decisions that involve changes to the FSP and QAPP
- Mobilize and prepare for field work
- Ensure sample custody, including chain-of-custody.

Field Quality Assurance Manager—Gene Revelas (Integral), the Sampling and Analysis Coordinator, will also serve as the QA manager for all Round 2 field sampling activities. He will oversee all aspects of the sampling events to ensure that the appropriate procedures and methods are used, that QA procedures are followed and requirements are met, and that documentation for all aspects of the field efforts is accurate and complete.

Chemistry Quality Assurance Manager—Maja Tritt (Integral) will be the QA manager for analytical chemistry. She will be responsible for the following activities:

- Coordinate the activities of the laboratories and track laboratory progress
- Ensure that method development is satisfactorily completed prior to analysis of project samples
- Verify that the laboratories implement the requirements of the FSP and QAPP and address quality assurance issues related to laboratory analyses
- Ensure that laboratory capacity is sufficient to undertake the required analyses in a timely manner
- Address scheduling issues related to laboratory analyses
- Direct the validation of the chemical data
- Communicate data quality issues to the data users
- Work with data users and EPA to address any data limitations.

Database Administrator—Tom Schulz (Integral) will have primary responsibility for data management and database maintenance and development. Mr. Schulz will be responsible for the following activities:

- Work with the field crew to ensure field data entries are correct and complete
- Work with the laboratories to ensure that data are delivered in the correct format for entry into the EQuIS database
- Work with data validators and the Chemistry QA Manager to ensure correct and efficient entry of data qualifiers
- Take all necessary measures to develop and maintain the integrity and completeness of the database
- Provide various data summaries to data users in the required formats for interpretation and for importing into EPA's database.

A4.2.4 Chemical Laboratories

Laboratory Project Manager—The Laboratory Project Manager carries overall responsibility for the successful and timely completion of sample analyses for this project. The Laboratory Project Manager will be responsible for the following tasks:

- Ensure that samples are received and logged in correctly, that the correct methods and modifications are used, and that data are reported within specified turnaround times
- Review analytical data to ensure that procedures were followed as required in this QAPP, the cited methods, and laboratory standard operating procedures (SOPs)
- Keep the Chemistry QA Manager apprised of the schedule and status of sample analyses and data package preparation
- Notify the Chemistry QA Manager if problems occur in sample receiving, analysis, or scheduling, or if control limits cannot be met
- Take appropriate corrective action as necessary
- Report data and supporting QA information as specified in this QAPP.

Laboratory Quality Assurance Manager—The Laboratory QA Manager is responsible for overseeing the QA activities in the laboratory and ensuring the quality of the data for this project. Specific responsibilities include the following:

- Oversee and implement the laboratory's QA program
- Maintain QA records for each laboratory production unit
- Ensure that QA/QC procedures are implemented as required for each method and provide oversight of QA/QC practices and procedures
- Review and address or approve non-conformity and corrective action reports
- Coordinate response to any QC issues that affect this project with the Laboratory Project Manager.

A4.2.5 Toxicity Testing

The project and quality assurance management structures for activities related to toxicity testing are provided in the Toxicity Testing QAPP (Appendix B).

A5 PROBLEM DEFINITION AND BACKGROUND

A5.1 Portland Harbor RI/FS

The overall purpose of the Portland Harbor RI/FS is "to investigate the nature and extent of contamination for the in-water portion of the Site, to assess the potential risk to human health and the environment, to develop and evaluate potential remedial alternatives, and to recommend a preferred alternative" (EPA 2001a). The critical objective of the RI/FS is to investigate the Site sufficiently to allow EPA to define site boundaries and select a remedy that is protective of the survival, growth and reproduction of ecological receptors and humans that may eat fish or shellfish or come in contact with sediments, surface water and seeps at the Site.

The current focus of the RI/FS is the initial study area (ISA), which is currently defined as the lower Willamette River from river mile (RM) 3.5 to 9.2, and adjacent areas logically associated with an evaluation of the in-water portion of this stretch of river (see Work Plan Map 1-1). Most industrial development along the banks of the Willamette River in Portland has occurred in this area, and the shoreline and channel have been significantly altered (e.g., bulkheads, piers) to accommodate industrial and urban growth. Information on the physical setting, geology, hydrology, chemical sources, water chemistry, sediment chemistry, and biological communities in Portland Harbor can be found in the Portland Harbor RI/FS Work Plan (Integral et al. 2004). The final boundaries of the site will be based on the findings of the RI/FS, and will be documented by EPA in one (or more) record of decision (ROD) when the final remedy is selected. Following finalization of the ROD, EPA will likely enter into a Consent Decree with one or more potentially responsible parties who will undertake remedial design, remedial action, and long-term monitoring of sediment management areas within the Site.

The data for the RI/FS will be gathered during four sampling programs:

- Pre- AOC tasks
- Round 1
- Round 2
- Round 3.

A summary of each of these sampling programs is provided in the Round 2 FSP for Sediment Sampling and Benthic Toxicity Testing (Integral and Windward 2004), and additional detail is found in the Work Plan (Integral et al. 2004).

A5.2 Round 2 Sampling

Building upon historical, pre-AOC, and Round 1 databases, the Round 2 sampling program is designed to gather the majority of the remaining data needed for the RI and risk assessments as well as initiate some FS data collection. The overall RI objectives that the Round 2 sampling efforts will support include:

- Identify and evaluate direct and indirect, known and unknown sources of significant contamination
- Identify and define local areas of in-water contamination (i.e., hot spots)
- Define the horizontal and vertical extent of contamination in all media
- Identify surface and buried sources of contamination that pose a potential risk to human health or the environment
- Evaluate impacts on sediments, surface water, and biota due to contaminated groundwater discharges
- Update and refine the conceptual site model with respect to temporal, physical, and chemical stability
- Develop a predictive model and/or support a direct toxicity approach to assess risks to the benthic community
- Evaluate whether the ISA should be expanded to define the Site.
- Assess what sources can be controlled by early actions
- Collect adequate data to fill data gaps identified as a result of the preliminary hydrodynamic model and to support the hydrodynamic model
- Collect data to understand contaminant fate and transport in the river system to adequately support remediation decisions.

Along with previous rounds of sampling data, the Round 2 information will be input directly into the baseline risk assessments.

The phasing and anticipated schedules of Round 2 field efforts, which will likely take place throughout 2004 and into 2005, are discussed in the Sediment Sampling and Benthic Toxicity Testing FSP (Integral and Windward 2004), Beach Sampling FSP (Integral et al. 2004), and Surface Water Sampling FSP (Integral 2004). The following types of data will be collected during Round 2 sampling:

- Surface sediment chemistry to support the ecological risk assessment (ERA) and human health risk assessment (HHRA) and to characterize the nature and extent of contamination, including contaminant distribution and potential source effects to the river
- Sediment bioassays to support the assessment of benthic risks for the ERA

- Subsurface sediment chemistry and physical data to characterize the nature and extent of contamination, including contaminant distribution and potential source effects to the river, and to support the FS
- Beach sediment chemistry to support the ERA and HHRA beach exposure scenarios
- Surface water chemistry to characterize water quality in the river, to evaluate potential effects of sources on the river system, and to support the HHRA and ERA
- Collection of groundwater, transition zone water, seep water, surface water, and/or sediment data to evaluate the impact to sediments and risk to environmental receptors from groundwater chemicals discharging to the river from upland areas
- Preliminary natural attenuation sampling (e.g., radioisotope cores) targeted for areas found to have potential processes that may support this alternative.

This QAPP addresses QA/QC activities, laboratory procedures, and data management procedures for surface sediment, Round 2A and 2B sediment cores, and beach sediment. The analytical requirements for natural attenuation sampling are presented under separate cover (Anchor and Texas A&M 2004). The analytical requirements and any additional QA/QC activities for surface water, groundwater, transition zone water, and seep water sampling will be addressed in future Round 2 QAPP addenda.

A6 TASK DESCRIPTION

The tasks to be completed for Round 2 include sample collection, laboratory analyses, data quality evaluation, data management, and report preparation. Summaries of these tasks and references to detailed descriptions are provided in this section.

A6.1 Field Tasks

Round 2 field tasks will include the collection of surface and subsurface sediment from nearshore and channel zones of the lower Willamette River and surface beach sediment from waterfowl and human access areas. The rationale for the field elements is discussed in the Portland Harbor RI/FS Work Plan (Integral et al. 2004). The contaminants of concern to be analyzed and the anticipated sampling schedule are discussed in the following sections. Sampling activities related to surface water and upland discharges into the ISA will be summarized in future addenda to this QAPP.

A6.1.1 Surface and Subsurface River Sediment

Based on the sampling program designed by EPA, approximately 500 stations have been selected for the collection of surface sediment samples in Round 2A (see

Appendix A, Integral and Windward 2004). Subsurface sediment vibracores will also be collected at approximately 200 of these stations. Collection of subsurface sediment will be completed in two sampling phases (i.e., Rounds 2A and 2B) to allow additional refinement of the sampling plan. The locations and number of surface and subsurface sediment samples to be collected are provided in the Sediment Sampling and Benthic Toxicity Testing FSP (Integral and Windward 2004). Subsurface samples from core intervals that are not designated for analysis will be placed in frozen archive.

All surface and subsurface sediment samples will be analyzed for the standard list of analytes: total organic carbon (TOC), grain-size distribution, total solids, metals, semivolatile organic compounds (SVOCs), organochlorine pesticides, and PCB Aroclors. At selected locations, additional analyses that include volatile organic compounds (VOCs), tributyltin (TBT), dioxins/furans, total petroleum hydrocarbons (TPH), chlorinated herbicides, and/or hexavalent chromium will be added to the analyte list based on proximity to potential sources of these compounds (e.g., TPH near fuel facilities, butyltins near shipyards). In addition, samples will be selected for PCB congener analysis in consultation with EPA following a review of data for PCB Aroclors in sediment and PCB congeners and Aroclors in fish tissue. Tables 2-2 and 2-3 of Integral and Windward (2004) provide a list of target analytes for each surface and subsurface sediment sampling station. Selected physical parameters that provide preliminary engineering information (i.e., specific gravity and Atterberg limits) will also be analyzed. Samples for specific gravity will be collected at all stations. Samples for Atterberg limits will also be collected at all stations, and then 10% of the samples will be selected for analysis. A registered geotechnical engineer or geologist will select appropriate samples for Atterberg analysis based on sample descriptions, as described in Section 4.6.2 of Integral and Windward (2004).

Samples from designated surface sediment stations will undergo two bioassay tests: a 28-day *Hyalella* test and a 10-day *Chironomus* test. Toxicity testing is addressed in the Toxicity Testing QAPP (Appendix B).

A6.1.2 Beach Sediment

Thirty composite surface sediment samples will be collected from riverbank beaches that provide waterfowl habitat or human access. The beach sediment samples will be analyzed for TOC, grain size, total solids, metals, SVOCs, organochlorine pesticides, and PCB Aroclors and congeners, as described in the Shorebird Area and Beach Sediment FSP (Integral et al. 2004).

A6.2 Laboratory Analyses and Deliverables

Chemical analysis of surface, subsurface, and beach sediment samples will be completed by three laboratories. Northeast Analytical (NEA; Schenectady, NY) will complete analyses for pesticides, PCB Aroclors, and SVOCs. Severn Trent Laboratories (STL; Tacoma, WA) will complete analyses for chlorinated herbicides. Columbia Analytical Services (CAS; Kelso, WA; Redding, CA; and Houston, TX) will complete all other analyses (i.e., conventional analyses, geotechnical characteristics, metals, butyltin compounds, petroleum hydrocarbons, VOCs, PCB congeners, and chlorinated dioxins and furans). Laboratories and analyses for sediment samples are provided in Table A6-1. A complete analyte list with analytical concentration goals (ACGs) and method reporting limits (MRLs) is provided in Table A6-2.

Analyses will be completed using EPA and Puget Sound Estuary Program (PSEP) methods (EPA 2004; PSEP 1986, 1997a,b) and other established methods as indicated in Table A6-1. Full laboratory data reports will be provided in hard copy, and electronic data deliverables (EDDs) will be provided in spreadsheet format as required for importing into the database. A list of hard copy data deliverables from the laboratory is provided in Section A9.2.

Toxicity testing procedures and deliverables are described in the Toxicity Testing QAPP (Appendix B). Details regarding laboratory methods for sediment samples are provided in Section B4.

A6.3 Data Quality Evaluation

Data quality evaluation includes the components, data verification and data validation (EPA 2002c). Data verification involves verifying that correct procedures were followed and that control limits were met. Data validation involves evaluating the quality and usability of the data in the context of project objectives. Data verification will initially be completed by the entity that generates the data. Integral's field staff will verify data generated in the field, and each laboratory will verify data generated at the laboratory in accordance with its SOPs and QA manual. The accuracy and completeness of the final database will be verified by Integral under the direction of the database administrator.

Third-party data validation and data verification will be completed by an independent validation firm, which will be selected at a later date. Verification and validation procedures are described in Section D2 of this QAPP.

A6.4 Data Management

Integral Consulting is responsible for data management for the Portland Harbor RI/FS. The Environmental Quality Information System (EQuIS™) database application is used as the final repository for all data related to the Portland Harbor RI/FS and will be used to manage the field and laboratory data for Round 2. Field data will by necessity be entered manually. Data will be provided electronically by the laboratories and imported directly into the database. Electronic data for Round 2 will be submitted to EPA in a format compatible with NOAA's Query Manager database system. Data management procedures are discussed in Section B10.

A6.5 Reports

A field sampling report will be prepared by Integral after each type of sampling event (e.g., Round 2 surface sediment sampling, Round 2A sediment coring, beach sediment, and surface water) has been completed, and will be provided to EPA 60 days after the completion of each sampling type, as described in each FSP. A site characterization summary report will be provided to EPA after laboratory analyses and the data quality review have been completed and the Round 2 data are final. Reports are described in Section C2.

A6.6 Project Schedule

Round 2 field sampling will be initiated following EPA's approval of the project Work Plan, FSPs, and QAPP, and following the completion of a site reconnaissance to evaluate the accessibility of proposed sampling stations. It is anticipated that Round 2 sediment sampling will begin in July 2004 and continue through October 2004. Round 2B subsurface sampling is planned for spring 2005. Beach sediment samples and sediment cores for the natural attenuation study (Anchor and Texas A&M 2004) will be collected in conjunction with the surface and subsurface sediment sampling. Round 2 surface water samples are likely to be collected during summer 2004, fall 2004, and late winter 2005. Groundwater sampling is expected to take place in 2005.

Laboratory data will be due to LWG 30 days from receipt of the last sample in each sample batch. A sample batch is a group of no more than 20 samples that are processed together at the laboratory. As specified in the AOC, and upon request, analytical data will be made available to EPA within 60 days of each sampling activity. Validated analytical laboratory data will be provided to EPA in an electronic format within 90 days of completion of each sampling event (i.e., surface sediment sampling, Round 2A subsurface sediment sampling, Round 2B sediment coring, beach sediment sampling, surface water sampling, groundwater pathways sampling, and natural attenuation sampling). The Round 2 data submittal and reporting schedule will be modified if additional data validation is required beyond the specifications of this QAPP, as described in Section D2. Data will be provided in electronic format showing location, medium, and results. A sampling event is considered complete when the last sample for the sampling event has been collected and shipped to the laboratory.

Field sampling reports, which will summarize field sampling activities, including sampling locations (maps), requested sample analyses, sample collection methods, and any deviations from the FSPs, will be prepared and submitted to EPA within 60 days of completing each individual field sampling event.

Round 2 information and data evaluations will be included in the comprehensive site characterization summary and data gaps analysis report and in the draft RI report and draft baseline risk assessments. The draft RI report will be prepared after all sampling and analysis rounds for the project are completed. A complete project schedule is provided in the Work Plan (Integral et al. 2004).

A7 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

DQOs were developed for the Portland Harbor RI/FS using EPA's DQO process (EPA 2000) to describe data and data quality needs for the project. Data quality indicators such as the PARCC parameters (EPA 2002d) and analytical sensitivity will be used to assess conformance of data with quality control criteria. DQOs and quality control criteria are described in this section. Quality objectives for toxicity testing are described in Appendix B.

A7.1 The Data Quality Objective Process

As part of the development of the RI/FS Work Plan (Integral et al. 2004), data needs were evaluated for assessing chemical distributions in Portland Harbor sediment and river water, for determining human health and ecological risk, and for developing remedial alternatives for Portland Harbor sediments. The seven-step DQO process (EPA 2000) was used for each study element to identify the adequacy of existing data and the need for additional data, to develop the overall approach to each study element, and ultimately to develop the various field sampling plans for Rounds 1 and 2. The DQO processes for the various aspects of the site characterization are provided in Section 7 of the Work Plan. The DQO tables that were developed as part of the Work Plan are provided in Appendix A of this QAPP.

The need for low-level reporting limits was identified in several of the DQO tables. ACGs have been established to identify analytical sensitivity levels that will be sufficient to determine risks to ecological and human health. Although ACGs can be met for many analytes, modifications to optimize laboratory MRLs are not sufficient to meet the ACGs in all cases. MRLs and ACGs for Round 2 sediment are provided in Table A6-2. Analytical sensitivity is discussed further in the following section.

A7.2 Data Quality Indicators

The overall quality objective for Round 2 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 PSEP guidance (EPA 2004; Plumb 1981; PSEP 1986, 1997a,b) and on established laboratory methods from other sources (ASTM 2003; Ecology 1997; Krone et al. 1989).

PARCC parameters (i.e., precision, accuracy or bias, representativeness, completeness, comparability) are commonly used to assess the quality of environmental data.

Bias represents the degree to which a measured concentration conforms to the reference value. The results for matrix spikes, laboratory control samples, field blanks, and method blanks will be reviewed to evaluate bias of the data. The following calculation is used to determine percent recovery for a matrix spike sample:

$$\%R = \frac{M - U}{C} \times 100$$

%R = Percent recovery
M = Measured concentration in the spiked sample
U = Measured concentration in the unspiked sample
C = Concentration of the added spike

The following calculation is used to determine percent recovery for a laboratory control sample or reference material:

$$\%R = \frac{M}{C} \times 100$$

%R = Percent recovery
M = Measured concentration in the reference material
C = Established reference concentration

Results for field and method blanks can reflect systematic bias that results from contamination of samples during collection or analysis. Any analytes detected in field or method blanks will be evaluated as potential indicators of bias.

Precision reflects the reproducibility between individual measurements of the same property. Precision will be evaluated using the results of matrix spike duplicates, laboratory duplicates, field splits, and field replicates. Precision is expressed in terms of the relative standard deviation for three or more measurements and the relative percent difference (RPD) for two measurements. The following equation is used to calculate the RPD between measurements:

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

RPD = Relative percent difference
C_1 = First measurement
C_2 = Second measurement

The relative standard deviation is the ratio of the standard deviation of three or more measurements to the average of the measurements, expressed as a percentage.

Completeness will be calculated as the ratio of usable data (i.e., unqualified data and U-, J-, or N-qualified data) to generated data, expressed as a percentage. Completeness will be calculated for each suite of analytes for each sample type and sampling event.

Additional laboratory QC results will be evaluated to provide supplementary information regarding overall quality of the data, performance of instruments and measurement systems, and sample-specific matrix effects.

QC samples and procedures are specified in each method protocol that will be used for this project. Methods are summarized in Table A6-1. All QC requirements will be completed by each laboratory as described in the protocols, including the following (as applicable to each analysis):

- Instrument tuning
- Initial calibration
- Initial calibration verification
- Continuing calibration verification
- Calibration or instrument blanks
- Method blanks
- Laboratory control samples
- Internal standards
- Surrogate spikes
- Serial dilutions
- Matrix spikes
- Matrix spike duplicates or laboratory duplicates.

To alert the data user to possible bias or imprecision, data qualifiers will be applied to reported analyte concentrations when associated QC samples or procedures do not meet control limits. Laboratory control limits for the methods that will be used for this site investigation are provided in Tables A7.1 and A7.2 and in the laboratory QA manuals (Appendices C to F). Data validation criteria and procedures are described in Sections D1 and D2 of this QAPP.

MRLs reflect the sensitivity of the analysis. The methods and modifications selected for this study will incorporate modifications recommended by PSEP (1997a) to optimize MRLs. Target MRLs for this study are summarized in Table A6-2. Method modifications are described in Section B4.

Method detection limits (MDLs) will be determined by each laboratory for each analyte, as required by EPA (2004). MDL studies have been completed for many of the methods (Table A6-2). MDL studies will be completed for all remaining analyses and provided to EPA before project samples are analyzed. MDLs are statistically derived and reflect the concentration at which an analyte can be detected in a clean matrix (e.g., sand or distilled water) with 99% confidence that a false positive result has not been reported. MRLs are established by the laboratories at levels above the MDLs for the project analytes. The MRL values are based on the laboratories' experience analyzing environmental samples and reflect the typical sensitivity obtained by the analytical system in environmental samples. For Round 2 analyses, the concentration of the lowest standard in the initial calibration curve for each analysis is at the level of the MRL. This allows reliable quantification of concentrations to the MRL in the absence of matrix interferences.

Analyte concentrations for this investigation will be reported to the MDL. Analytes detected at concentrations between the MRL and the MDL will be reported with a J qualifier to indicate that the value is an estimate (i.e., the analyte concentration is below the calibration range). Non-detects will be reported at the MDL. The MDL will be adjusted by each laboratory, as necessary, to reflect sample dilution or matrix interference. The use of the MDL to report data for non-detects will be evaluated during data quality review.

Representativeness and comparability are qualitative QA/QC parameters. Representativeness is the degree to which data represent a characteristic of an environmental condition. In the field, representativeness will be addressed primarily in the sampling design by the selection of sampling sites and sample collection procedures. In the laboratories, representativeness will be ensured by the proper handling and storage of samples and initiation of analysis within holding times.

Comparability is the qualitative similarity of one data set to another (i.e., the extent to which different data sets can be combined for use). Comparability will be addressed through the use of field and laboratory methods that are consistent with methods and procedures recommended by EPA and PSEP and are commonly used for sediment studies.

A8 SPECIAL TRAINING/CERTIFICATION

The LWG has assembled a project team with the requisite experience and technical skills to successfully complete the Round 2 investigation. All consultant team personnel involved in sample collection have extensive environmental sampling experience. Minimum training and certification requirements for laboratory personnel are described in the laboratory QA manuals (Appendices C – F of this QAPP).

The Superfund Amendments and Reauthorization Act of 1986 required the Secretary of Labor to issue regulations providing health and safety standards and guidelines for workers engaged in hazardous waste operations. In response to this requirement, the U.S. Occupational Safety and Health Administration developed regulation 29

CFR§1910.120, the "Hazardous Waste Operations and Emergency Response" standard (HAZWOPER). This standard includes requirements for workers engaged in hazardous waste operations to complete a 40-hour training course and annual 8-hour refresher courses. The training provides employees with knowledge and skills that enable them to perform their jobs safely and with minimum risk to their personal health. All sampling personnel will have completed the 40-hour HAZWOPER training course and 8-hour refresher courses, as necessary. Documentation of course completion will be maintained in personnel files.

A9 DOCUMENTS AND RECORDS

Records will be maintained documenting all activities and data related to sample collection and to laboratory analyses and bioassays. Results of data verification and validation activities will also be documented. Procedures for documentation of these activities are described in this section. Documentation for bioassays is described in the Toxicity Testing QAPP (Appendix B).

Each field sampling plan for Round 2, this QAPP, and the health and safety plan (HSP) will be provided to every project participant listed in Section A3. Any revisions or amendments to any of these documents will also be provided to these individuals.

A9.1 Field Documentation

The following field records will be maintained throughout the duration of sampling activities:

- Field logbooks
- Field data forms
- Sample description forms
- Sediment core logs
- Sample labels
- Sample chain-of-custody (COC) forms
- Custody labels
- Photographic documentation.

The content and use of these documents are described in Sections 4.3, 4.6., and 4.9 of the Round 2 Sediment Sampling and Benthic Toxicity Testing FSP (Integral and Windward 2004). Field documentation related to sample collection will be maintained in the project file in Integral's Olympia office. The laboratories will return original completed COC forms to Integral. These will also be maintained in the project file.

The following reports will be completed, as necessary, to document an audit or a deviation from a FSP or the Round 2 QAPP:

- Corrective action reports will be used, as necessary, to document any problems encountered during field activities and corrective actions taken.
- Field change request forms will be used, as necessary, to document the need for a procedural change or a station location change.
- System and performance audit reports will be used, as necessary, to document review or audit of field sampling activities.

The Sampling and Analysis Coordinator will ensure that the field team receives the final approved version of each FSP and this QAPP prior to the initiation of field activities.

A9.2 Laboratory Documentation

All activities and results related to sample analysis will be documented at each laboratory. Internal laboratory documentation procedures are described in the laboratory QA manuals (Appendices C - F of this QAPP).

Each laboratory will provide a data package for each sample delivery group or analysis batch that is comparable in content to a full Contract Laboratory Program (CLP) package. The format of the data may differ from CLP requirements. Each data package will contain all information required for a complete QA review, including the following:

- A cover letter discussing analytical procedures and any difficulties that were encountered
- A case narrative referencing or describing the procedures used and discussing any analytical problems and deviations from SOPs and this QAPP
- COC and cooler receipt forms
- A summary of analyte concentrations (to two significant figures, unless otherwise justified), method reporting limits, and method detection limits
- Laboratory data qualifier codes appended to analyte concentrations, as appropriate, and a summary of code definitions
- Sample preparation and cleanup logs
- Instrument tuning check data

- Initial and continuing calibration data, including instrument printouts and quantification summaries, for all analytes
- Results for method and calibration blanks
- Results for all QA/QC checks, including but not limited to surrogate spikes, internal standards, laboratory control samples, matrix spike samples, matrix spike duplicate samples, and laboratory duplicate or triplicate samples provided on summary forms
- Instrument data quantification reports for all analyses and samples
- Florisil® check data and GPC calibration data
- Copies of all laboratory worksheets and standards preparation logs.

Data will be delivered in both hard-copy and electronic format to the Integral Chemistry QA Manager, who will be responsible for oversight of data verification and validation and for archiving the final data and data quality reports in the project file. A copy of the first data package for each sample type and method will be provided to the EPA QA manager upon receipt from each laboratory. Integral will maintain data packages and EDDs for chemical analyses, and Windward will maintain hard copy and electronic data for bioassays.

Electronic data deliverables will be compatible with Integral's EQuIS database.

Laboratory data will be maintained by each laboratory for a period of at least 5 years. These data will include the original instrument data files, reduced and verified data stored in the laboratory information management system (LIMS,) and final hard-copy and electronic data deliverables. The laboratories will obtain approval from LWG prior to discarding these data.

A9.3 Data Quality Documentation

Data validation reports will be prepared by the contracted validation firm and provided to the Chemistry QA Manager. Results of the validation reports will be summarized in the applicable site characterization summary report for each sampling event. Any limitations to the usability of the data will also be discussed in this report.

All electronic database entries provided by each laboratory will be verified against the validated hard-copy data in the data package. All changes to the database will be documented in an electronic log file that automatically enters a current time stamp when opened and allows the data editor to enter notes about changes to the database. Any data tables prepared from the database for data users will include all qualifiers that were applied by the laboratories and during data validation, unless otherwise requested.

SECTION B: DATA GENERATION AND ACQUISITION

B1 SAMPLING PROCESS DESIGN

The Round 2 sampling effort will include the following sampling activities:

- Surface sediment and beach sediment samples collected with either a handheld coring device (subaerial beaches), small dredge sampler (shallow water beaches), or power grab sampler (river channel and river nearshore areas)
- Subsurface sediment cores collected with a vibracoring device.

The sampling stations for Round 2 are located between RM 2 and RM 11 of the Willamette River. The complete sampling design (including station locations, types and numbers of samples that will be collected, the rationale for collection, and the analysis that will be performed), as well as detailed sample collection and handling methods, are described in the Sediment Sampling and Benthic Toxicity Testing and the Shorebird Area and Beach Sediment FSPs (Integral and Windward 2004; Integral et al. 2004). The sampling programs are summarized below.

B1.1 River Surface and Subsurface Sediment

The river sediment sampling program is based on one proposed by EPA in November 2003 (see Appendix A of Integral and Windward 2004). Approximately 1,000 surface sediment and subsurface sediment samples will be collected during Rounds 2A and Round 2B, as summarized in Section A6.1 and described in detail in the sediment FSP (Integral and Windward 2004).

Field replicates will be collected at 5% of the sampling stations. Two field splits will be collected at 2.5% of the stations. One field split will be analyzed by the LWG, and the second will be provided to EPA for analysis at its laboratory in Manchester, WA. Equipment rinsate blanks will be collected at a frequency of 5% of field samples. A trip blank will be included in each cooler that contains samples to be analyzed for VOCs. Field QC samples are described further in Section B5 of this QAPP and Section 4.9 of the Sediment FSP (Integral and Windward 2004). The anticipated total numbers of field QC samples associated with surface and subsurface river sediment sampling are summarized in Table 4-3 of the Sediment Sampling and Benthic Toxicity Testing FSP (Integral and Windward 2004). The total number of trip blanks that will be required will be determined in the field based on the number of coolers that contain samples for volatile organics analyses.

B1.2 Shorebird Area and Beach Sediment

Composite grab surface sediment samples will be collected from shorebird habitat areas and human use areas. Sampling of shorebird areas will be performed by late

July 2004. Human use beach area samples will be collected during low water in August or September. Each composite sample will include discrete samples collected along shoreline transects that parallel the water line, as specified in the FSP (Integral et al. 2004). Field replicate samples, field split samples, and equipment rinse blanks will be collected at a frequency of 5%. The anticipated total numbers of field QC samples associated with shorebird area and beach sediment sampling are summarized in Table 3-2 of Integral et al. (2004).

B2 SAMPLING METHODS

This section outlines sample collection methods, equipment, and sample requirements for the various types of Round 2 samples. Additional details not provided here are included in the respective FSPs (Integral and Windward 2004; Integral et al. 2004).

B2.1 Sampling Methods

The sample collection procedures for the various types of Round 2 samples are summarized below.

B2.1.1 River Surface and Subsurface Sediment

Surface (0-30 cm) sediment grab samples will be collected from river channel and nearshore areas using a power grab sampler deployed from a sampling vessel. Subsurface sediment will be collected at selected surface sampling stations using vibracoring tubes (either 14 feet or 20 feet in length) deployed from a sampling vessel. The entire recovered core length will be described and sampled in subsurface intervals that are based on the lithologic units observed in each core. Sample intervals will be limited to between 1 to 4 feet thick. Generally, only samples from the top two subsurface intervals will be initially submitted for analysis. The surface interval and remaining subsurface intervals will be archived. Exceptions will be made if analytes with short holding times (i.e., VOCs, TPH as gasoline, or hexavalent chromium) are required at a station, in which case the bottommost sample interval will be submitted for those analyses. Core tube lengths and strategies for sectioning the cores at each station are provided in Table 2.4 of the Sediment FSP (Integral and Windward 2004). SOPs and additional sampling details specific to cores intended to support the FS and the CSM are also provided in the Sediment FSP.

Samples from each surface station and selected samples from each subsurface station will be described and submitted for SVOCs and metals analyses. Samples from selected surface and subsurface locations may be submitted for additional analyses, including chlorinated pesticides, PCB Aroclors, PCB congeners, TPH, butyltins, VOCs, dioxins and furans, chlorinated herbicides, and hexavalent chromium. Selected surface samples will be submitted for bioassays (see Appendix B).

B2.1.2 Shorebird Area and Beach Sediment

Composite grab samples of beach surface (0-15 cm) sediment will be collected with either a handheld coring device (subaerial locations) or a small dredge sampler (shallow water locations). The grab samples may be collected from shore or from a sampling vessel. Each component grab sample will be individually described, and the composite will be homogenized and submitted for analyses that include grain size, mercury, metals, total solids, SVOCs, organochlorine pesticides, PCB Aroclors and congeners, dioxins and furans, and TOC. A jar of each composite sample will be archived (-20° C) at CAS pending further analysis. Additional details of the beach sediment sampling methods are provided in the FSP (Integral et al. 2004).

B2.2 Sample Requirements

Sample containers, sample size requirements, preservation, and holding times for sediment samples and equipment rinse blanks are summarized in Table B2-1. All containers will have screw-type lids to ensure adequate sealing of the bottles. Lids of glass containers will have Teflon inserts to prevent sample contact with the plastic lid and to improve the quality of the seal.

Sample containers will be supplied by the laboratories. Commercially available pre-cleaned jars will be used, and each laboratory will maintain a record of certification from the suppliers. The bottle shipment documentation will include batch numbers for the bottles. With this documentation, bottles can be traced to the supplier, and bottle wash analysis results can be reviewed. The bottle wash certificate documentation will be archived in the Integral project file.

Prior to shipment to the field, the project laboratories will add the required preservatives to the sample bottles (i.e., for equipment rinse blanks) and supply additional preservative in a transportable container. The laboratory will note the lot number of the preservative on the bottle kit paperwork.

B2.3 Corrective Actions

The Field QA Manager will conduct field performance audits at least once during each field program, which may identify the need for corrective actions. The Field QA Manager will immediately institute the necessary corrective actions, complete a corrective action form (Figure B2-1), and conduct an additional audit to ensure that the correct procedures continue to be followed.

If corrective actions require a departure from the FSP, these changes will be documented on a field change request form (see Appendix D, Integral and Windward 2004). In any other circumstances where sampling conditions are unexpected, the appropriate sampling actions consistent with project objectives will be conducted. This change will be noted in the field log, and a change request form will be completed for the project files. Additional information regarding corrective actions and related documentation is provided in Section C1.

B3 SAMPLE HANDLING AND CUSTODY

B3.1 Field to Laboratory Sample Handling and Custody

Detailed descriptions of procedures for sample identification, handling, documentation, custody, and ultimate disposal are presented in the FSPs for Round 2 (Integral and Windward 2004; Integral et al. 2004).

From the time of collection, all samples and sediment cores awaiting processing will be stored on ice or refrigerated at an approximate temperature of 4ºC.

The field task leader, or the designated field sample custodian, will be responsible for sample tracking in the field. Samples will remain in Integral's custody until COC forms and final sample inventory are completed in the field or at the field sample processing facility. COC forms will be used for samples that are in transit from the field site to the testing laboratories. The custodian will relinquish the samples to a designated analytical laboratory courier service or commercial transport company (e.g., FedEx). Custody of samples in the field or at the field processing facility will be documented in the field logs.

Samples are considered to be in custody if they are 1) in the custodian's possession or view, 2) in a secured location (under lock) with restricted access, or 3) in a container that is secured with an official seal(s) such that the sample cannot be reached without breaking the seal(s). The principal documents used to identify samples and to document possession are COC records, field logbooks, and field tracking forms. COC procedures will be used for all samples at all stages in the analytical or transfer process and for all data and data documentation whether in hard-copy or electronic format. An example of a COC form is provided in Figure B3-1.

Sample packing and shipping procedures are detailed in the FSPs for Round 2 (e.g., Integral and Windward 2004; Integral et al. 2004). Samples will be shipped to the laboratory in ice chests sealed with custody seals. Each ice chest will have three seals, one on the front of the chest and one on each side. The laboratory sample custodian will establish the integrity of the seals at the laboratory. The way bill of the carrier used to ship samples will provide additional custody and sample tracking information. The way bills will be maintained in the project file.

The sample custodian at each laboratory will accept custody and log samples into the LIMS. The sample custodian will check that the COC forms were properly completed and signed, that a sample receipt form is completed for each cooler, and that samples are stored under the required temperature conditions. Each laboratory will deliver a copy of the COC and sample receipt form to the Chemistry QA Manager. Any breaks in the COC or non-conformances will be noted and reported in writing to the Chemistry QA Manager within 24 hours of receipt of samples.

Specific laboratory COC procedures are described in the laboratory QA manual for each of the designated labs (Appendices C-F).

B3.2 Intra-Laboratory and Sub-Laboratory Sample Transfer

The Laboratory Project Manager will ensure that a sample-tracking record is maintained that follows each sample through all stages of laboratory processing. The sample-tracking record must contain, at a minimum, the names of individuals responsible for performing the analysis; dates of sample extraction, preparation, and analysis; and the type of analysis being performed.

Any sample needing further analysis that is not performed by the initial contracted laboratory will be subject to all custody specifications provided in the previous section.

B3.3 Archived Samples

Archive samples will be collected for all of the sediment samples (i.e., beach sediment, surface sediment, and sediment core samples). Up to 16 ounces of sediment will be collected for archiving for samples that are scheduled for analysis, as available. In addition, any sample remaining in the sample jars after aliquots are removed for analysis by the laboratory will be archived. For sediment core samples that are not scheduled for analysis, 32 ounces will be collected for archiving.

All archive samples will be submitted to CAS and will be stored at -20±4°C. Sediment remaining after analysis will be archived by the laboratory that completed the analysis. The laboratories will maintain COC documentation and proper storage conditions for the entire time that the samples are in their possession. All laboratories for this project will store the archive and excess samples for 12 months following completion of data validation.

B4 ANALYTICAL METHODS

Laboratory methods to be used for Round 2 are consistent with requirements provided in SW-846 (EPA 2004), PSEP protocols and guidelines (PSEP 1986, 1997a,b), and other established and widely accepted protocols. Modifications will be made to these methods, as necessary and technically feasible, to improve MRLs. Analytes, MRLs, and ACGs for sediment samples are provided in Table A6-2. Method modifications will not be sufficient to reduce MRLs to the level of the ACGs for several analytes, and ACGs will not be attained in these cases. Laboratory methods are described below for sediment samples. Methods for surface water and groundwater samples will be addressed in addenda to this QAPP.

Sediment samples to be collected for Round 2 include surface sediment, sediment cores, and beach sediment. The methods described in this section apply to all of these sediment types. Sediment samples will be analyzed for the following:

- Conventional analyses
- Geotechnical characteristics
- Metals
- Butyltin compounds
- Petroleum hydrocarbons
- Chlorinated herbicides
- Organochlorine pesticides
- PCB Aroclors
- VOCs
- SVOCs
- PCB congeners
- Chlorinated dioxins and furans.

The total number of samples and the analyses that will be conducted on each sample are indicated in each FSP (Integral and Windward 2004; Integral et al. 2004). The laboratory methods for sample preparation and analysis are summarized in Table A6-1.

B4.1 Conventional Analyses in Sediment

Conventional analyses of sediment samples will include total solids, grain-size distribution, total sulfides, ammonia, and TOC. EPA and PSEP methods will be used as shown in Table A6-1.

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.

Grain-size analysis will also be completed using PSEP (1986) protocols. Organic material in the samples will not be oxidized prior to analysis. Sieve sizes 4, 10, 18, 35, 60, 120, and 230 will be used to determine gravel and sand fractions, and phi size intervals 4-5, 5-6, 6-7, 7-8, 8-9, 9-10, and >10 will be determined for the silt and clay fractions using the pipette method.

Total sulfide analysis will include distillation of the sulfide into a sodium hydroxide trap and analysis by colorimetry.

Ammonia will be analyzed by EPA Method 350.1. 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 EPA Method 9060. Samples will be pretreated with hydrochloric acid to remove inorganic carbon, dried at 70° C, and analyzed by combustion in an induction furnace.

B4.2 Metals and Butyltin Compounds in Sediment

Up to four different methods will be used to analyze sediment samples for total metals (Table A6-1). Strong acid digestion with nitric acid and hydrogen peroxide will be used to prepare samples for analysis of metals other than mercury and hexavalent chromium. Analysis will be completed by inductively coupled plasma/mass spectrometry (ICP/MS). If the results for arsenic and selenium are below the MRL by ICP/MS, these metals will additionally be analyzed by hydride generation and atomic absorption spectrometry (AAS) to meet MRLs provided in Table A6-2. Based on sediment data collected during Round 1, it is anticipated that most or all of the Round 2 sediment samples can be analyzed for arsenic by ICP/MS, and that most or all of the selenium analyses will be completed by hydride generation and AAS.

Mercury samples will be extracted with aqua regia and oxidized using potassium permanganate. Analysis will be completed by cold vapor atomic absorption spectrometry (CVAA). A buffered solution of sodium hydroxide and sodium carbonate will be used to extract hexavalent chromium from the sediment samples. The extracted hexavalent chromium will be analyzed by colorimetry.

The procedure for butyltins will include extraction, derivitization, and analysis of mono-, di-, tri-, and tetrabutyltin. Tropolone in methylene chloride will be used to extract the butyltins, followed by a Grignard reaction with hexylmagnesium bromide to form hexyl derivatives. The extract will be cleaned up using silica and alumina cartridge columns and analyzed by gas chromatography/flame photometric detection (GC/FPD).

B4.3 Organic Compounds in Sediment

Samples for gasoline-range hydrocarbons will be extracted with methanol followed by purge and trap with a carbon-based trap. The contents of the trap will be analyzed by GC with a flame ionization detector (FID). For diesel- and residual-range petroleum hydrocarbons, the samples will be extracted with methylene chloride and analyzed by GC/FID. Hydrocarbon identification will be completed for all petroleum hydrocarbon analyses. Chromatograms for standards used to establish hydrocarbon identification will be provided with the final data.

Chlorinated herbicides will be extracted with methanolic potassium hydroxide. The sample will then be acidified and extracted with ethyl ether and methylene chloride. The extract will be concentrated, and ester derivatives will be formed using diazomethane. Extracts will be analyzed by GC/MS rather than the customary gas chromatography/electron capture detector. This instrumentation, listed as an option in EPA Method 8151A, will provide lower reporting limits overall and mass spectral identification of the herbicides. However, for Round 2 the MRLs for 2,4,5-T and 2,4,5-TP will be 8 µg/kg for both analytes, above their ACGs of 2.8 and 2.2 µg/kg, respectively. The MRLs were below these ACGs for most of the Round 1 sediment samples. The detection limits for Round 2 are expected to be below the ACGs for all additional herbicide parameters and for pentachlorophenol. Pentachlorophenol will be added to the analyte list for herbicides to attain a lower MRL than can be provided by GC/MS, the customary method for this analyte.

Organochlorine pesticides will be extracted from samples using Soxhlet extraction procedures. PCBs (for Aroclor analysis) will be extracted from samples using pressurized fluid extraction procedures. This method provides extraction efficiencies similar to Soxhlet extraction (EPA 2004).

For PCB Aroclor analysis, a 25-gram sample aliquot will be extracted and the final extract volume will be 10 mL. Acid cleanup (EPA SW-846 Method 3665) will be performed on the sample extract followed by Florisil (EPA SW-846 Method 3620) and finally sulfur removal by tetrabutylammonium sulfite procedure (EPA SW-846 Method 3660). The surrogate compounds, tetrachloro-m-xylene and decachlorobiphenyl, will be added to every sample, QC sample, and to selected instrument standards. For GC analysis, splitless injection will be used to optimize the MRLs. The low-level calibration standard will be 5 ng/mL on column with a resultant MRL of 4.0 µg/kg, dry weight, for each Aroclor, assuming 50% solids. The actual MRL for each sample will be higher or lower in direct proportion to the solids content. NEA will complete an MDL study and initial demonstration of proficiency using the proposed method modifications prior to analysis of project samples.

For organochlorine pesticide analysis, a 25-gram sample aliquot will be extracted and the final extract volume will be 5 mL. Florisil column clean-up (EPA SW-846 Method 3620) will be performed on the sample extract followed by sulfur removal by tetrabutylammonium sulfite procedure (EPA SW-846 Method 3660). The surrogate compounds, tetrachloro-m-xylene and decachlorobiphenyl, will be added to every sample, QC sample, and to selected instrument standards. For GC analysis, a splitless injection will be used to optimize the MRLs. The low-level calibration standard will be 0.40 ng/mL on column. An MRL of 0.20 µg/kg, dry weight, is expected for the single-component pesticides, assuming 50% solids. The actual MRL for each sample will be higher or lower in direct proportion to the solids content. NEA will complete an MDL study and initial demonstration of proficiency using the proposed method modifications prior to analysis of project samples. Pesticide detections will be confirmed using the instrument data for the GC/MS analysis when concentrations are sufficiently high.

VOCs will be analyzed by purge and trap extraction and GC/MS. The mass spectrometer will be operated in full-scan mode. Tentatively identified compounds (TICs) will not be reported. The method will be modified to incorporate a larger sample mass (10 grams rather than 5 grams) and lower-range calibration to improve

MRLs. This will result in reporting limits of 1 µg/kg for most compounds (Table A6-2).

Sample extractions for SVOCs will be completed using pressurized fluid extraction and 50 grams of sample (wet weight). The extract volume will be reduced to 10 mL, of which 8.0 mL will be processed through GPC. A final extract volume of 0.4 mL will be used. SVOCs will be analyzed by GC/MS with ion trap to enhance sensitivity. TICs will not be reported. The mass spectrometer will be operated in full-scan mode with increased ion multiplier settings to meet project sensitivity requirements. To optimize MDLs and MRLs for analytes with very low ACGs, the mass spectrometer will be calibrated utilizing a low-level calibration standard at 0.25 µg/mL. A higher low-level calibration standard will be utilized for the remaining analytes. This procedure is expected to provide MRLs that are comparable to those obtained during Round 1 analyses without the requirement for a second analysis by selected ion monitoring (SIM). NEA will complete an MDL study and initial demonstration of proficiency using the proposed modifications prior to analysis of project samples.

B4.4 PCB Congeners and Chlorinated Dioxins and Furans in Sediment

PCB congeners in sediment samples will be extracted with hexane in a Soxhlet/Dean Stark extractor. Samples will be analyzed by high-resolution gas chromatography with high-resolution mass spectrometry (HRGC/HRMS). As described in EPA Method 1668A, detection limits are calculated on an individual compound and sample basis and depend on signal-to-background ratio for the specific labeled isomer.

The specific PCB congener analyte list has not yet been determined. LWG and EPA data users will continue to discuss the intended uses of the PCB congener data (e.g., exposure for shorebirds, food web model) to determine if selected sediment samples will be analyzed for a subset of PCB congeners (e.g., the coplanar PCB congeners) or the full list of 209 PCB congeners. The ACGs for the PCB congeners will be determined by the LWG and EPA data users based on the intended uses of the data and sensitivity required for these uses. The following method modifications will be used, as necessary, to optimize the analytical method to improve the MDLs and approach or meet the ACGs:

- The sample size of 10 grams specified by EPA Method 1668A will be increased as necessary; up to 75 grams of sample may be extracted.
- A smaller extract volume for the final extract may be used by the laboratory.
- The concentration of the calibration standards may be modified to incorporate a lower concentration standard.

- The carbon column option in Method 1668A may be used to separate the coplanar congeners from other congeners and interferences (e.g., residual lipids), which may improve the MDLs and eliminate coelutions for selected congeners.

After LWG and EPA data users resolve the PCB congener analyte list and ACGs, a QAPP addendum will be generated by the LWG. The QAPP addendum will include a description of the method modifications selected to achieve the MDLs, and the ACGs and MDLs will be added to Table A6-2.Chlorinated dioxins and furans in sediment samples will be extracted with toluene in a Soxhlet/Dean Stark extractor. To improve detection limits, the method will be modified to include extraction of up to 50 grams of sample rather than the standard sample mass of 10 grams. Cleanup procedures will include sulfuric acid cleanup and silica/carbon column cleanup. Additional cleanup procedures will be used as necessary. Samples will be analyzed by HRGC/HRMS. As described in EPA Method 1613B, detection limits 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 A6-2 are representative of MDLs typically obtained when using the described procedure and sample mass..

B5 QUALITY CONTROL

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

B5.1 Field Quality Control Samples

Field QC samples for this study will include field replicates, field splits, equipment rinse blanks, trip blanks, and temperature blanks. Field replicates are samples collected at the same station, but using different deployments of the sampling equipment. The data for field replicates are used to evaluate variability at the sampling site.

Field splits are different subsamples of a single homogenized sample. Data for field splits are used to evaluate sample collection procedures, and, in the case of analysis by different laboratories, interlaboratory precision.

Equipment rinse blanks are collected by pouring water over the sample homogenization equipment (i.e., for sediment samples). Equipment rinse blanks are used to monitor equipment decontamination procedures and to check for other sources of contamination.

Trip blanks are used to monitor for cross-contamination during sample shipment and storage. Trip blanks are used only for samples that will be analyzed for volatile constituents (i.e., VOCs and gasoline-range petroleum hydrocarbons).

A temperature blank (a jar filled with water) is included with each cooler of samples. The laboratory sample custodian measures the temperature of the water in the jar to determine the temperature of the cooler upon receipt at the laboratory.

Field replicates and equipment rinse blanks will minimally be collected for each type of sample (i.e., surface sediment, sediment cores, and beach sediment) at a frequency of 5% of the sample total. Field splits will minimally be collected at a frequency of 2.5% for sediment samples. Two field splits will be collected for the designated sediment samples. One split will be provided to EPA for analysis at its laboratory in Manchester, WA, and the other will be analyzed by the LWG-contracted laboratories.

Procedures for preparing field QC samples are provided in the Sediment Sampling and Benthic Toxicity Testing FSP (Integral and Windward 2004). Validation criteria and procedures for field QC samples are described in Sections D1 and D2.

B5.2 Laboratory Quality Control

Extensive and detailed requirements for laboratory QC procedures are provided in the method protocols that will be used for this study (Table A6-1). Every method protocol includes descriptions of QC procedures, and many incorporate additional QC requirements by reference to separate QC chapters. QC requirements include control limits and, in many cases, requirements for corrective action. QC procedures will be completed by the laboratories, as required in each method protocol and as indicated in this QAPP.

The frequency of analysis for laboratory control samples, matrix spike samples, matrix spike duplicates or laboratory duplicates, and method blanks will be one for every 20 samples or one per extraction batch, whichever is more frequent. Surrogate spikes and internal standards will be added to every field sample and QC sample, as required by the method. Calibration procedures will be completed at the frequency specified in each method description. As required for EPA SW-846 methods (EPA 2004), performance-based control limits have been established by each laboratory. These and all other control limits specified in the method descriptions will be used by the laboratories to establish the acceptability of the data or the need for reanalysis of the samples. Control limits for surrogate compounds, LCS/LCSDs, and MS/MSDs are provided in Tables A7-1 and A7-2.

Where discrepancies exist between this QAPP and laboratory SOPs and QA Manuals, this QAPP will take precedence.

Storage blanks for VOCs in sediment will be prepared and analyzed at the laboratory according to standard laboratory procedures. A pair of VOC blanks will be prepared for each cooler at the laboratory, but only one of the blanks will be shipped to the field. This blank will become a trip blank in a cooler of samples. The second blank will be retained at the laboratory and will become the storage blank when the cooler is received at the laboratory. A storage blank will be randomly selected for analysis on a weekly basis. In addition, the storage blank associated with a given cooler will be analyzed if results for the trip blank indicate that samples may be contaminated. Results for the storage blank will indicate whether contamination is related to field and shipping conditions or to laboratory storage conditions.

B6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

Analytical instrument testing, inspection, maintenance, setup, and calibration will be conducted by each laboratory in accordance with the requirements identified in the laboratory SOPs and manufacturer instructions. Instrument maintenance and repair will be documented in maintenance logs or record books.

B7 INSTRUMENT/EQUIPMENT CALIBRATION AND FREQUENCY

Laboratory instruments will be properly calibrated, and the calibration will be verified with appropriate check standards and calibration blanks for each parameter before beginning each analysis. Instrument calibration procedures and schedules will conform to analytical protocol requirements and descriptions provided in the laboratories' QA manuals and SOPs.

All calibration standards will be obtained from either the EPA repository or a commercial vendor, and the laboratory will maintain traceability to the National Institute of Standards and Technology. Stock standards will be used to make intermediate standards and calibration standards. Special attention will be given to expiration dating, proper labeling, proper refrigeration, and prevention of contamination. Documentation relating to the receipt, mixing, and use of standards will be recorded in a laboratory logbook. All calibration and spiking standards will be checked against standards from another source.

Field instruments will not be used in connection with sediment sampling for Round 2.

B8 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

The quality of supplies and consumables used during sample collection and laboratory analysis can affect the quality of the project data. All equipment that comes into contact with the samples and extracts must be sufficiently clean to prevent detectable contamination, and the analyte concentrations must be accurate in all standards used for calibration and quality control purposes.

During sample collection, solvents of appropriate, documented purity will be used for decontamination. Solvent containers will be dated and initialed when they are opened. The quality of laboratory water used for decontamination will be documented at the laboratory that provides that water. As discussed in Section B2, cleaned and documented sample containers will be provided by the laboratories. All containers will be visually inspected prior to use, and any suspect containers will be discarded.

Reagents of appropriate purity and suitably cleaned laboratory equipment will also be used for all stages of laboratory analyses. Details for acceptance requirements for supplies and consumables at the laboratories are provided in the laboratory SOPs and QA manuals (Appendices C – F). All supplies will be obtained from reputable suppliers with appropriate documentation or certification. Supplies will be inspected to confirm that they meet use requirements, and certification records will be retained by Integral (i.e., for supplies used in the field) or the laboratories.

B9 NON-DIRECT MEASUREMENTS

Existing chemical data from previous investigations in Portland Harbor will be used for the RI/FS. All historical data have been reviewed for quality assurance. Details are provided in the Work Plan (Integral et al. 2004). Non-direct measurements will not be needed for the completion of Round 2 activities.

B10 DATA MANAGEMENT

Data for Round 2 will be generated in the field and at the laboratories. The final repository for all sample information will be an EQuIS™ database. Procedures to be used to transfer data from the point of generation to the EQuIS™ database are described in this section.

B10.1 Field Data

Daily field records (a combination of field logbooks and field data sheets) and navigational records will make up the main documentation for field activities. The records and procedures most applicable to field activities are summarized in Section 6.1 (field logbooks), Section 6.2 (field data sheets), and Section 6.3 (field data management) of Integral and Windward (2004). A detailed data management plan is provided as an appendix to the Work Plan (Integral et al. 2004).

Data that are generated during sample collection and sample preparation will be manually entered into the field logbook and core logs. Data from these sources will be entered into an Excel® workbook template directly from the field logbook and core logs. These data include station location coordinates, station names, sampling dates, sample identification codes, and additional station and sample information (e.g., water depth, sample type, field split number). A second individual will review all entries for accuracy and completeness, and any errors will be corrected before the data are uploaded to the EQuIS™ database and approved for release to data users.

The completed Excel® workbook templates will be used to automatically generate COC/analysis request forms at the time that samples are packaged for delivery to analytical laboratories. The automatically generated forms will eliminate the potential for transcription errors and other commons errors associated with hand-written forms.

B10.2 Laboratory Data

A wide variety of manually entered and electronic instrument data are generated at the laboratories. Data are manually entered into:

- Standard logbooks
- Storage temperature logs
- Balance calibration log
- Instrument logs
- Sample preparation and analysis worksheets
- Maintenance logs
- Individual laboratory notebooks
- Results tables for conventional analyses (i.e., grain-size distribution, total solids, and TOC).

The LIMS is the central data management tool for each laboratory. All manual data entry into the LIMS is proofed at the laboratory. All data collected from each laboratory instrument, either manually or electronically, are reviewed and confirmed by analysts before reporting. The LIMS is used for every aspect of sample processing, including sample log-in and tracking, instrument data storage and processing, generation of data reports for sample and QC results, and preparation of EDDs.

Laboratory data will be entered directly into the EQuIS™ database from the EDD. A database printout will be used to verify database entries against the hard-copy laboratory data packages. Electronic data will also be provided to EPA in a format that is compatible with NOAA’s Query Manager database system.

SECTION C: ASSESSMENT AND OVERSIGHT

A formal chain of communication has been established for this project to optimize the flow of information and to keep the management team apprised of activities and events. The field team will stay in close verbal contact with the Sampling and Analysis Coordinator, Field Coordinator, and Chemistry and Bioassay QA Managers during all phases of the project. These individuals will, in turn, keep the CERCLA Coordinator informed of any significant developments in the field or at the laboratories.

C1 ASSESSMENTS AND RESPONSE ACTIONS

Assessment activities for Round 2 sampling will include 1) formal field performance and technical audits performed by the Field QA Manager at least once during each field sampling event; 2) a technical systems audit (TSA) of each laboratory conducted by the Chemistry QA Manager or her designee before sample collection commences; 3) a TSA of the team's data management systems conducted by the Chemistry QA Manager or her designee at least once during any phase of the project; and 4) routine internal performance and peer reviews of each phase of project tasks throughout the duration of the project.

C1.1 Technical Systems Audits

TSAs will be completed once by the Field QA Manager for each sampling program and may be conducted at any time if serious problems are encountered during sampling operations. The scheduled field audits are described in Section B2.3. At EPA's discretion, an EPA field staff member, hydrogeologist, and/or QA Manager may accompany the Field QA Manager on any field audit.

A TSA of laboratory procedures will be completed by the Chemistry QA Manager or her designee for each laboratory, except the CAS facility in Redding, prior to the start of sample collection. At EPA's discretion, the EPA QA Manager may accompany the Chemistry QA Manager or her designee on any of the laboratory audits. Additional audits may be conducted by or under the direction of each laboratory's QA Manager, the Chemistry QA Manager and/or the EPA QA Manager if laboratory performance issues are identified. The Chemistry QA Manager will accompany the EPA QA Manager on any audits initiated by EPA.

The Chemistry QA Manager or her designee will additionally conduct a TSA of Integral's data management systems. This audit will include a review of procedures for managing data provided by the field teams, the laboratories, and the data validation firm; management of the database and related documentation; and preparation of electronic data transmittals for data users and for EPA.

Results of all audits will be provided in the applicable site characterization summary report for each sampling event.

C1.2 Routine Internal Performance Reviews and Corrective Action

Readiness reviews are conducted to ensure that all necessary preparations have been made for efficient and effective completion of each critical phase of project work. Field readiness reviews will be conducted prior to initiation of each field sampling event. The Field Task Leader will verify that all field equipment is ready for transfer to the site. The Field Task Leader will also verify that the field team and subcontractor have been scheduled and briefed and that the contract for the subcontractor has been signed by both parties. Any deficiencies noted during this readiness review will be corrected prior to initiation of sampling activities.

A readiness review will also be completed before final data are released for use for each field program. The data manager will verify that all results have been received from the laboratories, data validation and data quality assessment have been completed for all of the data, and data qualifiers have been entered into the database and verified. Any deficiencies noted during this review will be corrected by the data manager, the Chemistry or Bioassay QA Manager, or their designee. Data will not be released for final use until all data have been verified and validated. No report will be prepared in conjunction with the readiness reviews. However, the project manager and data users will be notified when the data are ready for use.

Technical review of intermediate and final work products generated for this project will be completed throughout the course of all sample collection, laboratory analysis, data validation, and data management activities to ensure that every phase of work is accurate and complete and follows the QA procedures outlined in this QAPP and in the FSPs for Round 2 field work. Any problems that are encountered will be resolved between the reviewer and the person completing the work. Any problems that cannot be easily resolved or that affect the final quality of the work product will be brought to the attention of the Integral and EPA project managers. EPA will be notified of any problems that may affect the final outcome of the project.

Each laboratory has implemented a review system that serves as a formal surveillance mechanism for all laboratory activities. The analyst initially verifies the accuracy of the data and conformance of calibrations and QC results to control limits. A second review of sample, calibration, and QC results is conducted by the section supervisor, a senior chemist, or other qualified personnel, as designated by each laboratory. Details are provided in the laboratory QA manuals (Appendices C – F).

Any project team member who discovers or suspects a non-conformance is responsible for reporting the non-conformance to the CERCLA Project Coordinator, the Sampling and Analysis Coordinator, the Field Task Leader, or the Chemistry or Bioassay QA Manager, as applicable. The Project Coordinator will ensure that no additional work dependent on the non-conforming activity is completed until a confirmed non-conformance is corrected.

When a non-conformance is identified, a corrective action plan will be prepared. The plan will include identifying the corrective action, the person or organization responsible for implementing the corrective action, and procedures for confirming that the desired results are produced. The corrective measures will be appropriate to the severity of the non-conformance and realistic in terms of the resources required for implementation.

Corrective action reports (Figure B2-1) will be used to document non-conformances and subsequent corrective actions. The Sampling and Analysis Coordinator will review these reports and approve the corrective action. The CERCLA Project Coordinator may also submit the corrective action reports to the LWG or EPA, as appropriate. The Sampling and Analysis Coordinator is ultimately responsible for implementation of appropriate corrective action and maintenance of a complete record of QC issues and corrective actions. The Laboratory Project Manager and Chemistry and Bioassay QA Managers are responsible for maintaining records of QC issues related to laboratory work. The Sampling and Analysis Coordinator will be responsible for evaluating all reported non-conformances, conferring with the CERCLA Project Coordinator, and executing the corrective action as developed and scheduled.

C2 REPORTS TO MANAGEMENT

A field sampling report will be prepared and submitted to EPA within 60 days of completing each type of sampling event (e.g., Round 2 surface sediment sampling, Round 2A sediment coring, beach sediment, and surface water) for Round 2. The field sampling reports will summarize field sampling activities, including sampling locations (maps), requested sample analyses, sample collection methods, and a discussion of any deviations from the FSP and QAPP.

Data packages and EDDs will be prepared by the laboratory upon completion of analyses for each sample delivery group. The case narrative will include a description of any problems encountered, control limit exceedances, and a description and rationale for any deviations from protocol. Copies of corrective action reports generated at the laboratory will also be included with the data package.

LWG-validated data for the sediment chemistry samples will be delivered to EPA in electronic format within 90 days of completing all Round 2 sampling and analysis activities for each sampling program. An extended schedule for data submittal and reporting will be determined by EPA and LWG if additional data validation is required beyond procedures described in this QAPP. The schedule for the bioassay data report is provided in the Toxicity Testing QAPP (Appendix B).

A data validation report will be prepared for each data package by the data validation firm. These reports and the validated data will be provided to the Chemistry QA Manager when validation is completed for each package. A summary of any significant data quality issues will be provided to EPA with the data submittal for each sampling effort.

The laboratories will keep the Chemistry QA Manager apprised of their progress on a weekly basis. The laboratories will provide the following information:

- Inventory and status of samples held at the laboratory in spreadsheet format by sample delivery group
- Summaries of out-of-control laboratory QC data and any corrective actions implemented
- Descriptions and justification for any significant changes in methodology or QA/QC procedures.

The Chemistry QA Manager will provide this information to the Sampling and Analysis Coordinator and CERCLA Project Coordinator.

The laboratories have implemented routine systems of reporting non-conformance issues and their resolution. These procedures are described in the laboratory QA manuals (Appendices C – F). Laboratory non-conformance issues will also be described in the applicable site characterization summary report for each sampling event if they affect the quality of the project data.

The status of field and laboratory activities will be provided to the EPA project managers on a monthly basis as part of the LWG's monthly progress report. The following information will be included in this report:

- Actions taken during the previous month
- Status of field and laboratory data
- Scheduled events for the following two months
- Problems encountered, anticipated delays, and solutions
- Documents and issues awaiting EPA's response.

This report will be prepared by the CERCLA Project Coordinator and will be supplied to EPA by the LWG.

SECTION D: DATA VALIDATION AND USABILITY

Data generated in the field and at the laboratories will be verified and validated according to criteria and procedures described in this section. Data quality and usability will be evaluated, and a discussion will be included in the applicable site characterization summary report for each sampling event.

D1 CRITERIA FOR DATA REVIEW, VERIFICATION, AND VALIDATION

Field and laboratory data for this project will undergo a formal verification and validation process. All entries into the database will be verified. All errors found during the verification of field data, laboratory data, and the database will be corrected prior to release of the final data.

Data verification and validation will be conducted in accordance with *Guidance on Environmental Data Verification and Validation* (EPA 2002c). Data verification and validation for organic compounds and metals will be completed according to methods described in the EPA guidance for data review (EPA 1995, 1996, 1999, 2002f). Performance-based control limits established by the laboratory and control limits provided in the method protocols will be used to evaluate data quality and determine the need for data qualification. Laboratory control limits for surrogate compounds, LCSs and LCSDs, and MS/MSDs are provided in Tables A7-1 and A7-2 and will be used for data validation.

No guidelines are available for validation of data for conventional analyses and physical testing. These data will be validated using procedures described in the functional guidelines for inorganic data review (EPA 2002f), as applicable.

Results for field splits and replicates will be evaluated against a control limit of 50 RPD. Data will not be qualified as estimated if this control limit is exceeded, but RPD results will be tabulated, and any exceedances will be discussed in the applicable site characterization summary report for each sampling event. Equipment rinse blanks will be evaluated and data qualifiers will be applied in the same manner as method blanks, as described in the applicable EPA guidance documents for data review (EPA 1995, 1996, 1999, 2002f).

Data will be rejected if control limits for acceptance of data are not met, as described in EPA (1995, 1996, 1999, 2002f).

D2 VERIFICATION AND VALIDATION METHODS

Field data will be verified during preparation of samples and COCs. Field data and COCs will be reviewed by the Field Task Manager on a daily basis. After field data are entered into the project database, 100% verification of the entries will be completed by a second party to ensure the accuracy and completeness of the database. Any discrepancies will be resolved before the final database is released for use.

Procedures for verification and validation of laboratory data and field QC samples will be completed as described in the following EPA guidance documents for data validation:

- *Guidance on Environmental Data Verification and Validation* (EPA 2002c)
- *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review* (EPA 1999)
- *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review* (EPA 2002f)
- *EPA Region 10 SOP for the Validation of Method 1668 Toxic, Dioxin-like PCB Data* (EPA 1995)
- *EPA Region 10 SOP for the Validation of Polychlorinated Dibenzodioxin (PCDD) and Polychlorinated Dibenzofuran (PCDF) Data* (EPA 1996)

Control limits that will be used to qualify data are described in Section D1, above.

The first data package generated for each sample type and analysis method will be fully validated by a data validation firm under contract to LWG. If no major problems are encountered during validation of this package, full validation will be completed at a rate of approximately 10% of the samples analyzed by each method. Validation for the remaining data will be based on review of the summary forms for sample and QC data. However, all chromatograms will be reviewed for PCBs to verify the identity of the reported Aroclor, and for pesticides to evaluate any effects of interference by PCBs.

A copy of the first data package for each sample type and method will be provided to the EPA QA Manager upon receipt from the laboratory. These data packages will be fully validated by EPA. If a need for additional or focused validation is identified during EPA's or LWG's review of data, a new schedule for completion of validation and data submittal and reporting will be established to accommodate the additional work.

Method reporting limit goals for this project are provided in Table A6-2. Reporting limits for non-detects will be compared to the method reporting limit goals to evaluate method sensitivity for each sample. Any exceedance of actual MRLs over the target MRLs will be discussed in the applicable site characterization summary report for each sampling event.

In addition to verification of field and laboratory data and information, data qualifier entries into the database will be verified. Any discrepancies will be resolved before the final database is released for use. The accuracy and completeness of the database will be verified at the laboratory and again as part of data validation. All entries to the database from the laboratory EDDs will be checked against the hard-copy data packages.

D3 RECONCILIATION WITH USER REQUIREMENTS

The goal of data validation is to determine the quality of each data point and to identify data points that do not meet the project criteria. Nonconforming data may be qualified as undetected, estimated, or rejected as unusable during data validation if criteria for data quality are not met. Rejected data will not be used for any purpose. An explanation of the rejected data will be included in the applicable site characterization summary report for each sampling event.

Data qualified as estimated will be used for all intended purposes (Section A5; Integral et al. 2004; Integral and Windward 2004) and will be appropriately qualified in the final project database. These data may be less precise or less accurate than unqualified data. The data users, in cooperation with Integral project management staff and the Chemistry QA Manager, will evaluate the effect of the inaccuracy or imprecision of the qualified data on site assessment and risk assessment procedures used to evaluate the Site.

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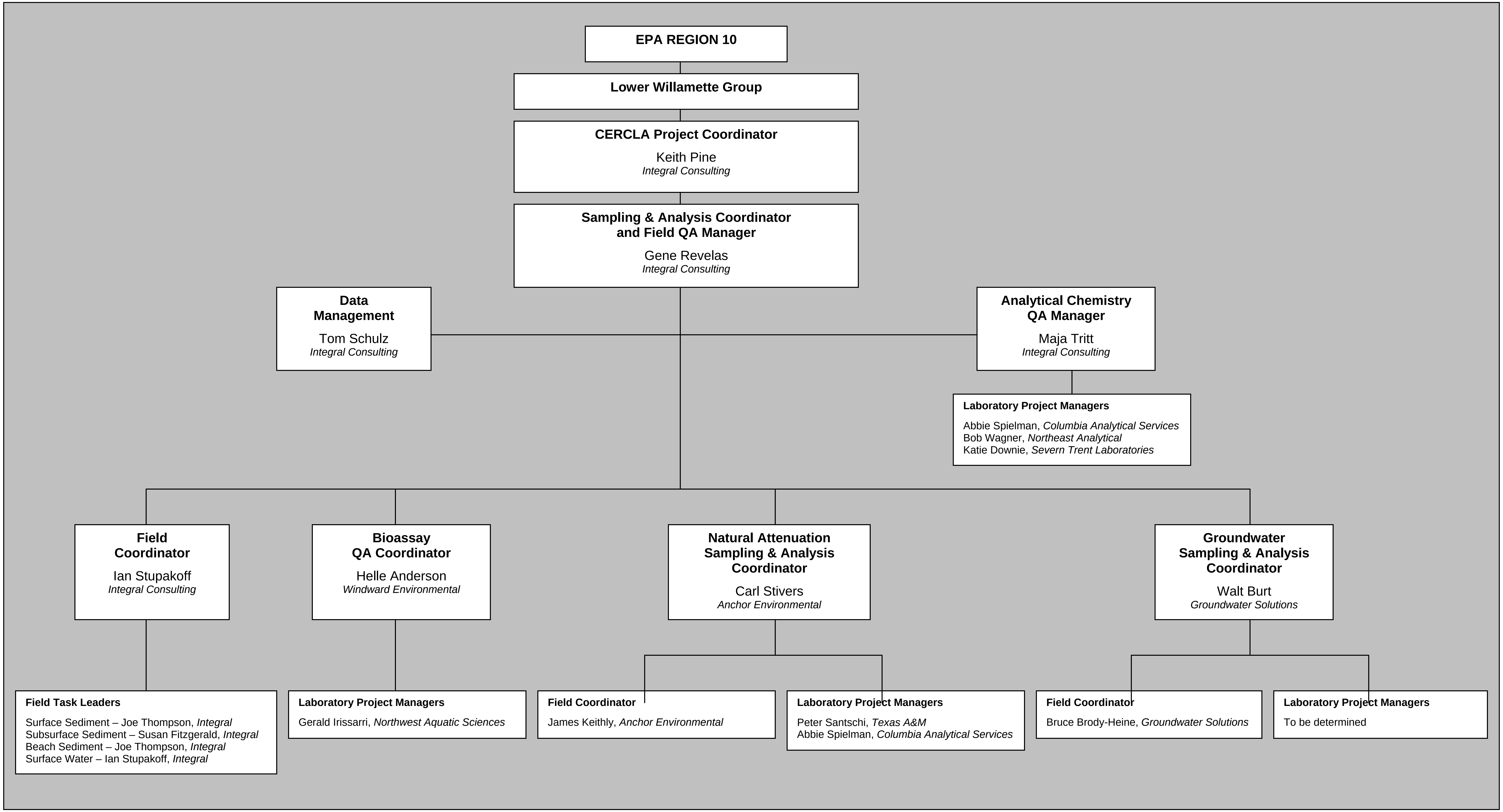

Figure A4-1. Round 2 Project Organization

CORRECTIVE ACTION RECORD

Page ____ of ____ Audit Report No.: ____________________________ Date: _____________________

Report Originator: __________________________________

Person Responsible for Response: _________________________________

DESCRIPTION OF PROBLEM:

Date and Time Problem Recognized: ___________________________________ By: _________________________________

Date of Actual Occurrence: _____________________________________ By: _________________________________

Analyte: _____________________________________ Analytical Method: _____________________________________

Cause of Problem:

CORRECTIVE ACTION PLANNED:

Person Responsible for Corrective Action: ___________________________________ Date of Corrective Action: ____________________________

Corrective Action Plan Approval: ____ Date: ______________________________

DESCRIPTION OF FOLLOW-UP ACTIVITIES:

Person Responsible for Follow-up Activities: _________________________________ Date of Follow-up Activity: ____________________________

Final Corrective Action Approval: _______________________________________ Date: ______________________________

Figure B2-1. Corrective Action Record Form

CHAIN OF CUSTODY/LABORATORY ANALYSIS REQUEST FORM

Page ______ of ______ Turn Around Requested: ________

Report to:	Phone:				Analyses Requested										Notes/Comments
Company:	Fax:														
Address:	Proj Name:														
City:	Proj Number:														
State:	Sampler:														
Zipcode:															
Sample ID	Sample Date	Sample Time	Sample Matrix	No. Containers											

Relinquished: (Signature)	Received by: (Signature)	Special Instructions/Notes	
Printed name:	Printed name:		
Company:	Company:		Number of Coolers: Cooler Temp(s):
Date: Time:	Date: Time:		COC Seals Intact? Bottles Intact?

Figure B3-1. Chain of Custody/Laboratory Analysis Request Form

Table A4-1. Project Team Contact Information.

Name	Project Role	Phone	Fax	Email
EPA Region 10				
Chip Humphrey	Project Manager	503-326-2678	503-326-3399	humphrey.chip@epa.gov
Eric Blischke	Project Manager	503-326-4006	503-326-3399	blischke.eric@epamail.epa.gov
Tara Martich	Project Manager	206-553-0039	206-553-0124	martich.tara-ann@epa.gov
Dana Davoli	Human health risk assessment	206-553-2135	206 553-0119	davoli.dana@epa.gov
Joe Goulet	Ecological risk assessment	206-553-6692	206 553-0119	goulet.joe@epa.gov
Rene Fuentes	Hydrogeologist	206-553-1599	206-783-4126	fuentes.rene@epa.gov
Ginna Grepo-Grove	Quality Assurance Manager	206-553-1632	206-553-8210	Grepo-Grove.Gina@epamail.epa.gov
Lower Willamette Group				
Jim McKenna (Port of Portland)	Co-Chairman	503-944-7325	503-944-7353	mckenj@portptld.com
Bob Wyatt (Northwest Natural)	Co-Chairman	503-226-4211 x5425	503-273-4815	rjw@nwnatural.com
LWG Common Consultants				
Keith Pine (Integral)	CERCLA Coordinator	206-230-9600 x26	206-230-9601	kpine@integral-corp.com
Laura Kennedy (KJC)	Human Health Risk Assessment Coordinator	415-243-2405	415-896-0999	laurakennedy@kennedyjenks.com
Lisa Saban (Windward)	Ecological Risk Assessment Coordinator	206-577-1288	206-217-0089	lisas@windwardenv.com
Carl Stivers (Anchor)	Feasibility Study Coordinator	206-287-9130	206-287-9131	cstivers@anchorenv.com
Gene Revelas (Integral)	Sampling and Analysis Coordinator	360-705-3534	360-705-3669	grevelas@integral-corp.com
Ian Stupakoff (Integral)	Field Coordinator	360-705-3534	360-705-3669	stupakoff@integral-corp.com
Maja Tritt (Integral)	Chemistry QA Manager	206-230-9600 x21	206-230-9601	mtritt@integral-corp.com
Shawn Hinz (Windward)	Bioassay QA Manager	206-838-5873	206-217-0089	shawnh@windwardenv.com
Helle B. Andersen (Windward)	Bioassay Laboratory Coordinator	206-577-1287	206-217-0089	hellea@windwardenv.com
Tom Schulz (Integral)	Database Administrator	360-705-3534	360-705-3669	tschulz@integral-corp.com
Chemical Laboratories				
Abbie Spielman (CAS)	Laboratory Project Manager	360-577-7222	360-636-1068	aspielman@kelso.caslab.com
Lee Wolf (CAS)	Laboratory QA Manager	360-577-7222	360-636-1068	lwolf@kelso.caslab.com
Bob Wagner (NEA)	Laboratory Project Manager	518-346-4592 x21	518-381-6055	bobw@nealab.com
William Kotas (NEA)	Laboratory QA Officer	518-346-4592 x17	518-381-6055	williamk@nealab.com
Katie Downie (STL)	Laboratory Project Manager	253 922-2310	253 922-5047	kdownie@stl-inc.com
Terri Torres (STL)	Laboratory QA Manager	253 922-2310	253 922-5047	ttorres@stl-inc.com
Bioassay Laboratory				
Gerald Irissarri (NAS)	Laboratory Project Manager	541-265-7225		girissarri@nwaquatic.com
Linda Nemeth (NAS)	Laboratory QA Manager	541-265-7225		lnemeth@inetw.net

Table A6-1. Laboratory Methods for River and Beach Sediment Samples

Analytes	**Laboratory**	**Sample Preparation**		**Quantitative Analysis**	
		Protocol	**Procedure**	**Protocol**	**Procedure**
Conventional Analyses	CAS Kelso				
Total solids		NA	--	PSEP 1986	Balance
Grain size		NA	--	PSEP 1986	Sieves and pipette method
Total sulfides		EPA 9030	Distillation	EPA 9030	Colorimetry
Ammonia		Plumb 1981	KCl extraction	EPA 350.1	Colorimetry
Total organic carbon		Plumb 1981	Acid pretreatment	Plumb et al., 1981	Combustion; coulometric titration
Geotechnical characteristics	CAS Redding				
Specific gravity		--	--	ASTM D-854	Gravimeter
Atterberg limits		--	--	ASTM D-4318	Moisture determination
Metals	CAS Kelso				
Aluminum, antimony, arsenic, cadmium, chromium, copper, lead, nickel, selenium, silver, zinc		EPA 3050	Strong acid digestion	EPA 6020	ICP/MS
Arsenic[a]		EPA 3050 EPA 7062	Strong acid digestion Hydride generation	EPA 7062	AAS
Selenium[b]		EPA 3050 EPA 7742	Strong acid digestion Hydride generation	EPA 7742	AAS
Mercury		EPA 7471A	Acid digestion/oxidation	EPA 7471A	CVAA
Hexavalent chromium		EPA 3060A	Alkaline digestion	EPA 7196A	Colorimetry
Butyltins	CAS Kelso	Krone et al. 1989	Solvent extraction Derivatization	Krone et. al 1989	GC/FPD
Petroleum hydrocarbons	CAS Kelso				
Gasoline-range hydrocarbons		NWTPH-Gx	Methanol extraction Purge and trap	NWTPH-Gx	GC/FID
Diesel- and oil-range hydrocarbons		NWTPH-Dx	Sovent extraction Silica gel cleanup (as needed)	NWTPH-Dx	GC/FID
Chlorinated herbicides and pentachlorophenol	STL Tacoma	EPA 8151A	Solvent extraction Esterification	EPA 8151A	GC/MS

Table A6-1. Laboratory Methods for River and Beach Sediment Samples

Analytes	**Laboratory**	**Sample Preparation**		**Quantitative Analysis**	
		Protocol	**Procedure**	**Protocol**	**Procedure**
Organochlorine pesticides and selected SVOCs	NEA	EPA 3540 EPA 3620B EPA 3660B	Soxhlet extraction Florisil® cleanup Sulfur cleanup	EPA 8081A	GC/ECD
PCB Aroclors	NEA	EPA 3545 EPA 3665A EPA 3620B EPA 3660B	Pressurized fluid extraction Sulfuric acid cleanup Florisil® cleanup Sulfur cleanup	EPA 8082	GC/ECD
Volatile organic compounds	CAS Kelso	EPA 5035	Purge and trap	EPA 8260B	GC/MS
Semivolatile organic compounds	NEA	EPA 3545 EPA 3640A	Pressurized fluid extraction Gel permeation chromatography	EPA 8270C	GC/MS - ion trap
PCB congeners	CAS Houston	EPA 1668A	Soxhlet/Dean Stark extraction Sulfuric acid cleanup Silica column cleanup	EPA 1668A	HRGC/HRMS
Chlorinated dioxins and furans	CAS Houston	EPA 1613B	Soxhlet/Dean Stark extraction Sulfuric acid cleanup Silica/carbon column cleanup	EPA 1613B	HRGC/HRMS

[a] Arsenic will be analyzed by EPA method 7062 if it is not detected at the MRL by EPA method 6020.

[b] Selenium will be analyzed by EPA method 7742 if it is not detected at the MRL by EPA method 6020.

Table A6-2. Analytes, Analytical Concentration Goals, and Method Reporting Limits for Sediment Samples

Analyte	**CAS number**	**ACG[a]**	**MDL[b]**	**MRL[c]**
Conventional Analyses				
Total solids (percent of whole weight)	--	*	0.01	0.01
Grain size (percent)[d]	--	*	0.1	0.1
Total sulfides (mg/kg)	--	*	0.1	0.2
Ammonia (mg/kg)	7664-41-7	*	0.05	0.1
Total organic carbon (percent)	--	*	0.02	0.05
Geotechnical characteristics				
Specific gravity (g/cc)	--	*	--	0.01
Atterberg limits (percent moisture)	--	*	--	0.1
Metals			**mg/kg dry wt**	
Aluminum	7429-90-5	*	2.0	2.0
Antimony	7440-36-0	*	0.02	0.05
Arsenic	7440-38-2	*	0.05	0.1
Cadmium	7440-43-9	*	0.006	0.02
Chromium	7440-47-3	*	0.04	0.2
Copper	7440-50-8	*	0.07	0.1
Lead	7439-92-1	*	0.02	0.05
Mercury	7439-97-6	*	0.01	0.02
Nickel	7440-02-0	*	0.03	0.2
Selenium	7782-49-2	*	0.05	0.1
Silver	7440-22-4	*	0.02	0.02
Zinc	7440-66-6	*	0.1	0.5
Hexavalent chromium	18540-29-9	*	0.2	0.5
Petroleum hydrocarbons			**mg/kg dry wt**	
Gasoline-range petroleum hydrocarbons	--	*	3.2	10
Diesel-range petroleum hydrocarbons	--	*	7.1	25
Motor oil-range petroleum hydrocarbons	--	*	4.6	100
Butyltins			**µg/kg dry wt**	
Monobutyltin	78763-54-9	*	0.071	1
Dibutyltin	14488-53-0	*	0.041	1
Tributyltin	36643-28-4	**0.08**	0.16	1
Tetrabutyltin	1461-25-2	*	0.12	1
Chlorinated Herbicides and Pentachlorophenol			**µg/kg dry wt**	
Dalapon	75-99-0	*	tbd	5
Dicamba	1918-00-9	*	tbd	5
MCPA	94-74-6	*	tbd	5
Dichlorprop	120-36-5	*	tbd	5
2,4-D	94-75-7	**2.8**	tbd[e]	5
2,4,5-TP (Silvex)	93-72-1	**2.2**	tbd[e]	8
2,4,5-T	93-76-5	**2.8**	tbd[e]	8
2,4-DB	94-82-6	**2.2**	tbd[e]	5
Dinoseb	88-85-7	*	tbd	8
MCPP	93-65-2	*	tbd	5
Pentachlorophenol	87-86-5	**0.58**	tbd[e]	5
Organochlorine Pesticides and Selected SVOCs			**µg/kg dry wt**	
2,4'-DDD	53-19-0	*	tbd	0.2
2,4'-DDE	3424-82-6	*	tbd	0.2

Table A6-2. Analytes, Analytical Concentration Goals, and Method Reporting Limits for Sediment Samples

Analyte	**CAS number**	**ACG[a]**	**MDL[b]**	**MRL[c]**
2,4'-DDT	789-02-6	*	tbd	0.2
4,4'-DDD	72-54-8	**0.083**	tbd	0.2
4,4'-DDE	72-55-9	**0.0588**	tbd	0.2
4,4'-DDT	50-29-3	**0.0588**	tbd	0.2
Total DDT		*	tbd	
Aldrin	309-00-2	**0.00038**	tbd	0.2
a - BHC	319-84-6	**0.001**	tbd	0.2
b - BHC	319-85-7	**0.0036**	tbd	0.2
d - BHC	319-86-8	*	tbd	0.2
g - BHC (Lindane)	58-89-9	**0.005**	tbd	0.2
a - Chlordane	5103-71-9	*	tbd	0.2
g - Chlordane	5103-74-2	*	tbd	0.2
Oxychlordane	27304-13-8	*	tbd	0.2
cis - Nonachlor	5103-73-1	*	tbd	0.2
trans - Nonachlor	39765-80-5	*	tbd	0.2
Total chlordane[f]		**0.057**	tbd	
Dieldrin	60-57-1	**0.0004**	tbd	0.2
Endosulfan I	959-98-8	1.7	tbd	0.2
Endosulfan II	33213-65-9	*	tbd	0.2
Endosulfan sulfate	1031-07-8	*	tbd	0.2
Endrin	72-20-8	**0.084**	tbd	0.2
Endrin aldehyde	7421-93-4	*	tbd	0.2
Endrin ketone	53494-70-5	*	tbd	0.2
Heptachlor	76-44-8	**0.0014**	tbd	0.2
Heptachlor epoxide	1024-57-3	**0.0007**	tbd	0.2
Methoxychlor	72-43-5	1.4	tbd	0.2
Mirex	2385-85-5	**0.056**	tbd	0.2
Toxaphene	8001-35-2	**0.0059**	tbd	20
Hexachlorobenzene	118-74-1	0.33	tbd	tbd
Hexachlorobutadiene	87-68-3	0.6	tbd	tbd
Hexachloroethane	67-72-1	2.0	tbd	tbd
PCB Aroclors			**µg/kg dry wt**	
Aroclor 1016	12674-11-2	*	tbd	4
Aroclor 1221	11104-28-2	*	tbd	4
Aroclor 1232	11141-16-5	*	tbd	4
Aroclor 1242	53469-21-9	**0.004**	tbd	4
Aroclor 1248	12672-29-6	**0.004**	tbd	4
Aroclor 1254	11097-69-1	**0.004**	tbd	4
Aroclor 1260	11096-82-5	**0.004**	tbd	4
Aroclor 1262	37324-23-5	*	tbd	4
Aroclor 1268	11100-14-4	*	tbd	4
Volatile Organic Compounds			**µg/kg dry wt**	
1,1,1,2-Tetrachloroethane	630-20-6	*	tbd	1
1,1,1-Trichloroethane	71-55-6	*	tbd	1
1,1,2,2-Tetrachloroethane	79-34-5	*	tbd	1
1,1,2-Trichloroethane	79-00-5	*	tbd	1
1,1-Dichloroethane	75-34-3	*	tbd	1
1,2,3-Trichloropropane	96-18-4	*	tbd	1
1,2-Dichloroethane	107-06-2	*	tbd	1
1,2-Dichloropropane	78-87-5	*	tbd	1
1,4-Dichlorobenzene	106-46-7	2.0	tbd	1
2-Butanone	78-93-3	*	tbd	4
2-Chloroethyl Vinyl Ether	110-75-8	*	tbd	2
2-Hexanone	591-78-6	*	tbd	4

Table A6-2. Analytes, Analytical Concentration Goals, and Method Reporting Limits for Sediment Samples

Analyte	**CAS number**	**ACG**[a]	**MDL**[b]	**MRL**[c]
4-Methyl-2-Pentanone	108-10-1	*	tbd	4
Acetone	67-64-1	*	tbd	4
Acrolein	107-02-8	*	tbd	20
Acrylonitrile	107-13-1	*	tbd	4
Bromochloromethane	74-97-5	*	tbd	1
Bromodichloromethane	75-27-4	*	tbd	1
Bromoethane	74-96-4	*	tbd	NE
Bromoform	75-25-2	*	tbd	1
Bromomethane	74-83-9	*	tbd	1
Carbon Disulfide	75-15-0	*	tbd	1
Carbon Tetrachloride	56-23-5	*	tbd	1
Chlorobenzene	108-90-7	*	tbd	1
Chlorodibromomethane	124-48-1	*	tbd	1
Chloroethane	75-00-3	*	tbd	1
Chloroform	67-66-3	*	tbd	1
Chloromethane	74-87-3	*	tbd	1
cis - 1,3-Dichloropropene	10061-01-5	*	tbd	1
Dibromomethane	74-95-3	*	tbd	1
Dichlorodifluoromethane	75-71-8	*	tbd	1
Iodomethane	74-88-4	*	tbd	4
Isopropyl benzene	98-82-8	*	tbd	4
Methylene chloride	75-09-2	*	tbd	2
Naphthalene	91-20-3	23	tbd	4
Styrene	100-42-5	*	tbd	1
trans -1,4-Dichloro-2-butene	110-57-6	*	tbd	4
Trichlorofluoromethane	75-69-4	*	tbd	1
Vinyl Acetate	108-05-4	*	tbd	4
1,1-Dichloroethene	75-35-4	*	tbd	1
Benzene	71-43-2	*	tbd	1
Ethyl Benzene	100-41-4	*	tbd	1
m,p -Xylene	179601-23-1	*	tbd	1
Methyl-*t* -butyl ether (MTBE)	1634-04-4	*	tbd	1
o -Xylene	95-47-6	*	tbd	1
Tetrachloroethene	127-18-4	*	tbd	1
Toluene	108-88-3	*	tbd	1
trans -1,2-Dichloroethene	156-60-5	*	tbd	1
trans -1,3-Dichloropropene	10061-02-6	*	tbd	1
Trichloroethene	79-01-6	*	tbd	1
Vinyl Chloride	75-01-4	*	tbd	1
Semivolatile Organic Compounds			**µg/kg dry wt**	
Halogenated Compounds				
1,2-Dichlorobenzene	95-50-1	184	tbd	20
1,3-Dichlorobenzene	541-73-1	*	tbd	20
1,4-Dichlorobenzene[g]	106-46-7	**2.0**	tbd	20
1,2,4-Trichlorobenzene	120-82-1	*	tbd	20
Hexachlorobenzene[h]	118-74-1	**0.3**	tbd	100
2-Chloronaphthalene	91-58-7	*	tbd	20
Hexachloroethane[h]	67-72-1	**2.0**	tbd	5
Hexachlorobutadiene[h]	87-68-3	**0.6**	tbd	100
Hexachlorocyclopentadiene	77-47-4	*	tbd	100
2,2'-oxybis(1-chloropropane)	108-60-1	*	tbd	20
Bis-(2-chloroethoxy) methane	111-91-1	*	tbd	20
Bis-(2-chloroethyl) ether	111-44-4	*	tbd	40
4-Chlorophenyl-phenyl ether	7005-72-3	*	tbd	20
4-bromophenyl-phenyl ether	101-55-3	*	tbd	20
3,3’-Dichlorbenzidine	91-94-1	*	tbd	100

Table A6-2. Analytes, Analytical Concentration Goals, and Method Reporting Limits for Sediment Samples

Analyte	CAS number	ACG[a]	MDL[b]	MRL[c]
4-Chloroaniline	106-47-8	*	tbd	tbd
Organonitrogen Compounds				
Nitrobenzene	98-95-3	*	tbd	20
Aniline	62-53-3	*	tbd	20
2-Nitroaniline	88-74-4	*	tbd	20
3-Nitroaniline	99-09-2	*	tbd	120
4-Nitroaniline	100-01-6	*	tbd	60
n-Nitrosodimethylamine	62-75-9	**0.0073**	tbd	100
n-Nitroso-di-n-propylamine	621-64-7	**0.053**	tbd	20
n-Nitrosodiphenylamine	86-30-6	*	tbd	20
1,2-Diphenylhydrazine	122-66-7	**0.0025**	tbd	tbd
2,4-Dinitrotoluene	121-14-2	*	tbd	100
2,6-Dinitrotoluene	606-20-2	*	tbd	100
Carbazole	86-74-8	6.12	tbd	5
Oxygen-Containing Compounds				
Benzoic Acid	65-85-0	*	tbd	200
Benzyl Alcohol	100-51-6	*	tbd	20
Dibenzofuran	132-64-9	8.2	tbd	5
Isophorone	78-59-1	*	tbd	20
Phenols and Substituted Phenols				
Phenol	108-95-2	3146	tbd	20
2-Methylphenol	95-48-7	*	tbd	20
4-Methylphenol	106-44-5	26	tbd	20
2,4-Dimethylphenol	105-67-9	*	tbd	20
2-Chlorophenol	95-57-8	26	tbd	20
2,4-Dichlorophenol	120-83-2	**16**	tbd	60
2,4,5-Trichlorophenol	95-95-4	524	tbd	100
2,4,6-Trichlorophenol	88-06-2	**1.8**	tbd	100
2,3,4,6-Tetrachlorophenol	58-90-2	157	tbd	100
2,3,4,5- and 2,3,5,6-Tetrachlorophenol	4901-51-3; 935-95-5	157	tbd	100
Pentachlorophenol[i]	87-86-5	**0.58**	tbd	34
4-Chloro-3-methylphenol	59-50-7	*	tbd	40
2-Nitrophenol	88-75-5	*	tbd	100
4-Nitrophenol	100-02-7	*	tbd	100
2,4-Dinitrophenol	51-28-5	*	tbd	200
4,6-Dinitro-2-methylphenol	534-52-1	*	tbd	200
Phthalate Esters				
Dimethylphthalate	131-11-3	20000	tbd	20
Diethylphthlalate	84-66-2	*	tbd	20
Di-n-butylphthalate	84-74-2	204	tbd	20
Butylbenzylphthalate	85-68-7	400	tbd	20
Di-n-octylphthalate	117-84-0	40.9	tbd	20
bis(2-Ethylhexyl)phthalate	117-81-7	**3.4**	tbd	20
Polycyclic Aromatic Hydrocarbons				
Naphthalene	91-20-3	24	tbd	20
2-Methylnaphthalene	91-57-6	*	tbd	20
Acenaphthylene	208-96-8	*	tbd	20
Acenaphthene	83-32-9	72	tbd	20
Fluorene	86-73-7	48	tbd	20
Phenanthrene	85-01-8	*	tbd	20
Anthracene	120-12-7	360	tbd	20
Fluoranthene	206-44-0	48	tbd	20
Pyrene	129-00-0	36	tbd	20
Benzo(a)anthracene	56-55-3	**0.038**	tbd	5
Chrysene	218-01-9	**3.8**	tbd	5
Benzo(b)fluoranthene	205-99-2	**0.038**	tbd	5
Benzo(k)fluoranthene	207-08-9	**0.38**	tbd	5

Table A6-2. Analytes, Analytical Concentration Goals, and Method Reporting Limits for Sediment Samples

Analyte	**CAS number**	**ACG**[a]	**MDL**[b]	**MRL**[c]
Benzo(a)pyrene	50-32-8	**0.0038**	tbd	5
Indeno(1,2,3-cd)pyrene	193-39-5	**0.038**	tbd	5
Dibenz(a,h)anthracene	53-70-3	**0.0038**	tbd	5
Benzo(g,h,i)perylene	191-24-2	*	tbd	5
PCB congeners			**pg/g dry wt**[j,k]	
2-MoCB	PCB-1	tbd	tbd	tbd
3-MoCB	PCB-2	tbd	tbd	tbd
4-MoCB	PCB-3	tbd	tbd	tbd
2,2'-DiCB	PCB-4	tbd	tbd	tbd
2,3-DiCB	PCB-5	tbd	tbd	tbd
2,3'-DiCB	PCB-6	tbd	tbd	tbd
2,4-DiCB	PCB-7	tbd	tbd	tbd
2,4'-DiCB	PCB-8	tbd	tbd	tbd
2,5-DiCB	PCB-9	tbd	tbd	tbd
2,6-DiCB	PCB-10	tbd	tbd	tbd
3,3'-DiCB	PCB-11	tbd	tbd	tbd
3,4-DiCB	PCB-12	tbd	tbd	tbd
3,4'-DiCB	PCB-13	tbd	tbd	tbd
3,5-DiCB	PCB-14	tbd	tbd	tbd
4,4'-DiCB	PCB-15	tbd	tbd	tbd
2,2',3-TrCB	PCB-16	tbd	tbd	tbd
2,2',4-TrCB	PCB-17	tbd	tbd	tbd
2,2',5-TrCB	PCB-18	tbd	tbd	tbd
2,2',6-TrCB	PCB-19	tbd	tbd	tbd
2,3,3'-TrCB	PCB-20	tbd	tbd	tbd
2,3,4-TrCB	PCB-21	tbd	tbd	tbd
2,3,4'-TrCB	PCB-22	tbd	tbd	tbd
2,3,5-TrCB	PCB-23	tbd	tbd	tbd
2,3,6-TrCB	PCB-24	tbd	tbd	tbd
2,3',4-TrCB	PCB-25	tbd	tbd	tbd
2,3',5-TrCB	PCB-26	tbd	tbd	tbd
2,3',6-TrCB	PCB-27	tbd	tbd	tbd
2,4,4'-TrCB	PCB-28	tbd	tbd	tbd
2,4,5-TrCB	PCB-29	tbd	tbd	tbd
2,4,6-TrCB	PCB-30	tbd	tbd	tbd
2,4',5-TrCB	PCB-31	tbd	tbd	tbd
2,4',6-TrCB	PCB-32	tbd	tbd	tbd
2',3,4-TrCB	PCB-33	tbd	tbd	tbd
2',3,5-TrCB	PCB-34	tbd	tbd	tbd
3,3',4-TrCB	PCB-35	tbd	tbd	tbd
3,3',5-TrCB	PCB-36	tbd	tbd	tbd
3,4,4'-TrCB	PCB-37	tbd	tbd	tbd
3,4,5-TrCB	PCB-38	tbd	tbd	tbd
3,4',5-TrCB	PCB-39	tbd	tbd	tbd
2,2',3,3'-TeCB	PCB-40	tbd	tbd	tbd
2,2',3,4-TeCB	PCB-41	tbd	tbd	tbd
2,2',3,4'-TeCB	PCB-42	tbd	tbd	tbd
2,2',3,5-TeCB	PCB-43	tbd	tbd	tbd
2,2',3,5'-TeCB	PCB-44	tbd	tbd	tbd
2,2',3,6-TeCB	PCB-45	tbd	tbd	tbd
2,2',3,6'-TeCB	PCB-46	tbd	tbd	tbd
2,2',3,4'-TeCB	PCB-47	tbd	tbd	tbd
2,2',4,5-TeCB	PCB-48	tbd	tbd	tbd
2,2',4,5'-TeCB	PCB-49	tbd	tbd	tbd
2,2',4,6-TeCB	PCB-50	tbd	tbd	tbd
2,2',4,6'-TeCB	PCB-51	tbd	tbd	tbd
2,2',5,5'-TeCB	PCB-52	tbd	tbd	tbd

Table A6-2. Analytes, Analytical Concentration Goals, and Method Reporting Limits for Sediment Samples

Analyte	CAS number	ACG[a]	MDL[b]	MRL[c]
2,2',5,6'-TeCB	PCB-53	tbd	tbd	tbd
2,2',6,6'-TeCB	PCB-54	tbd	tbd	tbd
2,3,3',4'-TeCB	PCB-55	tbd	tbd	tbd
2,3,3',4'-TeCB	PCB-56	tbd	tbd	tbd
2,3,3',5-TeCB	PCB-57	tbd	tbd	tbd
2,3,3',5'-TeCB	PCB-58	tbd	tbd	tbd
2,3,3',6-TeCB	PCB-59	tbd	tbd	tbd
2,3,4,4'-TeCB	PCB-60	tbd	tbd	tbd
2,3,4,5-TeCB	PCB-61	tbd	tbd	tbd
2,3,4,6-TeCB	PCB-62	tbd	tbd	tbd
2,3,4',5-TeCB	PCB-63	tbd	tbd	tbd
2,3,4',6-TeCB	PCB-64	tbd	tbd	tbd
2,3,5,6-TeCB	PCB-65	tbd	tbd	tbd
2,3',4,4'-TeCB	PCB-66	tbd	tbd	tbd
2,3',4,5-TeCB	PCB-67	tbd	tbd	tbd
2,3',4,5'-TeCB	PCB-68	tbd	tbd	tbd
2,3',4,6-TeCB	PCB-69	tbd	tbd	tbd
2,3',4',5-TeCB	PCB-70	tbd	tbd	tbd
2,3',4',6-TeCB	PCB-71	tbd	tbd	tbd
2,3',5,5'-TeCB	PCB-72	tbd	tbd	tbd
2,3',5',6-TeCB	PCB-73	tbd	tbd	tbd
2,4,4',5-TeCB	PCB-74	tbd	tbd	tbd
2,4,4',6-TeCB	PCB-75	tbd	tbd	tbd
2',3,4',5-TeCB	PCB-76	tbd	tbd	tbd
3,3',4,4'-TeCB	PCB-77	tbd	tbd	tbd
3,3',4,5-TeCB	PCB-78	tbd	tbd	tbd
3,3',4,5'-TeCB	PCB-79	tbd	tbd	tbd
3,3',5,5'-TeCB	PCB-80	tbd	tbd	tbd
3,4,4',5-TeCB	PCB-81	tbd	tbd	tbd
2,2',3,3',4-PeCB	PCB-82	tbd	tbd	tbd
2,2',3,3',5-PeCB	PCB-83	tbd	tbd	tbd
2,2',3,3',6-PeCB	PCB-84	tbd	tbd	tbd
2,2',3,4,4'-PeCB	PCB-85	tbd	tbd	tbd
2,2',3,4,5-PeCB	PCB-86	tbd	tbd	tbd
2,2',3,4,5'-PeCB	PCB-87	tbd	tbd	tbd
2,2',3,4,6-PeCB	PCB-88	tbd	tbd	tbd
2,2',3,4,6'-PeCB	PCB-89	tbd	tbd	tbd
2,2',3,4',5-PeCB	PCB-90	tbd	tbd	tbd
2,2',3,4',6-PeCB	PCB-91	tbd	tbd	tbd
2,2',3,5,5'-PeCB	PCB-92	tbd	tbd	tbd
2,2',3,5,6-PeCB	PCB-93	tbd	tbd	tbd
2,2',3,5,6'-PeCB	PCB-94	tbd	tbd	tbd
2,2',3,5',6-PeCB	PCB-95	tbd	tbd	tbd
2,2',3,6,6'-PeCB	PCB-96	tbd	tbd	tbd
2,2',3',4,5-PeCB	PCB-97	tbd	tbd	tbd
2,2',3',4,6-PeCB	PCB-98	tbd	tbd	tbd
2,2',4,4',5-PeCB	PCB-99	tbd	tbd	tbd
2,2',4,4',6-PeCB	PCB-100	tbd	tbd	tbd
2,2',4,5,5'-PeCB	PCB-101	tbd	tbd	tbd
2,2',4,5,6'-PeCB	PCB-102	tbd	tbd	tbd
2,2',4,5,6'-PeCB	PCB-103	tbd	tbd	tbd
2,2',4,6,6'-PeCB	PCB-104	tbd	tbd	tbd
2,3,3'4,4'-PeCB	PCB-105	tbd	tbd	tbd
2,3,3',4,5-PeCB	PCB-106	tbd	tbd	tbd
2,3,3',4',5-PeCB	PCB-107	tbd	tbd	tbd
2,3,3',4,5'-PeCB	PCB-108	tbd	tbd	tbd
2,3,3',4,6-PeCB	PCB-109	tbd	tbd	tbd
2,3,3',4',6-PeCB	PCB-110	tbd	tbd	tbd

Table A6-2. Analytes, Analytical Concentration Goals, and Method Reporting Limits for Sediment Samples

Analyte	CAS number	ACG[a]	MDL[b]	MRL[c]
2,3,3',5,5'-PeCB	PCB-111	tbd	tbd	tbd
2,3,3',5,6-PeCB	PCB-112	tbd	tbd	tbd
2,3,3',5',6-PeCB	PCB-113	tbd	tbd	tbd
2,3,4,4',5-PeCB	PCB-114	tbd	tbd	tbd
2,3,4,4',6-PeCB	PCB-115	tbd	tbd	tbd
2,3,4,5,6-PeCB	PCB-116	tbd	tbd	tbd
2,3,4',5,6-PeCB	PCB-117	tbd	tbd	tbd
2,3',4,4',5-PeCB	PCB-118	tbd	tbd	tbd
2,3',4,4',6-PeCB	PCB-119	tbd	tbd	tbd
2,3',4,5,5'-PeCB	PCB-120	tbd	tbd	tbd
2,3',4,5,6-PeCB	PCB-121	tbd	tbd	tbd
2',3,3',4,5-PeCB	PCB-122	tbd	tbd	tbd
2',3,4,4',5-PeCB	PCB-123	tbd	tbd	tbd
2',3,4,5,5'-PeCB	PCB-124	tbd	tbd	tbd
2',3,4,5,6'-PeCB	PCB-125	tbd	tbd	tbd
3,3',4,4',5-PeCB	PCB-126	tbd	tbd	tbd
3,3',4,5,5'-PeCB	PCB-127	tbd	tbd	tbd
2,2',3,3',4,4'-HxCB	PCB-128	tbd	tbd	tbd
2,2',3,3',4,5-HxCB	PCB-129	tbd	tbd	tbd
2,2',3,3',4,5'-HxCB	PCB-130	tbd	tbd	tbd
2,2',3,3',4,6-HxCB	PCB-131	tbd	tbd	tbd
2,2',3,3',4,6'-HxCB	PCB-132	tbd	tbd	tbd
2,2',3,3',5,5'-HxCB	PCB-133	tbd	tbd	tbd
2,2',3,3',5,6-HxCB	PCB-134	tbd	tbd	tbd
2,2',3,3',5,6'-HxCB	PCB-135	tbd	tbd	tbd
2,2',3,3',6,6'-HxCB	PCB-136	tbd	tbd	tbd
2,2',3,4,4',5-HxCB	PCB-137	tbd	tbd	tbd
2,2',3,4,4',5'-HxCB	PCB-138	tbd	tbd	tbd
2,2',3,4,4',6-HxCB	PCB-139	tbd	tbd	tbd
2,2',3,4,4',6'-HxCB	PCB-140	tbd	tbd	tbd
2,2',3,4,5,5'-HxCB	PCB-141	tbd	tbd	tbd
2,2',3,4,5,6-HxCB	PCB-142	tbd	tbd	tbd
2,2',3,4,5,6'-HxCB	PCB-143	tbd	tbd	tbd
2,2',3,4,5',6-HxCB	PCB-144	tbd	tbd	tbd
2,2',3,4,6,6'-HxCB	PCB-145	tbd	tbd	tbd
2,2',3,4',5,5'-HxCB	PCB-146	tbd	tbd	tbd
2,2',3,4',5,6-HxCB	PCB-147	tbd	tbd	tbd
2,2',3,4',5,6'-HxCB	PCB-148	tbd	tbd	tbd
2,2',3,4',5',6-HxCB	PCB-149	tbd	tbd	tbd
2,2',3,4',6,6'-HxCB	PCB-150	tbd	tbd	tbd
2,2',3,5,5',6-HxCB	PCB-151	tbd	tbd	tbd
2,2',3,5,6,6'-HxCB	PCB-152	tbd	tbd	tbd
2,2',4,4',5,5'-HxCB	PCB-153	tbd	tbd	tbd
2,2',4,4',5',6-HxCB	PCB-154	tbd	tbd	tbd
2,2',4,4',6,6'-HxCB	PCB-155	tbd	tbd	tbd
2,3,3',4,4',5-HxCB	PCB-156	tbd	tbd	tbd
2,3,3',4,4',5'-HxCB	PCB-157	tbd	tbd	tbd
2,3,3',4,4',6-HxCB	PCB-158	tbd	tbd	tbd
2,3,3',4,5,5'-HxCB	PCB-159	tbd	tbd	tbd
2,3,3',4,5,6-HxCB	PCB-160	tbd	tbd	tbd
2,3,3',4,5',6-HxCB	PCB-161	tbd	tbd	tbd
2,3,3',4',5,5'-HxCB	PCB-162	tbd	tbd	tbd
2,3,3',4',5,6-HxCB	PCB-163	tbd	tbd	tbd
2,3,3',4',5',6-HxCB	PCB-164	tbd	tbd	tbd
2,3,3',5,5',6-HxCB	PCB-165	tbd	tbd	tbd
2,3,4,4',5,6-HxCB	PCB-166	tbd	tbd	tbd
2,3,4,4',5,5'-HxCB	PCB-167	tbd	tbd	tbd
2,3',4,4',5',6-HxCB	PCB-168	tbd	tbd	tbd

Table A6-2. Analytes, Analytical Concentration Goals, and Method Reporting Limits for Sediment Samples

Analyte	**CAS number**	**ACG**[a]	**MDL**[b]	**MRL**[c]
3,3',4,4',5,5'-HxCB	PCB-169	tbd	tbd	tbd
2,2',3,3',4,4',5-HpCB	PCB-170	tbd	tbd	tbd
2,2',3,3',4,4',6-HpCB	PCB-171	tbd	tbd	tbd
2,2',3,3',4,5,5'-HpCB	PCB-172	tbd	tbd	tbd
2,2',3,3',4,5,6-HpCB	PCB-173	tbd	tbd	tbd
2,2',3,3',4,5,6'-HpCB	PCB-174	tbd	tbd	tbd
2,2',3,3',4,5',6-HpCB	PCB-175	tbd	tbd	tbd
2,2',3,3',4,6,6'-HpCB	PCB-176	tbd	tbd	tbd
2,2',3,3',4',5,6-HpCB	PCB-177	tbd	tbd	tbd
2,2',3,3',5,5',6-HpCB	PCB-178	tbd	tbd	tbd
2,2',3,3',5,6,6'-HpCB	PCB-179	tbd	tbd	tbd
2,2',3,4,4',5,5'-HpCB	PCB-180	tbd	tbd	tbd
2,2',3,4,4',5,6-HpCB	PCB-181	tbd	tbd	tbd
2,2',3,4,4',5,6'-HpCB	PCB-182	tbd	tbd	tbd
2,2',3,4,4',5',6-HpCB	PCB-183	tbd	tbd	tbd
2,2',3,4,4',6,6'-HpCB	PCB-184	tbd	tbd	tbd
2,2',3,4,5,5',6-HpCB	PCB-185	tbd	tbd	tbd
2,2',3,4,5,6,6'-HpCB	PCB-186	tbd	tbd	tbd
2,2',3,4,5,5',6-HpCB	PCB-187	tbd	tbd	tbd
2,2',3,4',5,6,6'-HpCB	PCB-188	tbd	tbd	tbd
2,3,3',4,4',5,5'-HpCB	PCB-189	tbd	tbd	tbd
2,3,3',4,4',5,6-HpCB	PCB-190	tbd	tbd	tbd
2,3,3',4,4',5',6-HpCB	PCB-191	tbd	tbd	tbd
2,3,3',4,5,5',6-HpCB	PCB-192	tbd	tbd	tbd
2,3,3',4',5,5',6-HpCB	PCB-193	tbd	tbd	tbd
2,2',3,3',4,4',5,5'-OcCB	PCB-194	tbd	tbd	tbd
2,2',3,3',4,4',5,6-OcCB	PCB-195	tbd	tbd	tbd
2,2',3,3',4,4',5,6'-OcCB	PCB-196	tbd	tbd	tbd
2,2',3,3',4,4',6,6'-OcCB	PCB-197	tbd	tbd	tbd
2,2',3,3',4,5,5',6-OcCB	PCB-198	tbd	tbd	tbd
2,2',3,3',4,5,5',6'-OcCB	PCB-199	tbd	tbd	tbd
2,2',3,3',4,5,6,6'-OcCB	PCB-200	tbd	tbd	tbd
2,2',3,3',4,5',6,6'-OcCB	PCB-201	tbd	tbd	tbd
2,2',3,3',5,5',6,6'-OcCB	PCB-202	tbd	tbd	tbd
2,2',3,4,4',5,5',6-OcCB	PCB-203	tbd	tbd	tbd
2,2',3,4,4',5,6,6'-OcCB	PCB-204	tbd	tbd	tbd
2,3,3',4,4',5,5',6-OcCB	PCB-205	tbd	tbd	tbd
2,2',3,3',4,4',5,5',6-NoCB	PCB-206	tbd	tbd	tbd
2,2',3,3',4,4',5,6,6'-NoCB	PCB-207	tbd	tbd	tbd
2,2',3,3',4,5,5',6,6'-NoCB	PCB-208	tbd	tbd	tbd
DeCB	PCB-209	tbd	tbd	tbd
Chlorinated Dioxins and Furans[j]			**pg/g dry wt**	
2,3,7,8-TCDD	1746-01-6	**0.0001**	0.026	0.2
2,3,7,8-TCDF	51207-31-9	**0.001**	0.020	0.2
1,2,3,7,8-PeCDD	40321-76-4	**0.001**	0.029	0.5
1,2,3,7,8-PeCDF	57117-41-6	**0.001**	0.017	0.5
2,3,4,7,8-PeCDF	57117-31-4	**0.0002**	0.017	0.5
1,2,3,4,7,8-HxCDD	39227-28-6	**0.01**	0.030	0.5
1,2,3,6,7,8-HxCDD	57653-85-7	**0.01**	0.034	0.5
1,2,3,7,8,9-HxCDD	19408-74-3	**0.01**	0.032	0.5
1,2,3,4,7,8-HxCDF	70648-26-9	**0.01**	0.013	0.5
1,2,3,6,7,8-HxCDF	57117-44-9	**0.01**	0.013	0.5
1,2,3,7,8,9-HxCDF	72918-21-9	**0.01**	0.017	0.5
2,3,4,6,7,8-HxCDF	60851-34-5	**0.01**	0.013	0.5
1,2,3,4,6,7,8-HpCDD	35822-46-9	0.09	0.035	0.5
1,2,3,4,6,7,8-HpCDF	67562-39-4	0.09	0.033	0.5
1,2,3,4,7,8,9-HpCDF	55673-89-7	0.09	0.052	0.5

Table A6-2. Analytes, Analytical Concentration Goals, and Method Reporting Limits for Sediment Samples

Analyte	**CAS number**	**ACG**[a]	**MDL**[b]	**MRL**[c]
OCDD	3268-87-9	9.4	0.061	1.0
OCDF	39001-02-0	9.4	0.065	1.0
Total tetrachlorinated dioxins	41903-57-5	*	--	--
Total pentachlorinated dioxins	36088-22-9	*	--	--
Total hexachlorinated dioxins	34465-46-8	*	--	--
Total heptachlorinated dioxins	37871-00-4	*	--	--
Total tetrachlorinated furans	30402-14-3	*	--	--
Total pentachlorinated furans	30402-15-4	*	--	--
Total hexachlorinated furans	55684-94-1	*	--	--
Total heptachlorinated furans	38998-75-3	*	--	--

[a] Values are provided in bold font when the MRL is not expected to meet the ACG. ACGs for PCB congeners to be determined.

[b] The laboratory's current MDL is provided when an MDL study has been completed for the proposed method.
When no MDL is provided, the laboratory will complete an MDL study prior to analysis of samples for this project.

[c] The MRL is provided on a dry-weight basis and assumes 50% moisture in the samples.
The MRL for project samples will vary with moisture content in the samples.
The MRL represents the level of lowest calibration standard (i.e., the practical quantitation limit).

[d] Grain-size intervals will include the following:

- Gravel
- Very coarse sand
- Coarse sand
- Medium sand
- Fine sand
- Very fine sand
- Coarse silt
- Medium silt
- Fine silt
- Very fine silt
- Clay, phi size 8-9
- Clay, phi size 9-10
- Clay, phi size >10

[e] The MDLs for the herbicides and pentachlorophenol are expected to be lower than the ACGs.

[f] Total chlordane will be calculated as the sum of the 5 components listed above this entry.

[g] 1,4-Dichlorobenzene will also be analyzed by purge-and-trap GC/MS with the VOCs to improve MRLs.

[h] Hexachlorobenzene, hexachloroethane, and hexachlorobutadiene will also be analyzed by GC/ECD with the pesticides to improve MRLs.

[i] Pentachlorophenol will also be analyzed with the herbicides to improve the MRL.

[j] Expected MDLs are shown. MDLs for PCB congeners and dioxins and furans are sample-dependent and will vary from the indicated values.

[k] MDLs and MRLs are shown for a sample weight of 10 g. MDLs and MRLs will be lower for a larger sample size.

ACG = Analytical concentration goals; established by EPA during *ad hoc* meeting with LWG May 10, 2002
MDL = Method detection limit
MRL = Method reporting limit
NA = Not applicable
tbd = To be determined
* = A risk-based ACG has not been established

Table A7-1 Laboratory Control Limits for Surrogate Samples

Analysis	Percent Recovery
Butyltins	
Tri-n-proplytin	10-132
Petroleum Hydrocarbons	
4-Bromofluorobenzene	50-150
n-Triacontane	50-150
o-Terphenyl	50-150
Chlorinated Herbicides and Pentachlorophenol	
2,4-Dichlorophenylacetic acid	51-129
Organochlorine Pesticides and Selected SVOCs	
Tetra-chloro-meta-xylene	60-140
Decachlorobiphenyl	60-140
PCB Aroclors	
Tetra-chloro-meta-xylene	60-140
Decachlorobiphenyl	60-140
Volatile organic compounds	
1,2-Dichloroethane-d4	80-118
4-Bromofluorobenzene	74-116
Dibromofluoromethane	81-120
Toluene-d8	77-131
Semivolatile Organic Compounds	
2-Fluorophenol	25-121
Phenol-d6	24-113
Tetra-Chloro-meta-Xylene	60-140
Decachlorobiphenyl	60-140
Tetra-Chloro-meta-Xylene	60-140
Decachlorobiphenyl	60-140
2-Fluorophenol	25-121
Phenol-d6	24-113
2,4,6-Tribromophenol	19-122
Nitrobenzene-d5	23-120
2-Fluorobiphenyl	30-115
Terphenyl-d14	18-137
PCB Congeners	
13C-2-MoCB	15-140
13C-4-MoCB	15-140
13C-2,2'-DiCB	25-150
13C-4,4'-DiCB	25-150
13C-2,2',6-TrCB	25-150
13C-3,4,4'-TrCB	25-150
13C-2,2',6,6'-TeCB	25-150
13C-3,3',4,4'-TeCB	25-150
13C-3,4,4',5-TeCB	25-150
13C-2,2'4,6,6'-PeCB	25-150

Table A7-1 Laboratory Control Limits for Surrogate Samples

Analysis	Percent Recovery
13C-2,3,3',4,4'-PeCB	25-150
13C-2,3,4,4',5-PeCB	25-150
13C-2,3',4,4',5-PeCB	25-150
13C-2',3,4,4',5-PeCB	25-150
13C-3,3'4,4',5-PeCB	25-150
13C-2,2',4,4',6,6'-HxCB	25-150
13C-2,3,3'4,4',5-HxCB	25-150
13C-2,3,3'4,4',5'-HxCB	25-150
13C-2,3',4,4'5,5'-HxCB	25-150
13C-3,3'4,4'5,5'-HxCB	25-150
13C-2,2',3,4',5,6,6'-HpCB	25-150
13C-2',3,3'4,4',5,5'-HpCB	25-150
13C-2,2',3,3',5,5',6,6'-OcCB	25-150
13C-2,3,3',4,4',5,5',6-OcCB	25-150
13C-2,2',3,3',4,4',5,5',6-NoCB	25-150
13C-2,2',3,3',4,5,5',6,6'-NoCB	25-150
13C-2,2',3,3',4,4',5,5',6,6'-DeCB	25-150
Chlorinated Dioxins and Furans	
13C-2378-TCDD	25-164
13C-12378-PeCDD	25-181
13C-123478-HxCDD	32-141
13C-123678-HxCDD	28-130
13C-1234678-HpCDD	23-140
13C-OCDD	17-157
13C-2378-TCDF	24-169
13C-12378-PeCDF	24-185
13C-23478-PeCDF	21-178
13C-123478-HxCDF	26-152
13C-123678-HxCDF	26-123
13C-234678-HxCDF	28-136
13C-1234678-HpCDF	28-143
13C-1234789-HpCDF	26-138

Note:

Control limits are updated periodically by the laboratories. Control limits that are in effect at the laboratory at the time of analysis will be used for sample analysis and data validation. These may differ slightly from the control limits shown in this table.

Table A7-2. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples

Analysis	Matrix Spike Recovery (percent)	Laboratory Control Sample Recovery (percent)	Type of Duplicate	Control Limit Relative Percent Difference
Conventional Analyses				
Total solids	NA	NA	LD	20
Grain size	NA	NA	Triplicate	Note-1
Total sulfides	60-130	NA	LD	20
Ammonia	85-115	NA	LD	20
Total organic carbon	85-115	NA	LD	20
Geotechnical Characteristics				
Specific gravity	NA	NA	LD	20
Atterberg limits	NA	NA	LD	20
Metals				
Aluminum	70-130	Note-2	LD	30
Antimony	70-130	Note-2	LD	30
Arsenic	77-116	Note-2	LD	30
Cadmium	70-130	Note-2	LD	30
Chromium	70-130	Note-2	LD	30
Copper	70-130	Note-2	LD	30
Lead	70-130	Note-2	LD	30
Mercury	84-114	Note-2	LD	30
Nickel	70-130	Note-2	LD	30
Selenium	78-114	Note-2	LD	30
Silver	70-130	Note-2	LD	30
Zinc	70-130	Note-2	LD	30
Hexavalent chromium	85-115	Note-2	LD	20
Butyltins				
Monobutyltin	10-99	10-137	MSD	50
Dibutyltin	10-153	10-150	MSD	50
Tributyltin	10-152	10-144	MSD	50
Tetrabutyltin	10-153	10-128	MSD	50
Petroleum Hydrocarbons				
Gasoline Range Organics (GRO)	42-125	78-128	LD	40
Diesel Range Organics (DRO)	66-146	74-150	LD	40
Residual Range Organics (RRO)	70-130	53-143	LD	40
Chlorinated Herbicides and Pentachlorophenol				
2,4-Dichlorophenoxyacetic acid	46-136	46-136	LCSD	30
Dalapon	16-74	16-74	LCSD	30
Dicamba	48-123	48-123	LCSD	30
Dinoseb	18-157	18-157	LCSD	30
MCPP	53-154	53-154	LCSD	30
Pentachlorophenol	50-150	50-150	LCSD	30
Silvex (2,4,5-TP)	52-137	52-137	LCSD	30
Organochlorine Pesticides and Selected SVOCs				
All target analytes	60-140	70-130	MSD	20
PCB Aroclors				
All target analytes	60-140	70-130	MSD	20
Volatile organic compounds				
1,1,1,2-Tetrachloroethane	17-138	76-122	MSD	40
1,1,1-Trichloroethane	41-127	68-126	MSD	40
1,1,2,2-Tetrachloroethane	15-132	69-120	MSD	40
1,1,2-Trichloroethane	31-135	77-118	MSD	40
1,1-Dichloroethane	45-122	66-120	MSD	40
1,1-Dichloroethene	46-128	71-127	MSD	40
1,2,3-Trichloropropane	20-144	73-120	MSD	40
1,2-Dichloroethane	34-134	63-129	MSD	40

Table A7-2. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples

Analysis	Matrix Spike Recovery (percent)	Laboratory Control Sample Recovery (percent)	Type of Duplicate	Control Limit Relative Percent Difference
1,2-Dichloropropane	36-129	69-123	MSD	40
2-Butanone	10-143	37-137	MSD	40
2-Chloroethyl Vinyl Ether	70-130	34-149	MSD	40
2-Hexanone	22-126	60-124	MSD	40
4-Methyl-2-Pentanone	29-137	55-136	MSD	40
Acetone	20-127	43-119	MSD	40
Acrolein	70-130	18-148	MSD	40
Acrylonitrile	70-130	10-197	MSD	40
Benzene	45-129	78-124	MSD	40
Bromochloromethane	10-143	75-127	MSD	40
Bromodichloromethane	41-134	75-126	MSD	40
Bromoethane	19-138	67-129	MSD	40
Bromoform	10-136	72-121	MSD	40
Bromomethane	15-140	37-145	MSD	40
Carbon Disulfide	19-139	63-139	MSD	40
Carbon Tetrachloride	25-131	69-128	MSD	40
Chlorobenzene	17-130	76-119	MSD	40
Chlorodibromomethane	10-137	74-119	MSD	40
Chloroethane	28-149	44-143	MSD	40
Chloroform	37-132	70-123	MSD	40
Chloromethane	36-148	51-147	MSD	40
cis - 1,3-Dichloropropene	11-135	73-127	MSD	40
Dibromomethane	34-137	74-124	MSD	40
Dichlorodifluoromethane	34-154	43-163	MSD	40
Ethyl Benzene	15-138	75-126	MSD	40
Hexachloro-1,3-Butadiene	10-117	58-132	MSD	40
Iodomethane	70-130	70-130	MSD	40
Isopropyl Benzene	10-129	62-125	MSD	40
m,p-Xylene	15-138	76-131	MSD	40
Methylene Chloride	41-131	70-127	MSD	40
Methyl-t-butyl ether (MTBE)	37-132	59-124	MSD	40
Naphthalene	10-131	69-134	MSD	40
o-Xylene	12-143	76-128	MSD	40
Styrene	10-138	75-127	MSD	40
Tetrachloroethene	10-139	70-124	MSD	40
Toluene	31-136	75-128	MSD	40
trans - 1,2-Dichloroethene	33-128	68-122	MSD	40
trans - 1,3-Dichloropropene	10-131	68-114	MSD	40
trans - 1,4-Dichloro-2-Butene	70-130	70-130	MSD	40
Trichloroethene	37-135	55-134	MSD	40
Trichlorofluoromethane	46-110	58-129	MSD	40
Vinyl Acetate	70-130	10-144	MSD	40
Vinyl Chloride	34-165	55-155	MSD	40
Semivolatile Organic Compounds				
Phenol	35 - 128	35 - 128	MSD	20
2-Chlorophenol	25 - 130	25 - 130	MSD	20
Bis(2-chloroethyl)ether	27 - 126	27 - 126	MSD	20
1,3-Dichlorobenzene	25 - 119	25 - 119	MSD	20
1,4-Dichlorobenzene	27 - 123	27 - 123	MSD	20
1,2-Dichlorobenzene	27 - 130	27 - 130	MSD	20
2-Methylphenol	26 - 128	26 - 128	MSD	20
Bis(2-chloroisopropyl)ether	48 - 136	48 - 136	MSD	20
4-Methylphenol	28 - 121	28 - 121	MSD	20
Hexachloroethane	24 - 124	24 - 124	MSD	20
N-Nitroso-di-n-propylamine	29 - 132	29 - 132	MSD	20
Nitrobenzene	34 - 119	34 - 119	MSD	20
Isophorone	46 - 112	46 - 112	MSD	20
2,4-Dimethylphenol	12 - 108	12 - 108	MSD	20
2-Nitrophenol	35 - 105	35 - 105	MSD	20
Bis(2-chloroethoxy)methane	39 - 109	39 - 109	MSD	20
2,4-Dichlorophenol	39 - 112	39 - 112	MSD	20

Table A7-2. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples

Analysis	Matrix Spike Recovery (percent)	Laboratory Control Sample Recovery (percent)	Type of Duplicate	Control Limit Relative Percent Difference
1,2,4-Trichlorobenzene	32 - 116	32 - 116	MSD	20
Hexachlorobutadiene	31 - 110	31 - 110	MSD	20
Naphthalene	24 - 119	24 - 119	MSD	20
4-Chloroaniline	D - 111	D - 111	MSD	20
4-Chloro-3-methylphenol	46 - 112	46 - 112	MSD	20
2-Methylnaphthalene	37 - 112	37 - 112	MSD	20
Hexachlorocyclopentadiene	26 - 105	26 - 105	MSD	20
2,4,6-Trichlorophenol	D - 139	D - 139	MSD	20
2,4,5-Trichlorophenol	30 - 128	30 - 128	MSD	20
2-Chloronaphthalene	30 - 111	30 - 111	MSD	20
2-Nitroaniline	34 - 123	34 - 123	MSD	20
Dimethyl phthalate	26 - 112	26 - 112	MSD	20
Acenaphthylene	36 - 112	36 - 112	MSD	20
2,6-Dinitrotoluene	31 - 117	31 - 117	MSD	20
3-Nitroaniline	28 - 111	28 - 111	MSD	20
Acenaphthene	33 - 117	33 - 117	MSD	20
4-Nitrophenol	22 - 123	22 - 123	MSD	20
Dibenzofuran	42 - 108	42 - 108	MSD	20
Diethylphthalate	34 - 123	34 - 123	MSD	20
2,4-Dinitrophenol	D - 150	D - 150	MSD	20
2,4-Dinitrotoluene	37 - 121	37 - 121	MSD	20
4-Chlorophenyl phenyl ether	36 - 110	36 - 110	MSD	20
Fluorene	29 - 120	29 - 120	MSD	20
N-nitrosodiphenylamine	30 - 113	30 - 113	MSD	20
4-Nitroaniline	41 - 124	41 - 124	MSD	20
4,6-Dinitro-2-methylphenol	11 - 144	11 - 144	MSD	20
4-Bromophenyl phenyl ether	39 - 115	39 - 115	MSD	20
Hexachlorobenzene	42 - 117	42 - 117	MSD	20
Pentachlorophenol	4 - 113	4 - 113	MSD	20
Phenanthrene	41 - 118	41 - 118	MSD	20
Anthracene	33 - 125	33 - 125	MSD	20
Carbazole	45 - 117	45 - 117	MSD	20
Di-n-butyl phthalate	38 - 128	38 - 128	MSD	20
Fluoranthene	33 - 118	33 - 118	MSD	20
Pyrene	42 - 122	42 - 122	MSD	20
Butyl benzyl phthalate	45 - 122	45 - 122	MSD	20
Bis(2-ethylhexyl) phthalate	46 - 119	46 - 119	MSD	20
3,3'-Dichlorobenzidine	16 - 116	16 - 116	MSD	20
Benzo(A)anthracene	41 - 127	41 - 127	MSD	20
Chrysene	41 - 123	41 - 123	MSD	20
Di-n-octyl phthalate	30 - 139	30 - 139	MSD	20
Benzo(B)fluoranthene	36 - 126	36 - 126	MSD	20
Benzo(K)fluoranthene	44 - 125	44 - 125	MSD	20
Benzo(A)pyrene	27 - 127	27 - 127	MSD	20
Dibenzo(A,H)Anthracene	35 - 118	35 - 118	MSD	20
Indeno(1,2,3-C,D) Pyrene	35 - 113	35 - 113	MSD	20
Benzo(G,H,I) perylene	25 - 120	25 - 120	MSD	20
PCB Congeners				
All target analytes	NA	50-150	LCSD	50
Chlorinated Dioxins and Furans				
All target analytes	NA	50-150	LCSD	50

Notes
Note 1 - RPD control limit is not applicable. Laboratory control limit is ± 10 percent in the weight of the fraction.
Note 2 - Percent recovery control limits are not applicable. Laboratory control limits are established based on the manufacturer's established range of acceptable concentrations.

LD - laboratory duplicate
MSD - matrix spike duplicate
LCSD - laboratory control sample duplicate

Control limits are updated periodically by the laboratories. Control limits that are in effect at the laboratory at the time of analysis will be used for sample analysis and data validation. These may differ slightly from the control limits shown in this table.

Table B2-1. Sample Containers and Preservation Requirements for Surface, Subsurface, and Beach Sediment

Container[1]		Laboratory	Analysis	Preservation	Holding Time	Sample Size[2]
Type	**Size**					
Sediment samples						
G/P	8 oz	CAS	Grain size (sediment)	4±2ºC	6 months	100 g
G/P	8 oz	CAS	Atterberg limits and specific gravity	4±2ºC	6 months	225 g
WMG	2 oz	CAS	Total sulfides	No headspace; 4±2ºC (do not freeze)	7 days	5 g
WMG	16 oz [3]	CAS	Ammonia	4±2ºC	7 days	40 g
			Total organic carbon		28 days[4]	1 g
			Mercury		28 days[5]	5 g
			Metals and total solids		6 months[4]	10 g
			Hexavalent chromium		1 month/7days[6]	5 g
			Butyltins		14 days[4]	20 g
			TPH - diesel- and oil-range		14 days[4]	20 g
WMG	2 oz	CAS	TPH - gasoline range	No headspace; 4±2ºC (do not freeze)	14 days	5 g
WMG	2 oz	CAS	VOCs	No headspace; 4±2ºC (do not freeze)	14 days	5 g
WMG	16 oz	NEA	SVOCs	Deep Frozen (-20ºC)	1 year	50 g
			Pesticides		1 year	25 g
			PCBs		1 year	25 g
WMG	8 oz	STL	Herbicides	Deep Frozen (-20ºC)	1 year	50 - 80 g
WMG	8 oz	CAS	PCDD/PCDFs	Deep Frozen (-20ºC)	1 year	50 g
			PCB congeners		1 year	10 g
HDPE	4 L	NAS	Bioassay	4±2ºC in the dark (do not freeze)	14 days	750-1000 g
WMG	Two 8 oz	CAS	Archival	Deep Frozen (-20ºC)	1 year	not applicable
Equipment Rinse Blanks						
HDPE	500 mL	CAS	Metals and Mercury	5 ml of 1:1& HNO_3 & 4±2ºC	6 months/60 days[7]	100 ml
Poly-carbonate	500 mL	CAS	Butyltins	Dark; 4±2ºC	7 days	500 mL
VOA vial	2 oz/ septum	CAS	TPH - gasoline	No headspace; HCl to pH 2; 4±2ºC	14 days	5 mL
AG	500 mL	CAS	TPH - diesel and oil	HCl to pH 2; 4±2ºC	14 days	500 mL
VOA vial	2 oz/ septum	CAS	VOCs	No headspace; HCl to pH 2; 4±2ºC	14 days	5 mL
AG	500 mL	NEA	SVOCs	Dark; 4±2ºC	7 days/40 days[8]	500 mL
AG	500 mL	NEA	Pesticides	Dark; 4±2ºC	7 days/40 days[8]	500 mL
AG	500 mL	NEA	PCBs	Dark; 4±2ºC	7 days/40 days[8]	500 mL
AG	500 mL	STL	Herbicides	Dark; 4±2ºC	7 days/40 days[8]	500 mL
AG	500 mL	CAS	Dioxins/Furans	Dark; 4±2ºC/-10ºC[8]	1 year/1 year[8]	500 mL
AG	500 mL	CAS	PCB congeners	Sulfuric acid to pH 2-3; 4±2ºC/-10ºC[8]	1 year/1 year[9]	500 mL

WMG = Wide Mouth Glass
HDPE = High Density Polyethylene
AG = Amber Glass
G/P = Glass or Plastic

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[1] The size and number of containers may be modified by analytical laboratory. Archive samples will be collected for all of the sediment samples. Up to 16 ounces of sediment will be collected, as available, for samples scheduled for analysis and 32 ounces will be collected, as available, for archiving for samples that are not initially scheduled for analysis.

[2] All samples will need a minimum of 5% QA. Collection of 3x normal sample size listed will be necessary.

[3] An additional 8 oz to 16 oz jar needed for lab QC for 5% of samples.

[4] Holding times for frozen samples are as follows: Total organic carbon, 1 year; metals (except mercury), 2 years; butyltin species, 6 months; diesel- and oil-range TPH, 1 year.

[5] The holding time for mercury in frozen (i.e, archived) samples is 180 days, as approved by EPA (Humphrey 2002).

[6] Holding time is 1 month to extraction and extracts must be analyzed within 7 days from extraction.

[7] Based on EPA Method 1631 Revision D.

[8] Holding time is 7 days to extraction and extracts must be analyzed within 40 days from extraction.

[9] Conditions for equipment blanks/conditions for extracts.

APPENDIX A

PROJECT DQOS

As provided in the Programmatic Work Plan dated April 23, 2004.

Table A-1. The DQO Process for Understanding the Physical River System.

DQO Step	**Output**
1. State the Problem	The spatial and temporal scales of sediment movement and transport are not known. Sediment transport may affect contaminant nature and extent, source transport, recontamination potential, and ecological and human health exposure. Sediment transport processes / hydrodynamics also affect selection of remedial alternatives.
2. Identify the Decision to be Made	Determine the effect of sediment transport on risk estimates. Determine whether physical processes could expose previously buried contaminated sediment. Determine whether physical processes could result in burial of contaminated sediment. Determine physical system types for SMA development. Determine short- and long-term flow regimes for remedial alternatives development.
3. Identify the Inputs to the Decision	Time-series bathymetric surveys (high and low flow conditions). Sediment erosion/accretion stake measurements in beach areas where bathymetry cannot be measured. Temporal surface sediment chemistry comparisons in areas with appropriate historical data. Physical and chemical surface and subsurface sediment data Hydrodynamic/sediment transport model; inputs will include: • Bathymetry • Surface and subsurface bed sediment data • Tidal data • River flows • Sediment inflows (sands and fines) • Wind speed and direction • Others (TBD by modeler) The model must document uncertainties and identify which parameters most strongly affect the outcome of the model.

Table A-1. The DQO Process for Understanding the Physical River System.

DQO Step	**Output**
4. Define the Boundaries	Model hydrodynamic conditions from Willamette to confluence with Columbia River, focus sediment transport modeling on RM 2 to 11. Model to span annual high and low flow conditions. Hydrodynamic portion of model needs to predict both major flood years and non-flood years.
5. Develop a Decision Rule	Define the distributions of physical system types (i.e., potential for and magnitudes of erosional, depositional, transitional transport and stable regimes under a range of flow conditions). Define peak bed velocities under a variety of flow conditions at various locations for use in remedial alternatives evaluation.
6. Specify Tolerable Limits on Decision Error (per US EPA DQO guidance)	Null Hypothesis 1: Potential sediment transport does not significantly affect risk. Alternative Hypothesis 1: Potential sediment transport does significantly affect risk (by exposing subsurface sediments). Null Hypothesis 2: Potential sediment transport does not significantly affect remedial alternatives and evaluations. Alternative Hypothesis 2: Potential sediment transport does significantly affect remedial alternatives and evaluations. Decision Error: Error rate in physical measurements cannot be greater than the depth over which a decision will be made: • Need ± 6 inches on bathymetric measurements. Based on model calibration and validation results, model must be sufficiently accurate at a reasonable confidence level. Risk assessments and remedial alternatives identification and evaluation must take into account model results and associated uncertainties.

Table A-1. The DQO Process for Understanding the Physical River System.

DQO Step	**Output**
7. Optimize the Design	Collect bathymetry and flow (ADCP) data during a high flow (> 100,000 cfs) in the LWR. Continue to monitor sediment stakes in beach areas in Round 2. Select and set up hydrodynamic model using existing data during Round 2. Calibrate model to period December 2001 to September 2002 using bathymetric change data from that period. Validate the model over the period from September 2002 to February 2004 using bathymetric change data from that period. Following the preliminary modeling effort: • Identify data types and subareas where additional data are needed to meet modeling objectives • Identify subareas that may warrant additional focus due to the sediment exposure potential or for the remedial alternatives evaluation

Table A-2. The DQO Process for Understanding Chemical Distributions in Sediments and Sources.

DQO Step	**Output**
1. State the Problem	Historical data show that chemicals are present in sediments in the Lower Willamette River. However, documentation of the distribution of sediment chemical concentrations, although extensive, is not complete in all areas and the influence of sources is also not well understood in all areas. Surface sediments may act as a source of chemicals to other areas of the river and the transport of chemicals into and out of the ISA is not well understood. The stability of sediment chemical distributions is uncertain, based on known physical transport processes. Chemicals bound to sediments may pose a risk to human and ecological receptors.
2. Identify the Decision to be Made	Determine the nature and extent of chemicals in sediment including in areas that have not been characterized previously. Determine whether spatial trends in chemical distributions in sediment are consistent over time. Determine whether potential source areas influence sediment chemical distributions. Determine whether contiguous contamination posing unacceptable risk extends beyond the ISA.
3. Identify the Inputs to the Decision	Existing Category 1 sediment data from the Weston (1998) study and other in-water investigations (assume historical Category 1 data are acceptable for characterization of sediment quality). Sediment physical properties based on bathymetric studies, sediment trend analysis, sediment profile imaging, sediment stakes, and hydrodynamic modeling. Information on upland sources. New surface and subsurface sediment data.

Table A-2. The DQO Process for Understanding Chemical Distributions in Sediments and Sources.

DQO Step	**Output**
4. Define the Boundaries	Focus on the ISA and include limited sampling above and below the ISA. Surface sediment is defined as the surficial 1 ft (30 cm) of sediment. Subsurface sediment is defined as sediment deeper than 30 cm below the mudline. Bank areas to bottom of channel to coincide with risk assessment exposure areas. Collect new surface sediment data during low flow conditions (i.e., July – October) when physical transport processes are reduced. Detection limits will be those currently achievable by the analytical laboratory conducting the analyses under the EPA-approved QAPP. Detection limits will be lower (if analytically achievable) than risk-based values for protection of sediment.
5. Develop a Decision Rule	Areas affected by sources will be identified by an analysis of concentration gradients of COPCs. Areas exceeding risk-based thresholds will be referred to the feasibility study as potential sediment management areas.
6. Specify Tolerable Limits on Decision Error	Sampling density is sufficient to evaluate ecological receptor and human use areas. Temporal variability in chemical concentrations does not mask chemical distribution trends associated with sources.

Table A-2. The DQO Process for Understanding Chemical Distributions in Sediments and Sources.

DQO Step	**Output**
7. Optimize the Design	Stratify sediment sampling by depth of the riverbed with a greater number of stations in shallower areas near known and suspected sources and fewer stations in the channel. Sample where potential human or ecological receptor exposure areas have been identified and where historical Category 1 data are lacking. Evaluate temporal trends in surface sediment concentrations by collecting data in some areas with historic data and comparing spatial trends over time. Define and sample surface sediment as the top 1 ft (30 cm) of sediment because that thickness accounts for the majority of sediment elevation changes over time and includes the biologically active zone. Sample subsurface sediments where scour may re-expose buried sediment deposits and in navigation/maintenance dredge and sediment management areas.

Table A-3. The DQO Process for Understanding Chemical Distributions in Surface Water.

DQO Step	**Output**
1. State the Problem	Information on concentrations and distribution of chemicals in the water column is limited.
2. Identify the Decision to be Made	Determine the nature and extent of chemicals in surface water entering, within, and exiting the ISA. Determine whether the distribution of chemicals in surface water is spatially and temporally consistent. Determine whether chemical concentrations representing a risk extend contiguously beyond the ISA. Determine potential for widespread source effects to river water and associated risks to ecological and human receptors. Refer to EPA and DEQ for source control as appropriate.
3. Identify the Inputs to the Decision	Existing Category 1 water quality data. Distributions and concentrations of chemicals in the water column (Round 2). Risk-based water quality action levels may need to be identified.
4. Define the Boundaries	Immediately upstream, downstream, and within the ISA. Generate new water column data during an early fall “first flush” stormwater event and both low-flow and high-flow conditions near the surface and near the bottom of the river. Detection limits will be those currently achievable by the analytical laboratory conducting the analyses, as approved by EPA in the QAPP. Detection limits will be lower (if analytically achievable) than risk-based values for protection of water quality.
5. Develop a Decision Rule	If concentrations exceed risk-based water quality screening levels, refer to EPA and DEQ for source control follow-up. Time-series chemistry data for each chemical are within the same range of concentrations over time and trigger the same risk management decision.

Table A-3. The DQO Process for Understanding Chemical Distributions in Surface Water.

DQO Step	**Output**
6. Specify Tolerable Limits on Decision Error	Sampling density is sufficient to estimate water quality entering, within, and exiting the ISA. Sampling frequency is sufficient to estimate water quality under low flow and high flow conditions. Detection limits for sampling and analytical methods are adequate to be below risk-based water quality screening levels. Sample location and density must be adequate to understand the potential for source effects to river water and sediments, but not for identifying and characterizing individual sources.
7. Optimize the Design	Collect water samplesusing a cross-sectional integrated flow sampling method (high-volume and grabs) suitable to achieve the target detection limits. Collect water samples using high-volume and grab sampling methods in Round 2 along three transects from shore to shore in the ISA. Locations of transects will be chosen to measure water quality parameters entering the ISA (RM 11), within the ISA (RM 6), and leaving the ISA (RM 3.5). Collect water samples using grab sampling methods at potential swimming areas. Collect water samples in Round 2 during summertime low flows and in the fall shortly after flows have increased and storm drains have potentially been flushed of particulates that accumulated over the previous summer. Collect water samples using high-volume and grab sampling methods in Round 2 at four locations (Rhone-Poulenc, Willamette Cove, ATOFINA, and Portland Shipyard) during optimum-flow sampling event.

Table A-4. The DQO Process for Understanding the Hydrogeologic Physical System, the Effects of Groundwater Discharges on Ecological and Human Health Risks and the Distribution of Chemicals in Sediment.

DQO Step	**Output**
1. State the Problem	**Ecological Risk Assessment**: Potential significant contributions to risk to ecological receptors from groundwater flow through sediments within the ISA are unknown. **Human Health Risk Assessment**: The potential exposure of human receptors at potential human uses areas within the ISA to groundwater COIs is unknown. **Hydrogeologic Physical System**: A better understanding of the relationship between groundwater and surface water is necessary as a basis for the evaluation of the potential effects of the groundwater physical system on exposure pathways for the site. **Chemical Distribution and Sources**: Flow of contaminated groundwater through sediments can affect sediment quality. A better understanding of the locations of upland contaminated groundwater sources is needed to identify locations and prioritize sites where groundwater may adversely affect sediments and environmental receptors in the river.

Table A-4. The DQO Process for Understanding the Hydrogeologic Physical System, the Effects of Groundwater Discharges on Ecological and Human Health Risks and the Distribution of Chemicals in Sediment.

DQO Step	**Output**
2. Identify the Decision to be Made	**Ecological Risk Assessment**: Determine where COIs in groundwater have the potential to adversely impact to sediment and/or porewater quality. Determine where potentially ecotoxic groundwater contaminants are not captured through whole sediment analysis. Determine the scale of investigation necessary for assessing ecological risks from groundwater COIs Determine whether and where collection of bulk sediment and porewater data may be necessary for ecological risk assessment purposes. Identify locations where groundwater data are lacking and where additional investigation and/or sampling may be needed to support risk-based evaluations. Determine whether contaminated groundwater in the harbor represents unacceptable risks to ecological receptors. If risk is unacceptable, determine the location and extent of source control and/or remediation. **Human Health Risk Assessment**: Determine if and where COIs in groundwater could discharge to the surface in potential human use areas. Determine if additional investigation of the groundwater pathway is necessary in potential human use areas. **Hydrogeologic Physical System**: Determine the scope of sample collection activities for the ecological and human health risk assessments based on understanding the spatial and temporal relationships between groundwater and surface water in the ISA. **Chemical Distribution and Sources**: Determine where COIs in groundwater have the potential to adversely affect sediments and environmental receptors in the river.

Table A-4. The DQO Process for Understanding the Hydrogeologic Physical System, the Effects of Groundwater Discharges on Ecological and Human Health Risks and the Distribution of Chemicals in Sediment.

DQO Step	**Output**
3. Identify the Inputs to the Decisions	**Ecological Risk Assessment**: Existing regional, ISA-wide and site specific hydrogeologic data; Existing upland groundwater quality data from upland groundwater investigations under DEQ oversight including the locations and the ranges of concentrations of COIs in groundwater from upland sites adjacent to the ISA and are likely to affect sediment (including porewater) quality. Porewater data from locations where groundwater COIs in sediments are not adequately characterized by the whole sediment sampling and analysis or the benthic approach are likely present, and could cause a significant risk to ecological receptors. **Human Health Risk Assessment**: Locations of identified seeps based on seep reconnaissance survey results Locations of identified potential human use areas Existing upland groundwater quality data in areas adjacent to potential human use areas and/or groundwater quality data from the seeps. **Hydrogeologic Physical System**: Existing regional, ISA-wide and site specific hydrogeologic data; **Chemical Distribution and Sources**: Existing regional, ISA-wide and site specific hydrogeologic data; Existing upland groundwater quality data from upland groundwater investigations including the locations and the ranges of concentrations of COIs in groundwater from upland sites adjacent to the ISA and are likely to affect sediment quality.

Table A-4. The DQO Process for Understanding the Hydrogeologic Physical System, the Effects of Groundwater Discharges on Ecological and Human Health Risks and the Distribution of Chemicals in Sediment.

DQO Step	**Output**
4. Define the Boundaries	**Ecological Risk Assessment**: Within the ISA The initial assessment of groundwater contributions to risks to ecological receptors will be spatially focused on areas where COIs are likely to be discharging to sediments. Porewater sample collection will be spatially focused on areas where COIs are determined to be discharging to sediments, the COIs may cause an unacceptable risk, and the risk from COIs will not be captured by bulk sediment analysis and bioassays. **Human Health Risk Assessment**: Within the ISA Assessment of potential human health risks from groundwater will be spatially focused on locations where COIs potentially discharge in seeps at potential human use areas. **Hydrogeologic Physical System**: RM 2 to 11 for hydrogeologic conceptual model refinement. The conceptual model will encompass the hydrostratigraphic units from the CRBG up through surficial soils. The shallow and intermediate groundwater systems (e.g., groundwater present in the FGF) will be the basis for determining where groundwater contaminants may affect sediment quality except at locations where available information indicates that deeper units (e.g., the CGF and CRBG) are impacted by groundwater contaminants. **Chemical Distribution and Sources**: RM 2 to 11 for identifying the locations where contaminated groundwater has the potential to adversely affect sediment quality. Locations where potential for known groundwater plumes to reach the river both in the past and present.

Table A-4. The DQO Process for Understanding the Hydrogeologic Physical System, the Effects of Groundwater Discharges on Ecological and Human Health Risks and the Distribution of Chemicals in Sediment.

DQO Step	**Output**
5. Develop a Decision Rule	**Ecological Risk Assessment**: If the chemical concentration in groundwater or porewater at the point of exposure for COIs is greater than the NOEC (potential risk to sensitive species) or AWQC (for aquatic organisms), the locations will be targeted for future sediment/biota sampling (Round 2) and the area will be referred to DEQ for further evaluation or action. **Human Health Risk Assessment**: Presence of groundwater COIs adjacent to the location of a seep or in the seep itself in a potential human use area. Refer site to DEQ for further assessment under the source control program. **Hydrogeologic Physical System**: Concentrate risk assessment screening and characterization activities at the locations with COIs in groundwater where discharge to the river from the flow system is focused. **Chemical Distribution and Sources**: Locate shallow grab sediment sample stations in the vicinity of where COIs detected in upland groundwater are identified as having a potential to intersect the river

Table A-4. The DQO Process for Understanding the Hydrogeologic Physical System, the Effects of Groundwater Discharges on Ecological and Human Health Risks and the Distribution of Chemicals in Sediment.

DQO Step	**Output**
6. Specify Tolerable Limits on Decision Errors	**Ecological Risk Assessment**: Existing upland groundwater COI data are representative of groundwater COI concentrations reasonably expected to reach the Transition Zone. **Human Health Risk Assessment**: Existing upland groundwater data are sufficient to assess the presence of groundwater COIs in the vicinity of seeps at potential human use areas. **Hydrogeologic Physical System**: Existing data are sufficient to assess temporal and spatial variability in groundwater interactions with the river to identify locations of potential contaminant discharge at a scale relevant to site risk decisions. **Chemical Distribution and Sources**: Existing data are sufficient to identify the types and general locations where COIs in groundwater could intersect the river at a scale relevant to site risk decisions.

Table A-4. The DQO Process for Understanding the Hydrogeologic Physical System, the Effects of Groundwater Discharges on Ecological and Human Health Risks and the Distribution of Chemicals in Sediment.

DQO Step	Output
7. Optimize the Design for Obtaining Data	Review available upland groundwater and geologic data to identify the locations, types and concentrations of groundwater COIs that potentially could discharge to the river. Screen for groundwater COIs and identify high priority sites. Review data from the high priority sites, as available, to assess risk, identify where groundwater data are lacking, and assess the need for additional assessment and/or porewater or sediment sampling. Conduct preliminary porewater/sediment investigations at select locations to help determine the overall likelihood that contaminated groundwater affects porewater/sediment exposures in the river. Collect porewater/sediment samples at locations identified by ecological risk screening process (1) if the data are not already available from PRP efforts conducted under individual upland source control or early action programs, and (2) if and where warranted because of RI/FS timing issues. Apply the results of the ecological risk screening process and sampling results to assess if other porewater/sediment sampling is necessary within the ISA.

Table A-5. The DQO Process for the Ecological Risk Evaluation: Fish.

DQO Step	**Output**
1. State the Problem	Fish may be at risk from exposure to chemicals resulting from historical and ongoing releases and / or sources within the ISA.
2. Identify the Decision to be Made	Determine whether exposure to hazardous substances in the ISA poses an unacceptable risk to fish in the area. The testable hypotheses are: Are levels of contaminants in abiotic and biotic media in the ISA sufficient to cause adverse effects to the growth, survival or reproduction of detritivorous fish utilizing the habitat within the ISA? Are levels of contaminants in abiotic and biotic media in the ISA sufficient to cause adverse effects to the growth, survival or reproduction of invertivorous fish utilizing the habitat within the ISA? Are levels of contaminants in abiotic and biotic media in the ISA sufficient to cause adverse effects to the growth, survival or reproduction of piscivorous fish utilizing the habitat within the ISA? Are levels of contaminants in abiotic and biotic media in the ISA sufficient to cause adverse effects to the growth, survival or reproduction of herbivorous fish utilizing the habitat within the ISA?
3. Identify the Inputs to the Decision	Existing Category 1 and Category 2 data were evaluated to define the conceptual site model and identify data gaps. Category 1 data will continue to be used to update the conceptual site model and re-evaluate data gaps. Existing fish life history information (described in Appendix B) will be evaluated to determine potential exposure areas. Toxicological literature will be evaluated to determine potential toxicity and/or bioavailability issues. Tissue residue data from the literature will be used to determine adverse effect levels. Surface sediment, surface water, invertebrate tissue (benthic infauna) and fish tissue (sculpin, juvenile chinook salmon, largescale sucker, peamouth, pikeminnow, smallmouth bass) data were or will be collected in exposure areas.* Tissue concentrations of dioxin-like compounds and PCB coplanars measured in carp for the human risk assessment will be used to assess risk to fish (and higher trophic levels, see Appendix B, Section 5.3).

Table A-5. The DQO Process for the Ecological Risk Evaluation: Fish.

DQO Step	**Output**
4. Define the Boundaries	The ISA will be the initial geographic boundary. Spatial boundaries on exposure areas are different for each fish species and will depend on fish foraging habits within the river. Fish and invertebrate tissue and surface sediment have been collected and surface water will be collected. Detection limits will be those currently achievable by the analytical laboratory conducting the analyses. Detection limits will be lower (if analytically achievable) than risk-based values for protection of fish.
5. Develop a Decision Rule	If the COPC concentration using the 95th UCL or maximum concentration is greater than the NOEC in the special-status species assessment, the COPC will be retained for further evaluation. If the COPC concentration using the 95th UCL or maximum concentration is greater than the LOEC in the population level assessment, the COPC will be retained for further evaluation.
6. Specify Tolerable Limits on Decision Errors	Null Hypothesis: Fish may have unacceptable risk from exposure to hazardous substances within the ISA. Alternate hypothesis: There is no risk to fish from exposure to hazardous substances within the ISA. Evaluate ecosystem and receptor characteristics that may modify/impact risk management decision. Evaluate uncertainty of exposure concentrations relative to sample design. Evaluate uncertainty of toxicity values relative to decision rule.
7. Optimize the Design	Collect surface water samples for comparison to effects-based criteria (e.g., AWQC). Collect additional fish tissue, if warranted, from exposure areas to compare to tissue residue effects data. Collect additional invertebrate tissue, if warranted, and sediment grab samples to evaluate dietary pathway (dietary-based NOEAL or LOAEL) in exposure areas.

*The ecological risk assessment will also evaluate the tissue samples of carp, brown bullhead, and black crappie collected for the human health risk assessment. This evaluation will be done in the uncertainly section to provide additional information in order to address uncertainties with the risk characterization conducted on the representative species.

Table A-6. The DQO Process for the Ecological Risk Evaluation: Birds.

DQO Step	**Output**
1. State the Problem	Birds may be at risk from exposure to chemicals resulting from historical and ongoing releases and / or sources within the ISA.
2. Identify the Decision to be Made	Determine whether or not exposure to hazardous substances in the ISA poses an unacceptable risk to birds that may forage in the area. The testable hypotheses are: Are levels of contaminants in abiotic and biotic media in the ISA sufficient to cause adverse effects to the growth, survival or reproduction of diving carnivorous birds utilizing the habitat within the ISA? Are levels of contaminants in abiotic and biotic media in the ISA sufficient to cause adverse effects to the growth, survival or reproduction of sediment probing invertivorous birds utilizing the habitat within the ISA? Are levels of contaminants in abiotic and biotic media in the ISA sufficient to cause adverse effects to the growth, survival or reproduction of piscivorous birds utilizing the habitat within the ISA?
3. Identify the Inputs to the Decision	Existing Category 1 and Category 2 data were evaluated to determine potential exposure areas and data gaps. Existing life history information of representative avian species will be reviewed to select appropriate representative species and exposure parameters. Toxicological literature will be searched to develop no observed adverse effects level (NOAEL) and lowest observed adverse effects level (LOAEL) for birds to determine relative sensitivities. Bird exposure areas will be determined based on a reconnaissance habitat survey and evaluation of their local life-history characteristics. Surface sediment and prey (crayfish, clams, fish) were collected in bird exposure areas.
4. Define the Boundaries	The ISA will be the initial geographic boundary. Sediment and fish and invertebrate tissue were collected in bird exposure areas within the ISA. Data was collected in summer/fall 2002. Detection limits will be those currently achievable by the analytical laboratory conducting the analyses as described in the approved QAPP. Detection limits will be lower (if analytically achievable) than risk-based values for protection of avian species.

Table A-6. The DQO Process for the Ecological Risk Evaluation: Birds.

DQO Step	**Output**
5. Develop a Decision Rule	If the dose estimate using the 95th UCL or maximum concentration is greater than the NOAEL in the special-status species assessment, the COPC will be retained for further evaluation. If the dose estimate using the 95th UCL or maximum concentration is greater than the LOAEL in the population level assessment, the COPC will be retained for further evaluation.
6. Specify Tolerable Limits on Decision Errors	Null Hypothesis: Birds may have unacceptable risk from exposure to hazardous substances within the ISA. Alternate Hypothesis: There is no risk to birds from exposure to hazardous substances within the ISA. Evaluate ecosystem and receptor characteristics that may modify/impact risk management decision. Evaluate uncertainty of exposure concentrations relative to sample design. Evaluate uncertainty of toxicity values relative to decision rule.
7. Optimize the Design	Collect additional surface sediment samples, if warranted, in each shorebird bird exposure area and at other bird habitat areas. Collect additional prey tissue (invertebrate and/or fish tissue), if warranted, from each bird exposure area. Additional sampling may be conducted based on the results of the iterative risk assessment to reduce uncertainties.

Table A-7. The DQO Process for the Ecological Risk Evaluation – Mammals.

DQO Step	**Output**
1. State the Problem	Mammals may be at risk from exposure to chemicals resulting from historical and ongoing releases and / or sources within the ISA.
2. Identify the Decision	Determine whether exposure to hazardous substances in the ISA poses an unacceptable risk to mammals that may forage in the area. The testable hypothesis is: Are levels of contaminants in abiotic and biotic media in the ISA sufficient to cause adverse effects to the growth, survival or reproduction of mammals utilizing the habitat within the ISA?
3. Identify the Inputs to the Decision	Existing Category 1 and Category 2 data were evaluated to determine potential exposure areas and data gaps. Existing life history information of representative mammals were reviewed to select an appropriate representative species and exposure parameters. Toxicological literature will be searched to develop no observed adverse effects level (NOAEL) and lowest observed adverse effects level (LOAEL) for mammals to determine relative sensitivities. Surface sediment and prey data (crayfish, clams, fish) were collected in mammalian exposure areas.
4. Define the Boundaries	The ISA will be the initial geographic boundary. Surface sediment and fish/invertebrate tissue chemistry data were collected from mammalian exposure areas. Detection limits will be those currently achievable by the analytical laboratory conducting the analyses as described in the approved QAPP. Detection limits will be lower (if analytically achievable) than risk-based values for protection of mammalian species.
5. Develop a Decision Rule	If the dose estimate using the 95th UCL or maximum concentration is greater than the NOAEL in the special-status species assessment, the COPC will be retained for further evaluation. If the dose estimate using the 95th UCL or maximum concentration is greater than the LOAEL in the population level assessment, the COPC will be retained for further evaluation.

Table A-7. The DQO Process for the Ecological Risk Evaluation – Mammals.

DQO Step	**Output**
6. Specify Tolerable Limits on Decision Errors	Null Hypothesis: Mammals may have unacceptable risk from exposure to hazardous substances within the ISA. Alternate Hypothesis: There is no risk to mammals from exposure to hazardous substances within the ISA. Evaluate ecosystem and receptor characteristics that may modify/impact risk management decision. Evaluate uncertainty of exposure concentrations relative to sample design. Evaluate uncertainty of toxicity values relative to decision rule.
7. Optimize the Design	Collect additional surface sediment samples, if warranted, in each mammalian exposure area. Collect additional co-located prey tissue (invertebrate and/or fish tissue), if warranted, from each mammalian exposure area.

Table A-8. The DQO Process for the Ecological Risk Evaluation: Amphibians, Reptiles, and Plants.

DQO Step	**Output**
1. State the Problem	Amphibians and Reptiles: Amphibians and/or reptiles may be at risk from exposure to chemicals resulting from historical and ongoing releases and/or sources within the ISA. Aquatic Plants: Aquatic plants may be at risk from exposure to chemicals resulting from historical and ongoing releases and / or sources within the ISA.
2. Identify the Decision	Determine whether exposure to hazardous substances in the ISA poses an unacceptable risk to amphibians or reptiles that may forage in the area or aquatic plants. The testable hypothesis is: Are levels of contaminants in abiotic and biotic media in the ISA sufficient to cause adverse effects to the growth, survival or reproduction of amphibians, reptiles or aquatic plants utilizing the habitat within the ISA?
3. Identify the Inputs to the Decision	Presence/absence of amphibian and aquatic plants was confirmed with field reconnaissance survey. Reptiles were not found but have been found in the ISA during other field surveys. Evaluation of existing amphibian life history information and plant community information to determine potential habitat areas and potential for exposure. Evaluation of toxicological literature to determine potential toxicity and/or bioavailability. Collection of surface water in quiescent areas and other potential exposure areas.
4. Define the Boundaries	The ISA will be the initial geographic boundary. Risk to amphibians will be assessed quantitatively if possible. Risk to aquatic plants cannot be quantitatively assessed because of lack of appropriate toxicity data. Amphibians will be used as surrogate to assess the risk to reptiles. If suitable reptile habitat is found within the ISA a comparative evaluation of toxicity will be performed using literature data. Temporal variability will influence study design. Detection limits will be those currently achievable by the analytical laboratory conducting the analyses as described in the approved QAPP. Detection limits will be lower (if analytically achievable) than risk-based values for protection of amphibians and aquatic plants.

Table A-8. The DQO Process for the Ecological Risk Evaluation: Amphibians, Reptiles, and Plants.

DQO Step	**Output**
5. Develop a Decision Rule	If the COPC concentration using the maximum concentration is greater than the NOEC/LOEC for amphibians, the COPC will be retained for further evaluation (NOEC used for sensitive species). Aquatic plants will be assessed qualitatively. Amphibian assessment will be a surrogate for the reptile evaluation since the amphibians are more sensitive.
6. Specify Tolerable Limits on Decision Errors	Null Hypothesis: Amphibians and aquatic plants are exposed to hazardous substances within the ISA. Alternate Hypothesis: Amphibians and aquatic plants are not exposed to hazardous substances within the ISA. Evaluation of variability of exposure concentrations relative to sample design. Evaluation of the variability of toxicity values relative to the decision rule.
7. Optimize the Design	Surface water samples will be collected in quiescent areas and within other potential habitat areas.

Table A-9. The DQO Process for the Human Health Risk Assessment.

DQO Step	**Output**
1. State the Problem	Need to estimate potential risks to human health associated with exposure to chemicals in sediment, surface water, groundwater seeps, and/or biota that are a result of historic and ongoing activities in the ISA.
2. Identify the Decision	Determine whether chemicals in sediment, surface water, groundwater seeps, or biota that are the result of historic and ongoing activities in the Site result in unacceptable risks to human health and warrant consideration of further investigation or possible response action.
3. Identify the Inputs to the Decision	Zoning maps, city plans, discussions with EPA and its partners, and site reconnaissance surveys were used to identify potential human use areas prior to Round 1 and Round 2. Beach sediment samples collected in potential human use areas during Round 1 and in-water sediment samples collected in the Site will be used to estimate potential exposure to chemicals in sediment. Surface water data will be collected during Round 2 and will be used to estimate potential exposure to chemicals in surface water. Technically defensible studies or EPA guidance that are appropriate for Portland Harbor will be used to identify ingestion rates that can be used for biota. Resident fish and shellfish tissue samples collected during Round 1, and salmon, sturgeon, and lamprey samples collected in the summer of 2003 by ODHS, ATSDR, ODF&W, City of Portland, and USEPA, Region 10 along with identified appropriate ingestion rates, will be used to estimate potential exposure to chemicals in tissue. A Seep Reconnaissance Survey was conducted to identify locations of groundwater seeps where human exposure may occur. Existing groundwater data or new groundwater or seep data collected during the RI may be used to estimate potential exposures to and risks from groundwater. Toxicity information will be derived in concordance with EPA Directive OSWER Directive 9285.7-53, Human Health Toxicity Values in Superfund Risk Assessments (December 5, 2003). Analytical concentration goals were developed to be protective of human health.

Table A-9. The DQO Process for the Human Health Risk Assessment.

DQO Step	**Output**
4. Define the Boundaries	Target populations: • Sediment samples • Surface water samples • Tissue samples Spatial boundaries: • Beach sediment – Surface beach sediment within human use areas of the Site • In-water sediment – Selected in-water sediments collected in Round 2 in areas within the Site where fishing occurs or commercial diving has been documented. • Surface water – River water samples within areas of the Site adjacent to beaches potentially used for recreation (e.g., Swan Island Lagoon) • Tissue – Resident fish and shellfish collected within the Site • Tissue – Salmon, sturgeon, and lamprey collected by ODHS, ATSDR, ODF&W, City of Portland, and USEPA, Region 10 during summer 2003. Time frame: • Beach sediment – During low water when most of bank is exposed and during summer when beach use is most likely. • In-Water sediment – All times • Surface water – During summer when swimming would occur • Tissue – All times with emphasis during April through October Practical constraints: • Field samples collected during times when access is adequate • Tissue – Sufficient quantity of individual species within ISA for composite samples

Table A-9. The DQO Process for the Human Health Risk Assessment.

DQO Step	**Output**
5. Develop a Decision Rule	If the risk estimate exceeds 1 x 10^{-6} for cancer risks and/or the hazard index exceeds 1.0 for noncancer risks, then evaluate the need for further investigations to gather additional site-specific data. The necessity for such site-specific data in making risk management decisions required for the ROD will be assessed prior to conducting further studies.
6. Specify Tolerable Limits on Decision Error	Conservative assumptions will be used and risks will be estimated using ranges of potential exposure values.
7. Optimize the Design	Collect surface sediment samples in human use areas Collect fish and shellfish tissue – whole body and fillets Collect surface water samples in human use areas

Table A-10. The DQO Process for Natural Attenuation Potential.

DQO Step	**Output**
1. State the Problem	Need to understand specific elements of the physical system sufficient to make a determination of candidate natural attenuation areas.
2. Identify the Decision	Determine if natural attenuation is a viable alternative that needs further investigation. If so, identify the areas most likely to be suitable for natural attenuation that require further study.
3. Identify Inputs to the Decision	Need the following data sufficient to run proposed natural attenuation models: • Surface sediment chemistry • Water content, specific gravity, and grain size • Hydrodynamic model results • Uncertainty and sensitivity analysis of the hydrodynamic model • Sedimentation rates based on select Be^7 and Pb^{210} cores (Rounds 2 and 3) • Chemistry of incoming sediments based on select water column samples for TSS, dissolved and total chemical analyses (Round 2) and sediment trap studies (Round 3) • Mixed Layer Depth – Select Be^7 and Pb^{210}cores (Rounds 2 and 3) • Mixing Rate – Radioisotope studies (Round 3) • Biodegradation Rates – from literature values • Groundwater velocities and chemical concentrations where this process is important – Round 3 • Analysis of existing sediment chemistry trends information to understand if natural attenuation already occurring (only applies to areas where sources have been controlled).

Table A-10. The DQO Process for Natural Attenuation Potential.

DQO Step	**Output**
4. Define the Boundaries to the Study	Conduct select Round 2 data gathering in areas of the ISA that may have potential for processes that support natural attenuation. Conduct Round 3 data gathering in areas that likely support natural attenuation processes including within the ISA as well as any expanded ISA areas that are at risk based on results of risk assessment
5. Develop a Decision Rule	Use existing physical information to determine most likely types of physical environments that have processes that may support natural attenuation. Conduct select sampling described in step 3, in these areas. Use resulting information to define a range of model parameter values. Input range of values into model and identify types of areas with physical processes that have a reasonable probability of supporting natural attenuation. Focus Round 3 investigations on types of areas with likely physical processes and refine natural attenuation estimates in these areas that also exhibit unacceptable risks based on the preliminary risk assessment. Define areas for the FS that may have natural attenuation as a viable remedial alternative for consideration and comparison against other remedial alternatives.
6. Specify Tolerable Limits on Decision Errors	Null hypothesis: Natural attenuation is an unacceptable remedial alternative in the ISA. Sampling and hydrodynamic modeling must be sufficient to provide a reasonable confidence that the spatial range of possible conditions has been sampled. This includes groundwater data collected through other efforts in this Work Plan and directed by DEQ at upland sites. Potential ranges of model inputs for each parameter must be established. Therefore sampling must include understanding of both spatial and temporal variability within areas that appear to support natural attenuation processes.
7. Optimize the Design for Obtaining Data	The detailed approach to natural attenuation modeling is described in the natural attenuation memorandum (Appendix A, Attachment A4). Keep Round 3 efforts flexible so that they can benefit from information gathered in Round 2.

APPENDIX B

SEDIMENT TOXICITY TESTING DRAFT

PORTLAND HARBOR RI/FS

ROUND 2A
SEDIMENT TOXICITY TESTING QAPP
APPENDIX B: SEDIMENT TOXICITY TESTING

June 24, 2004

Prepared for:
The Lower Willamette Group

Prepared by:
Windward environmental LLC
200 West Mercer Street, Suite 401 ⬩ Seattle, Washington ⬩ 98119

A1 Title and Approval Page
Bioassay Testing
Quality Assurance Project Plan

Windward Project Manager

Name	Date

EPA Project Manager

Name	Date

EPA Project Manager

Name	Date

A2 Table of Contents

A3 Distribution list

This list identifies all individuals to receive a copy of the approved QA Project Plan, either in hard copy or electronic format, as well as any subsequent revisions. In addition to the distribution list in the main text a copy should be sent to the following:

- Lisa Saban, Windward Project Manager
- Shawn Hinz, Windward QA/QC Manager
- Helle Andersen, Windward Laboratory Coordinator
- Dick Caldwell, Bioassay Laboratory Manager
- Gerald Irissarri, Bioassay Project Manager
- Michele Redmond, Bioassay Data Manager
- Linda Nemeth, Bioassay Laboratory QA/QC Officer
- Paul Dinnel, third-party QA/QC reviewer of bioassay data

Acronyms

Acronym	definition
ASTM	American Society for Testing and Materials
DEQ	Oregon Department of Environmental Quality
DQO	data quality objective
EPA	US Environmental Protection Agency
FC	field coordinator
LC	laboratory coordinator
LWG	Lower Willamette Group
NAS	Northwestern Aquatic Sciences
PM	project manager
QA/QC	quality assurance/quality control
QAPP	quality assurance project plan
RI	Remedial Investigation

A4 Introduction and Project Organization

A4.1 INTRODUCTION

This quality assurance project plan (QAPP) describes the quality assurance (QA) objectives, methods, and procedures for conducting sediment toxicity testing in support of the Portland Harbor remedial investigation (RI) as described in the Programmatic Work Plan (Integral 2004). US Environmental Protection Agency (EPA) guidance for QAPPs was followed in the preparation of this project plan (EPA 2002). This plan is organized into the following sections:

- Section A – project management
- Section B – data generation and acquisition
- Section C – assessment and oversight
- Section D – data validation and usability
- Section E – references

A4.2 PROJECT AND TASK ORGANIZATION

The overall project organization and the individuals responsible for the various tasks required for the sediment toxicity testing effort are shown in Figure A4-1. Responsibilities of these individuals are described in the following sections.

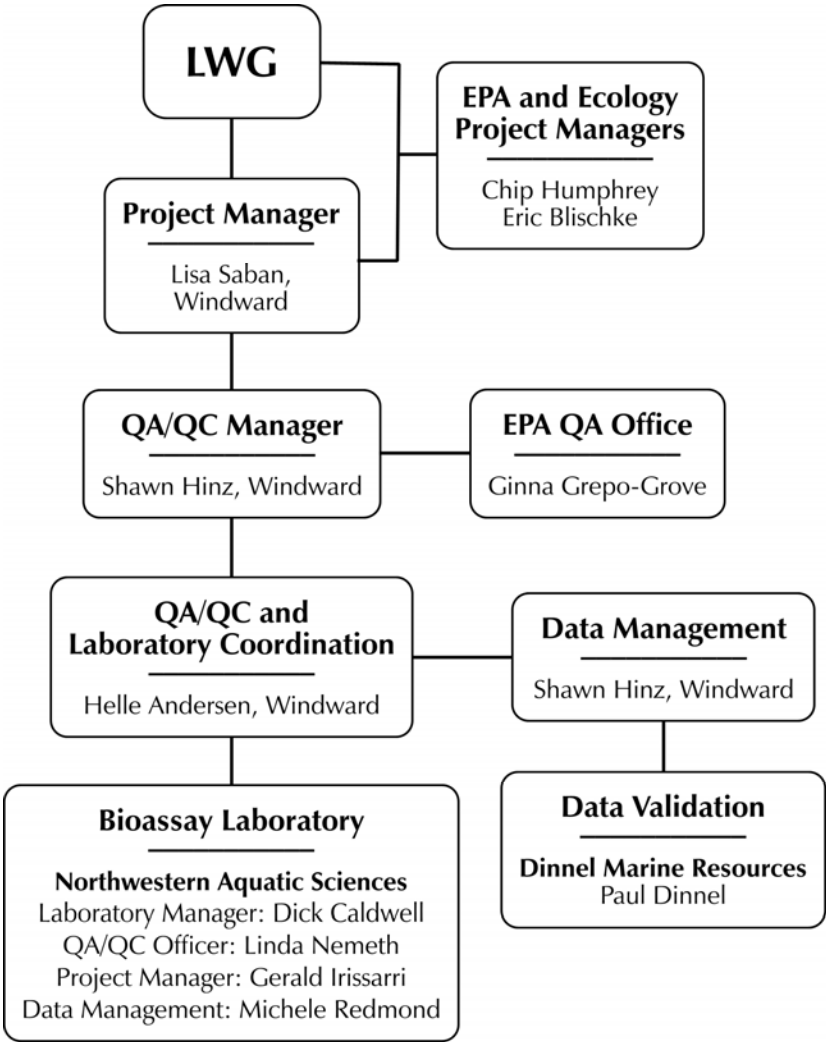

Figure A4-1. Project organization

A4.2.1 Project management

The Lower Willamette Group (LWG), Chip Humphrey (the EPA Project Manager [PM]), and Eric Blischke (EPA PM) will be involved in all aspects of this project, including discussion, review, and approval of this QAPP.

Lisa Saban will serve as the Windward PM. The PM is responsible for overall project coordination and provides oversight on planning and coordination, production of work plans, production of all project deliverables, and performance of the administrative tasks needed to ensure timely and successful completion of the project. The PM is also responsible for coordinating with LWG, and with the PMs for EPA and the Oregon Department of Environmental Quality (DEQ), on schedule, deliverables, and other administrative details. The PM can be reached as follows:

Lisa Saban
Windward Environmental LLC
200 W. Mercer St., Suite 401
Seattle, WA 98119
Telephone: 206.577.1288
Facsimile: 206.217.0089
E-mail: lisas@windwardenv.com

A4.2.2 Field coordination

See main text (Section A4.2.3)

A4.2.3 Quality assurance/quality control

Shawn Hinz of Windward will oversee quality assurance/quality control (QA/QC) for the project. As the QA/QC manager, he will provide oversight for the laboratory program, and supervise data validation and project QA coordination. The QA/QC manager can be reached as follows:

Shawn Hinz
Windward Environmental LLC
200 W. Mercer St., Suite 401
Seattle, WA 98119
Telephone: 206.838.5873
Facsimile: 206.217.0089
Email: shawnh@windwardenv.com

The QA/QC manager will provide all necessary information to EPA's QA office so that EPA can independently evaluate data quality. Ginna Grepo-Grove will be EPA's QA office representative for this project. She can be reached as follows:

Ginna Grepo-Grove
US Environmental Protection Agency, Region 10
1200 6th Avenue
Seattle, WA 98101
Telephone: 206.553.1632
Email: grepo-grove.gina@epa.gov

Helle Andersen will serve as Windward's QA/QC coordinator. The QA/QC coordinator will coordinate with the bioassay laboratory to ensure that QAPP requirements are followed. She can be reached as follows:

Helle Andersen
Windward Environmental LLC
200 W. Mercer St., Suite 401
Seattle, WA 98119
Telephone: 206.577.1287
Facsimile: 206.217.0089
Email: hellea@windwardenv.com

Independent third-party data review and validation will be provided by Paul Dinnel of Dinnel Marine Resources. Mr. Dinnel can be reached as follows:

Paul Dinnel
Dinnel Marine Resources
1519 13th St.
Anacortes, WA 98221
Telephone: 360.293.2188
Email: (b) (6) [redacted]

A4.2.4 Laboratory project management

Helle Andersen of Windward will serve as the laboratory coordinator (LC).

Northwestern Aquatic Sciences (NAS) will perform toxicity testing of the sediment samples. Dick Caldwell, the NAS Laboratory Manager, will be kept informed of the progress of the project, and will review and sign all final reports before they are sent to Windward.

Gerald Irissarri at NAS will be the PM responsible for overall project coordination, planning oversight, and production of all project deliverables. As the PM for the required tests, he is responsible for all aspects of the freshwater sediment toxicity testing, including receipt of sediment samples, chain-of-custody procedures, storage of sediment samples prior to testing, test initiation and termination, and production of all project deliverables. The laboratory PM can be reached as follows:

Gerald Irissarri
P.O. Box 1437
Newport, OR 97365
Telephone: 541.265.7225
Email: girissarri@nwaquatic.com

Michele Redmond will be the Data Manager at NAS. Her responsibilities will include the compilation, formatting, and summarizing of all bioassay data. The Data Manager can be reached as follows:

Michele Redmond
P.O. Box 1437
Newport, OR 97365
Telephone: 541.265.7225
Email: mredmond@nwaquatic.com

Linda Nemeth will be the QA/QC Officer at NAS. She will be responsible for the QA/QC review of the bioassay data. She can be reached as follows:

Linda Nemeth
P.O. Box 1437
Newport, OR 97365
Telephone: 541.265.7225
Email: lnemeth@inetw.net

The bioassay laboratory will accomplish the following:

- adhere to the methods outlined in this QAPP, including those methods referenced for each toxicity test procedure
- adhere to documentation, custody, and sample logbook procedures
- implement QA/QC procedures defined in this QAPP
- meet all reporting requirements
- deliver electronic data files as specified in this QAPP
- meet turnaround times for deliverables as described in the QAPP
- allow EPA and the QA/QC contractor to perform laboratory and data audits

A4.2.5 Data Management

Shawn Hinz will oversee data management to ensure that toxicity data are incorporated into the LWG database with appropriate qualifiers following acceptance of the data validation. QA/QC of the database entries will ensure accuracy for future use.

A5 PROBLEM DEFINITION/BACKGROUND

The Programmatic Work Plan (Integral 2004) identified the need for additional co-located toxicity tests and sediment chemistry analysis to support the development of a predictive model(s) that characterizes the relationship between sediment chemistry and benthic invertebrate toxicity at the Portland Harbor Superfund site. The data will also be used to confirm toxicity in high priority areas and provide information on the nature and extent of sediment toxicity to benthic invertebrates in the ISA. This appendix presents the sediment toxicity test procedures. The sediment chemistry analysis is covered in the sediment QAPP.

A6 TASK DESCRIPTION

Two sediment toxicity tests will be conducted on each of the approximately 200 sediment samples to be collected in the lower Willamette River (the exact number of samples will be determined in the FSP): 1) the chronic 28-day freshwater amphipod (*Hyalella azteca*) test, and 2) the acute 10-day freshwater midge (*Chironomus tentans*) test. The test procedures are based on the American Society for Testing and Materials (ASTM) Method E 1706-00 (ASTM 2003) and EPA 600/R-99/064 (EPA 2000). The test conditions, endpoints, and test acceptability criteria are summarized in Table B1-1 and B2-2. For additional information on test procedures see Attachments 2 (*H. azteca*) and 3 (*C. tentans*).

A7 QUALITY OBJECTIVES AND CRITERIA

The overall data quality objective (DQO) for the toxicity testing is to produce data that meet EPA’s acceptability criteria for the 28-day chronic *H. azteca* and the 10-day acute *C. tentans* sediment toxicity tests. To ensure that the toxicity laboratory produces acceptable data a performance-based criteria approach will be used (see Table B4-1 and B4-2). The toxicity data will be generated to address the objectives listed in Section B1.

A8 SPECIAL TRAINING/CERTIFICATION

No special training requirements are necessary for NAS personnel to conduct the toxicity tests as described in Section B. Field sampling requirements are presented in the main text.

A9 DOCUMENTATION AND RECORDS

This section describes documentation and records kept during laboratory toxicity testing. In addition, the data reduction process and contents of the data report are described.

A9.1 Field observations

See main text (Section A9.1).

A9.2 Laboratory records

The laboratory will be responsible for internal checks on sample handling and toxicity data reporting and will correct errors. Close contact will be maintained with the laboratory to resolve any QC problems in a timely manner. The laboratory data package will include the following:

- **Project narrative**: This summary, in the form of a cover letter, will present any problems encountered during any aspect of the toxicity testing. The summary will include, but not be limited to, discussion of quality control, sample shipment, sample storage, and testing difficulties. Any problems encountered, actual or perceived, and their resolutions, will be documented in as much detail as necessary.
- **Records**: Legible copies of the chain-of-custody forms will be provided as part of the data package. This documentation will include the time of receipt and the condition of each sample received by the laboratory. Additional internal tracking of sample custody by the laboratory will also be documented.
- **Testing results**: The electronic data package summarizes all the raw test results and calculated survival and biomass endpoints for each toxicity test. The summary will include the following information:
    - test initiation and termination dates
    - field sample identification code and the corresponding laboratory identification code for each test sediment
    - test acceptability (based on the negative control)
    - all replicate survival and biomass data for each test sediment
    - average percent survival of each test sediment
    - average individual biomass of each test sediment
    - water quality measurements

The laboratory will submit the electronic data in Excel format. Examples of the electronic data files are presented in Attachment 4.

- **QA/QC summary:** The summary will contain the results of the QA/QC procedures. The required summaries are listed below; additional information may be requested.
    - percent survival and dry weight of negative control (*H. azteca* test)
    - percent survival and ash-free dry weight of negative control (*C. tentans* test)

    - result from the 96-h positive control test
    - control chart of the positive control
    - water quality measurements
- **Original data:** Photocopies of all the raw data generated by the laboratory will be provided, including the following:
    - survival and weight data for each replicate of the negative control
    - survival and weight data for each replicate of each test sediment
    - positive control
    - water quality measurements

A9.3 Data reduction

Data reduction is the process by which raw data are converted or reduced to a specified format to facilitate analysis of the data. The laboratory will perform the first data reduction by calculating average survival and biomass for each test sediment and the negative controls. An internal review of the data will be performed by the NAS QA/QC officer. For the external review process the laboratory will provide both the reduced and raw data. The data will be generated in a form amenable to review and evaluation. The raw (replicate) and reduced data will be reviewed and validated by Dinnel Marine Resources and reviewed by the Project QA/QC manager.

A9.4 Data report

A data report will be prepared documenting all activities associated with the collection, handling, and analysis of samples. At a minimum, the following will be included in the data report:

- brief review of the study design and methods
- data tables summarizing the testing
- deviations from the protocols appended to the approved QAPP
- copies of chain-of-custody forms (appendix)
- data validation report (appendix)
- tables of all raw data (appendix)

Dinnel Marine Resources will compile the batches (see Section B2) in groups of three and provide a validation report to Windward three weeks after receiving the last data package from the laboratory. A final QA report will be submitted to EPA 90 days after completing validation of the last data package. The final QA report will include all laboratory and data audits. A draft data report will be provided to EPA in an electronic format 60 days following submittal of the final QA report to EPA. The draft data report will include all reports submitted by Dinnel Marine resources. A draft benthic interpretation report will be submitted to EPA 180 days following submittal of the final QA report.

Section B Data Generation and Acquisition

This section describes the methods that will be used to conduct the toxicity tests. Elements include experimental design, sample handling and custody requirements, toxicity test method requirements, quality control, instrument/equipment testing, inspection and maintenance, instrument calibration, supply inspection/acceptance, and data management.

B1 EXPERIMENTAL DESIGN

The objectives of the benthic toxicity testing are to:

- provide co-located sediment chemistry and toxicity data for the development of a predictive model(s) that characterize the relationship between sediment chemistry and benthic invertebrate toxicity
- confirm toxicity in high priority areas
- determine toxicity where the physical environment or the form of the chemical may modify toxicity

Toxicity testing and chemistry analysis will be performed on a total of approximately 200 co-located sediment samples to address these objectives (see the Field Sampling Plan for station locations). The exact number will be determined in the FSP. Two sediment toxicity tests will be conducted 1) the 28-day chronic test with *H. azteca* and 2) the 10-day acute test with *C. tentans*. The test conditions, endpoints, and test acceptability criteria are summarized in Table B1-1 and B2-2. Reference sediment will not be included in the experimental design. Hence, the negative control sediment will be used in interpreting toxicity responses in test sediments. For detailed information on the experimental design, test and QA procedures see Attachment 1, 2 and 3. The initial "hit/no hit" designation will be based on statistically significant differences from negative control. The toxicity evaluation process will be decided upon in future meetings between EPA and LWG. Further discussion of the evaluation process of the toxicity test results is presented in the technical memorandum "Estimating risks to the benthic community using sediment toxicity tests" submitted to EPA May 28, 2004.

Table B1-1. Summary of test conditions for the 28-day chronic *H. azteca* sediment toxicity test

Test Conditions for the 28-day Chronic *H. azteca* Sediment Toxicity Test	
1. Test type	whole sediment toxicity test with renewal of overlying water
2. Test duration	28 days
3. Temperature	23 ± 1°C
4. Light quality	daylight fluorescent light
5. Illuminance	100-1,000 lux

Test Conditions for the 28-day Chronic *H. azteca* Sediment Toxicity Test	
6. Photoperiod	16L:8D
7. Test chamber size	300-mL high-form lipless beakers, (Pyrex® 1040 or equivalent)
8. Sediment volume	100 mL
9. Overlying water volume	175 mL
10. Renewal overlying water	2 volume additions/day (static renewal)
11. Age of test organisms	7-8 days old at test initiation
12. Organisms per test chamber	10
13. Replicates per treatment	8
14. Organisms per treatment	80
15. Feeding regime	YCT food, fed 1.0 mL daily/chamber
16. Cleaning	if screens are used, clean as needed
17. Aeration	None, unless DO falls below 2.5 mg/L
18. Overlying (test) water	Dechlorinated city water with hardness adjusted to 30 mg/L
19. Overlying water quality	Hardness, alkalinity, conductivity, ammonia-N beginning and end; temperature daily; conductivity weekly; DO & pH 3X/wk
20. Endpoints	Survival & growth (based on dry weight)
21. Test acceptability criteria	Minimum control survival of 80%
22. Sample holding	≤ 8 weeks at 4°C in the dark, preferable 2 weeks
23. Sample volume required	1L (800 mL per sediment)
24. Reference toxicant	Concurrent testing required with cadmium as toxicant

Table B2-2. Summary of conditions for the 10-day acute *C. tentans* sediment toxicity test

Test Conditions for the 10-day Acute *C. tentans* Sediment Toxicity Test	
1. Test type	whole sediment toxicity test with renewal of overlying water
2. Test duration	10 days
3. Temperature	23 ± 1°C
4. Light quality	daylight fluorescent light
5. Illuminance	100-1,000 lux
6. Photoperiod	16L:8D
7. Test chamber size	300-mL high-form lipless beakers (Pyrex® 1040 or equivalent)
8. Sediment volume	100 mL
9. Overlying water volume	175 mL
10. Renewal overlying water	2 volume additions/day (static renewal)
11. Age of test organisms	2nd to 3rd instar or younger larvae (≥ 50% of organisms must be 3rd instar)
12. Organisms per test chamber	10
13. Replicates per treatment	8
14. Organisms per treatment	80
15. Feeding regime	Fish food flakes, fed 1.5 mL chamber (1.5 mL contains 6.0 mg of dry solids) daily on days 0 - 9.
16. Aeration	None, unless DO falls below 2.5 mg/L.

Test Conditions for the 10-day Acute *C. tentans* Sediment Toxicity Test	
17. Overlying (test) water	Dechlorinated city water with hardness adjusted as close to 30 mg/L as the organisms can tolerate
18. Overlying water quality	Hardness, alkalinity, conductivity, pH, ammonia-N beginning and end; temperature and DO daily
19. Endpoints	Survival and growth (based on ash-free dry weight)
20. Test acceptability criteria	Minimum control survival of 70%; mean weight of surviving control organisms 0.48 mg AFDW
21. Sample holding	≤ 8 weeks at 4°C in the dark, preferable 2 weeks
22. Sample volume required	1L (800 mL per sediment)
23. Reference toxicant	Concurrent testing required with potassium chloride as toxicant

B2 SAMPLING METHODS

It is anticipated that the sediment samples to be used for toxicity testing will be collected weekly in batches of 15 – 20 for a total of approximately 200 samples. The exact number will be determined in the FSP. A portion of the homogenized sediment collected at each station will be placed into I-Chem 1L high-density polyethylene wide-mouth jars. The required volume per station is 4L (1L for each test and sufficient sediment to perform re-testing if needed). For further details on sediment sampling methods see the main text (Section A6.1.1).

The sediment samples will be transported to NAS and the toxicity tests will be initiated on each batch within 8 weeks, preferably 2 weeks, after arrival. Field documentation is addressed in the main text.

B3 SAMPLE HANDLING AND CUSTODY

Sample custody is a critical aspect of environmental investigations. Sample possession and handling must be traceable from the time of sample collection, through the laboratory and toxicity testing, to the generation of sample results. This section describes the minimum project requirements for sample handling and custody procedures.

B3.1 Sample custody procedures

Samples are considered to be in custody if they are: 1) in the custodian’s possession or view, 2) retained in a secured place (under lock) with restricted access, or 3) placed in a container and secured with an official seal(s) such that the sample cannot be reached without breaking the seal(s). Custody procedures will be used for all samples throughout the collection, transport, and testing process, and for all data and data documentation whether in hard copy or electronic format. Custody procedures will be initiated during sample collection. A chain-of-custody form will accompany samples to the toxicity laboratory. Each person who has custody of the samples will sign the chain-of-custody form and ensure that the samples are not left unattended unless properly secured. Minimum documentation of sample handling and custody will include:

- sample location, project name, and unique sample number
- sample collection date and time
- any special notations on sample characteristics or problems
- initials of the person collecting the sample
- date sample was sent to the laboratory

The sample coolers containing samples for toxicity testing will be hand-carried to NAS. The Laboratory PM will ensure that chain-of-custody forms are properly signed upon receipt of the samples and will note questions or observations concerning sample integrity on the chain-of-custody forms. The laboratory will contact the FC and LC immediately if discrepancies are discovered between the chain-of-custody forms and the sample shipment upon receipt. The temperature inside the cooler(s) will be checked upon receipt of the samples. The Laboratory PM will specifically note any coolers that do not contain ice packs or that are not sufficiently cold (4 ± 2°C) upon receipt. Each sample will be assigned a unique laboratory number, and samples will be grouped in appropriate sample delivery groups. The laboratory will not dispose of the sediment samples for this project until notified in writing by the LC. If this notice is not received within three months of the end of the last test, the laboratory will dispose of sediments or return them to Windward in order to clear space.

All samples will be handled so as to prevent contamination or loss of any sample. Samples will be assigned a specific storage area within the laboratory and will be kept there until tested.

The Laboratory PM will ensure that a sample-tracking record follows each sample through all stages of laboratory processing. The sample-tracking record must contain, at a minimum, dates of test initiation and test method.

The tracking and custody procedures for the field collection of the sediment samples are presented in the main text (see Section B3).

B4 TOXICITY TEST METHOD REQUIREMENTS

The method requirements and acceptability criteria (EPA 2000, ASTM 2003) for the two sediment toxicity tests are summarized in Table B4-1 and B4-2.

Table B4-1. Summary of test acceptability requirements for the 28-day chronic *H. azteca* sediment toxicity test

Testing Requirements for the 28-day Chronic *H. azteca* Sediment Toxicity Test	
1.	Age of *H. azteca* at test initiation should be 7 – 8 days old
2.	Average survival of *H. azteca* in the negative control sediment should be greater than or equal to 80%
3.	All organisms in a test must be from the same source
4.	Negative-control sediment must be included in a test
5.	Test organisms must be cultured at 23°C (± 3°C) and tested at 23°C (± 1°C)
6.	The mean of the daily test temperature must be within ± 1°C of 23°C. The instantaneous temperature must always be within ± 3°C of 23°C
7.	All test chambers should be identical and should contain the same amount of sediment and overlying water
8.	Hardness, alkalinity, and ammonia in the overlying water typically should not vary more than 50% during the sediment exposure, and dissolved oxygen should be maintained above 2.5 mg/L in the overlying water
9.	Storage of sediment collected in the field should less than or equal to 8 weeks, preferable less than or equal to 14 days
10.	Natural physico-chemical characteristics of sediment collected from the field should be within tolerance limits of the test organisms

Table B4-2. Summary of test acceptability requirements for the 10-day acute C*. tentans* sediment toxicity test

Testing Requirements for the 10-day Acute *C. tentans* Sediment Toxicity Test	
1.	Test must start with second- to third-instar larvae
2.	Average survival of *C. tentans* in the negative control sediment must be greater than or equal to 70% at the end of the test
3.	Average size of *C. tentans* in the negative control must be at least 0.48 mg AFDW at the end of the test
4.	All organisms in a test must be from the same source
5.	Negative-control sediment must be included in a test
6.	Test organisms must be cultured at 23°C (± 3°C) and tested at 23°C (± 1°C)
7.	The mean of the daily test temperature must be within ± 1°C of 23°C. The instantaneous temperature must always be within ± 3°C of 23°C
7.	All test chambers should be identical and should contain the same amount of sediment and overlying water
8.	Hardness, alkalinity, and ammonia in the overlying water typically should not vary more than 50% during the sediment exposure, and dissolved oxygen should be maintained above 2.5 mg/L in the overlying water
9.	Storage of sediment collected in the field should less than or equal to 8 weeks, preferable less than or equal to 14 days
10.	Natural physico-chemical characteristics of sediment collected from the field should be within tolerance limits of the test organisms

B5 LABORATORY QUALITY ASSURANCE/QUALITY CONTROL

Both sediment toxicity tests will incorporate standard QA/QC procedures for evaluation of the validity of the test results. Standard QA/QC procedures include the use of negative and positive controls and measurement of water quality during testing.

The negative control sediment is collected at Beaver Creek, Lincoln County. The physical and chemical characteristics of this sediment will be determined prior to test initiation. Cadmium is used as the positive control toxicant in the *H. azteca* test and potassium chloride in the *C. tentans* test. The positive control is used to establish the relative sensitivity of the test organism and is a 96-h test conducted without sediment. Quality control is maintained through control charting of the positive control tests. Corrective action will be taken if the positive control endpoint value falls outside ± 2 standard deviations of the cumulative mean endpoint. To ensure proper water quality conditions several parameters are measured in the overlying water throughout the test. For the 28-day *H. azteca* test total ammonia, hardness, and alkalinity are measured at test initiation and termination. Conductivity is measured weekly. Dissolved oxygen and pH are measured three times a week, and temperature is measured daily. For the 10-day *C. tentans* test total ammonia, conductivity, pH, hardness, and alkalinity are measured at test initiation and termination. Dissolved oxygen and temperature are measured daily. The water quality control limits are summarized in Table B4-1 and B4-2.

The balance used for the weight determination is an electronic microbalance Sartorius Model M3P. The balance is calibrated before each use with certified weights. The balance is serviced and calibrated every year and the weights are calibrated every year. The test weighing procedures are described in details in Attachment 1, 2 and 3.

B6 INSTRUMENT CALIBRATION AND FREQUENCY

Calibration will be performed on each instrument throughout the project. Dissolved oxygen is measured with a polarographic oxygen probe and is calibrated daily according to the manufacturer's recommendations. The pH is measured with a pH probe and meter with scale divisions of 0.1 pH units. The pH meter and probe are calibrated daily. The conductivity meter is calibrated monthly and the thermometer is calibrated yearly. Hardness, alkalinity, and total ammonia-N are measured using commercially available kits.

B8 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

Supplies and consumables for the laboratory testing effort will be inspected upon delivery and accepted if the conditions of the supplies are satisfactory.

B9 DATA MANAGEMENT

The laboratory is expected to submit data in electronic format as described in Section A9.4 and Attachment 4. The Laboratory PM should contact the Project QA/QC Coordinator prior to data delivery. The data will be submitted directly to Dinnel Marine Resources for validation with a concurrent submittal to Windward.

The bioassay electronic data will be formatted and QA/QC'd before importing into the Windward Bioassay Database. The Windward database uses a series of stored routines to verify the integrity of the bioassay data prior to calculating test responses and endpoints.

Section C Assessment and Oversight

C1 COMPLIANCE ASSESSMENTS AND RESPONSE ACTIONS

C1.1 Compliance assessments

Laboratory performance assessments consist of on-site reviews designated by EPA of QA systems and equipment for calibration and measurement. EPA personnel may conduct a laboratory audit prior to toxicity testing. Third-party audit(s) by Dinnel Marine Resources will be performed while the laboratory is conducting the toxicity testing. Any pertinent laboratory audit reports will be made available to the Project QA/QC Coordinator upon request. The laboratory is required to have written procedures addressing internal QA/QC; these procedures will be submitted for review by the Project QA/QC Coordinator to ensure compliance with the QAPP. The laboratory and the QA/QC Coordinator are required to ensure that all personnel engaged in the toxicity testing have appropriate training.

C1.2 Corrective action for laboratory testing

The laboratory is required to comply with the standard operating procedures previously submitted to the Project QA/QC Coordinator. The Laboratory PM will be responsible for ensuring that appropriate corrective actions are initiated as required for conformance with this QAPP. All laboratory personnel will be responsible for reporting problems that may compromise the quality of the data.

The Project QA/QC Coordinator will be notified immediately if any problem arises that may affect the acceptability of the toxicity test. The Laboratory PM will document the corrective action taken in a memorandum submitted to the Project QA/QC Coordinator within 5 days of the initial notification. A narrative describing the problem and the steps taken to identify and correct it will be submitted with the data package.

C2 REPORTS TO MANAGEMENT

At the end of every month during which bioassay results are received, Windward will prepare and deliver a status report that includes:

- inventory and status of samples held at the laboratory
- descriptions and justification for any significant changes in QA/QC procedures
- any changes to or deviations from SOPs
- any changes in lab procedures that could affect data quality

- summary of project-related communications regarding sample handling and analysis.

Intermittent or otherwise unscheduled status reports may be required on an as needed basis. A data report will be written after validated data are available for the last toxicity test, as described in Section A9.4.

Section D Data Validation and Usability

D1 DATA REVIEW, VALIDATION, AND VERIFICATION REQUIREMENTS

Data are not considered final until validated. The validation reports and the final QA report submitted by Dinnel Marine Resources will be included as an appendix to the data report (see Section A9.4 and D2).

D2 DATA REVIEW AND VALIDATION

Data validation is a process in which data are reviewed and evaluated by the laboratory QA/QC officer. The laboratory technicians performing the tests are responsible for ensuring that the appropriate procedures have been followed during the testing. The Project QA/QC Coordinator is responsible for ensuring that all testing performed by the laboratory meets the test acceptability criteria, is properly documented, and complete, and that they satisfy the project DQOs specified in this QAPP.

Independent third-party data review and validation of the sediment toxicity data will be conducted by Paul Dinnel of Dinnel Marine Resources. Tasks to be conducted by the third-party reviewer include:

- ensuring that all laboratory test protocols are up to date and include any modifications to the protocol that have recently been published. Troubleshoot any questions or problems as they may arise.
- conducting test audits during sediment testing. This may involve several audit visits if tests are run in multiple batches. Identify any problems or deviations from established protocols or SOPs.
- reviewing all toxicity and QA data from the testing laboratory for each batch of tests. Note any data gaps or items that were out of compliance with the bioassay protocols. Where appropriate, provide guidance regarding the severity of any out-of-compliance items. Recommend retesting where necessary. Prepare QA reports for the testing laboratory for each bioassay, and a Final QA Report to Windward covering all laboratory and data audits.

All discrepancies and requests for additional information will be discussed with the laboratory prior to issuing the formal data validation report. All contacts with the laboratory will be documented in a communication report. Procedures used during data review and findings made during data validation will be documented on worksheets. A validation report will be prepared for each toxicity test; that report will summarize QC results and possible data limitations. All data will be accessible, but only validated data (including replicate and reduced data) will be released for general use.

D3 RECONCILIATION WITH DATA QUALITY OBJECTIVES

Data quality will be assessed by the Project QA/QC coordinator in consultation with EPA guidelines. The results of the third-party independent review and validation will be reviewed and cases where project DQOs were not met will be identified. The usability of the data will be determined, as will the importance of the data with respect to decision-making for the project. The data report will summarize the data quality with respect to project DQOs.

Section E References

ASTM. 2003. Standard test methods for measuring the toxicity of sediment-associated contaminants with fresh water invertebrates. ASTM Standard Method No. E 1706-00. ASTM annual book of standards volume 11.05, American Society for Testing and Materials, West Conshohocken, PA.

EPA. 2000. Methods for measuring the toxicity and bioaccumulation of sediment-associated contaminants with freshwater invertebrates. Second edition. EPA 600/R-99/064.
US Environmental Protection Agency, Duluth, MN.

EPA. 2002. Guidance for quality assurance project plans EPA QA/G-5. U.S. Environmental Protection Agency Office of Environmental Information, Washington, DC.

Integral. 2004. Portland Harbor RI/FS programmatic work plan. Revised. Prepared for Lower Willamette Group. Integral Consulting, Inc., Mercer Island, WA.

USACE. 1998. Biostat 2.0 users guide. Review draft. Seattle District, US Army Corps of Engineers, Seattle, WA.

Attachment #1 Laboratory QA Plan

QUALITY ASSURANCE/QUALITY CONTROL MANUAL

FOR

AQUATIC TOXICOLOGY STUDIES

At

NORTHWESTERN AQUATIC SCIENCES
3814 YAQUINA BAY ROAD
P.O. BOX 1437
NEWPORT, OR 97365

January 7, 1992
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Revision 2: October 6, 1997
Revision 3: March 30, 2004

APPROVAL FOR IMPLEMENTATION:

Richard S. Caldwell, Ph.D. Date
Laboratory Director, Northwestern Aquatic Sciences, and
Technical Director, Aquatic Toxicology Laboratory

Linda K. Nemeth, M.B.A. Date
Quality Assurance Officer

I. POLICY STATEMENT

This manual summarizes the policies and operational procedures associated with Northwestern Aquatic Sciences and provides a detailed description of quality assurance and quality control (QA/QC) procedures for toxicity testing with marine and freshwater organisms at Northwestern Aquatic Sciences (NAS) in Newport, Oregon. These procedures address all aspects of toxicity testing that can potentially affect data quality and interpretation, including sample handling and storage of test materials, collection and conditioning of test organisms, test conditions and equipment, calibration of instruments, replication, reference toxicants, corrective action, record keeping and reporting, and data reduction. Further details on these policies and procedures are contained in SOPs, test protocols, and related documents.

NAS performs toxicity testing of environmental samples including marine and freshwater sediments, sediment pore waters, sediment elutriates, effluents, other products (chemical products, ores, mine tailings), and receiving waters. Test methods may be acute, chronic, or bioaccumulation methods all of which are based on published test methods.

NAS is accredited by the State of Washington Department of Ecology for bioassay (toxicity) testing. As part of this accreditation, NAS analyzes proficiency testing samples once a year from the DMR QA Program. NAS also maintains internal QC with the use of reference toxicant testing for every type of test and the use of control charts for references toxicant tests.

The technical and service requirements of all requests to provide testing are thoroughly evaluated by the Laboratory Director/QAO and Project Managers before commitments are made to accept the work. This includes a review of facilities and instrumentation, staffing, and any special QC or reporting requirements to ensure that testing can be performed within the expected schedule. All test methods are performed based on published reference methods such as U.S. Environmental Protection Agency (EPA), American Society for Testing Materials (ASTM), Environment Canada, and other officially published test methods or methods developed by NAS to meet specific client needs, such as for research purposes.

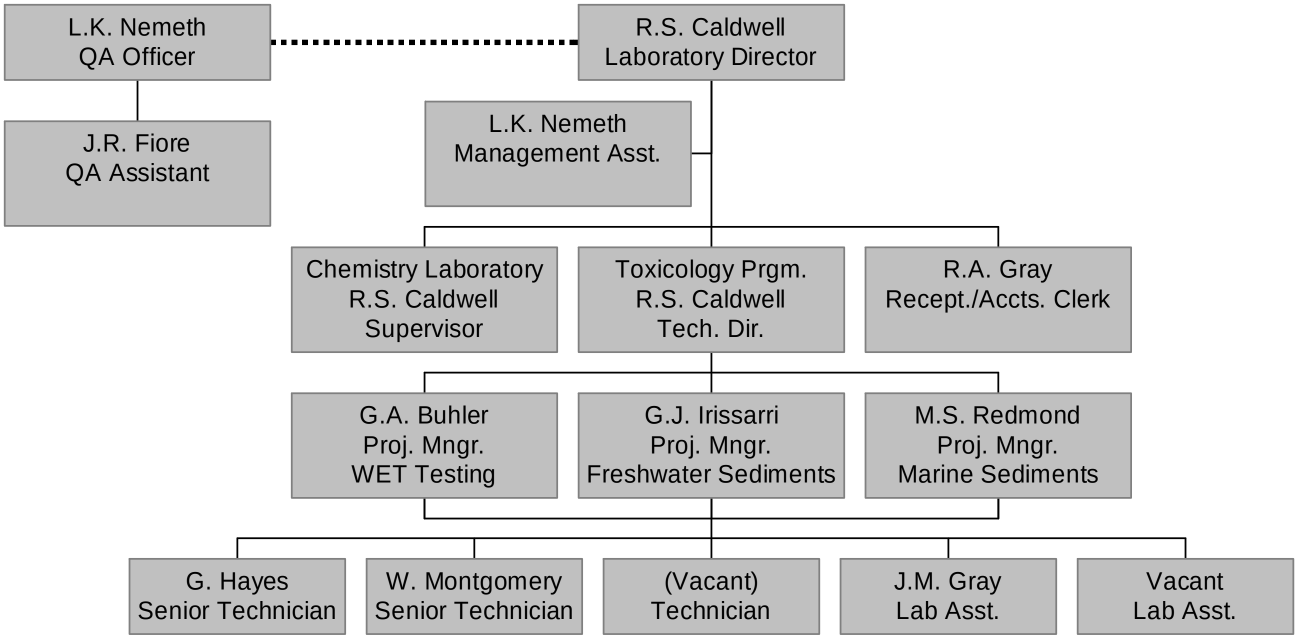
II. ORGANIZATION AND RESPONSIBILITIES

Northwestern Aquatic Sciences is an independent testing laboratory that provides aquatic toxicology testing throughout the United States and internationally, with emphasis on the Pacific Northwest. The overall operation is currently comprised of the aquatic toxicology laboratory with a chemistry support laboratory that is not an accredited entity at this time. There is an independent formal Quality Assurance program headed by the QA Officer. There is a small clerical staff that supports the laboratory program.

An organization chart for Northwestern Aquatic Sciences is shown below. The chart includes all individuals discussed below. Job descriptions for al individuals are maintained by the Management Assistant.

A. ORGANIZATION CHART

NAS Organizational Chart
February 2004



B. LABORATORY DIRECTOR

The Laboratory Director is responsible for ensuring that:

- The responsibility, authority and interrelationships of all personnel who manage, perform, or verify work affecting the quality of tests are specified and documented, including a clear description of the lines of responsibility in the laboratory to ensure adequate supervision, and job descriptions for all positions.
- The minimum level of basic laboratory and other skills, education, and experience necessary for all laboratory positions are defined.
- All technical laboratory staff has demonstrated proficiency in the activities for which they are responsible.
- Training of personnel is kept up to date.
- All analytical and operational activities are documented.
- All personnel are adequately supervised.
- All sample acceptance criteria are verified, and samples are logged into the sample tracking system and properly labeled and stored.
- An annual management review of the quality system is performed.
- The quality of all data reported by the laboratory is documented.
- A proactive program for prevention and detection of improper, unethical, or illegal actions is in place.

- The laboratory has the appropriate resources and facilities to perform requested work.
- Corrective actions relating to findings from internal audits are completed.
- Appropriate persons-in-charge are designated in the absence of the Technical Director (Laboratory Supervisor) and QA officer.
- Supplies and any outside support services are of adequate quality to sustain confidence in the laboratory's tests.

C. QUALITY ASSURANCE (QA) OFFICER

As shown in the organization chart, the QA Officer is independent of direct job involvement and day-to-day operations, and has direct access to the Laboratory Director. The QA Officer:

- Remains independent from laboratory operations where QA oversight is provided.
- Evaluates data objectively and performs assessments independent of managerial influence.
- Is responsible for the quality system and its implementation.
- Serves as the focal point for QA/QC.
- Develops and maintains procedures for audits and data review.
- Maintains the quality manuals and keeps them current.
- Is responsible for the oversight and/or review of quality control data.
- Arranges for or conducts internal audits on the entire technical operation annually.
- Notifies laboratory management of deficiencies in the quality system.
- Monitors corrective actions.
- Ensures that the laboratory performs proficiency testing as required for certifications.
- Supervises assistants in the QA Unit.

D. TECHNICAL DIRECTOR

The Technical Director for the Aquatic Toxicology Program reports to the Laboratory Director and is responsible for:

- Certifying that personnel with appropriate educational and/or technical background perform all tests for which the laboratory is accredited
- Monitoring standards of performance in quality control and quality assurance;
- Monitoring the validity of the analyses performed and data generated in the laboratory to assure reliable data;
- Ensuring that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory; and
- Providing educational direction to laboratory staff.

E. TECHNICAL STAFF

Technical staff is responsible for sample analysis and identification of corrective actions. The staff report directly to the Laboratory Director. All personnel are responsible for complying with all quality assurance/quality control (QA/QC) requirements that pertain to their organizational/technical function. As documented in the employee records, each technical staff member has the experience and education to adequately demonstrate knowledge of their particular function and a general knowledge of laboratory operations, analytical test methods, quality assurance/quality control procedures and records management.

Within the Toxicology Program, each individual project is managed by a specific Project Manager (PM), who specializes in work for that type of project. There are three PMs: WET Testing Project Manager, Freshwater Sediment Project Manager, and Marine Sediment Project Manager. Each project manager is responsible for the overall scheduling of the entire project, with each type of test assigned to one person to oversee as the Study Director. The Study Director supervises the conduct of the study or test, analyzes the data, and writes the report and then gives it to the QA unit for data verification and review. After corrections and QA approval, the report goes to the Project Manager for review, revision, and approval. The final step is for the report to go to the Laboratory Director for final review and approval.

F. TRAINING

Each employee has read, understood, and is using the latest version of the laboratory's SOPs, which relates to his/her job responsibilities. Current test method SOPs are signed and dated by the responsible employees on the SOP signature page. When test method SOPs are revised, the same procedure is followed. Most training with toxicology testing is conducted as hands on training with the person being trained working side-by-side with an experienced person. Additional training in the form of reading assignments, seminars, and workshops are also provided as appropriate. Training records (e.g., continuing education, participation in technical conferences, internal training activities) are kept with Human Resource files.

III. SAMPLING, HANDLING AND STORAGE OF TEST MATERIALS

Test materials may include, but are not limited to, marine and freshwater sediments, sediment pore waters, sediment elutriates, effluents, other products (chemical products, ores, mine tailings), and receiving waters. General guidelines are given below for sampling and handling of test materials. SOPs and protocols should be consulted for more specific information regarding collection, volume requirements, container specifications, preservation techniques, and storage conditions.

A. SAMPLING

Sampling for most projects at NAS is performed by the client and shipped to NAS. Sampling requirements will be specific to the types of test materials and the specific study design in effect. Effluents may be grab or 24-hr composite samples and should be taken from the sampling point specified in the NPDES discharge permit if applicable. Grab samples are commonly taken for receiving waters. Sediment samples may be taken using various coring devices or surface samplers such as a Smith-McIntyre grab. Sediment sampling locations are specified in a written sampling plan.

Sampling implements should be made of non-contaminating materials (e.g. stainless steel, plastic, etc.) and properly cleaned for the type of analyses to be performed. Sample containers may be glass or plastic (polyethylene or polypropylene) bottles, or disposable cubitainers. Samples should completely fill the storage container leaving no air space.

All samples should have the following information recorded on the sample container label: sample location, test to be performed, date and time of collection, initials of persons collecting the sample, and special remarks if appropriate. Since most samples are not collected by NAS personnel, this is not under NAS control most of the time. At a minimum, the sample container usually has a sample number, date and time of collection, and initials of persons collecting the sample.

When sampling involves a large number of samples and/or detailed records are required by the sampling plan, a field notebook is used in which are recorded the dates, locations, and procedures used for collecting samples as well as any other necessary data. The notebook should be hardbound and all entries are made in waterproof ink.

B. SHIPPING

Shipping is normally by overnight express service (e.g. Federal Express, UPS, Greyhound) and should be scheduled to avoid weekend delivery when possible. Although Saturday delivery by UPS is available, other weekend delivery is not readily available in Newport. Under certain circumstances, however, special arrangements can be made. Shipping containers must be strong and insulated (e.g. plastic ice chests, styrofoam-insulated corrugated boxes). Samples are shipped on ice (cold packs). Samples should be shipped under chain-of-custody.

C. HANDLING, PRESERVATION AND STORAGE

Upon receipt at the laboratory, samples are immediately logged in by the designated sample custodian (See Section III.C.) Each sample is given a unique sample number with numbers listed consecutively in the sample logbook. The logbook includes all laboratory samples including those not a part of the Aquatic Toxicology Program. Data logged at the time of receipt include the sample number, the sample description, the analysis required, the date and time of collection, the initials of the person collecting the sample, the date and time of receipt at the laboratory, and the initials of the person logging the sample. Upon completion of the analysis, the date and time of analysis, initials of the analyst, sample disposition, date of disposition and initials of the person responsible for the ultimate sample disposition are recorded in the logbook. Samples are stored in designated refrigerators or in a locked refrigerated (4°C) cold-room. Sediment samples may be stored under nitrogen if required for prolonged storage.

D. HOLDING TIMES

Samples for bioassays are used within prescribed time limits from the date of collection depending on the type of sample and the regulations governing that particular study (e.g. Puget Sound Estuary Program, California State Water Resources Control Board, Washington Department of Ecology, Oregon Department of Environmental Quality, U.S. Army Corps of Engineers, U.S. Environmental Protection Agency, etc.). In general, effluents and receiving waters are tested within 36 hours of the completion of the collection period, and sediments are tested within 14 days of collection. Sediments for bioassays should be stored at 4°C, never frozen or dried. Sediments for certain types of bioassay testing (e.g., Army Corps of Engineers DMMO) may be stored under nitrogen for 6 to 8 weeks. Alternatively, sediments for some projects may be collected and put into containers with no headspace rather than under nitrogen. If the sample holding time for test materials for a project has been or is about to be exceeded, the client is contacted for instructions on whether to proceed. For some projects, the samples are already more than two weeks old.

E. SAMPLE ACCEPTANCE/REJECTION

Samples may be deemed unacceptable if: 1) the sample is received after the allowed holding time has elapsed (or so late that a test cannot be started within the time limits); 2) the sample is not properly labeled and identifiable; 3) the sample container arrived broken or the sample container has had its chain-of-custody seal broken. In the event that one of these events occurs, the client is contacted and informed that the sample is unacceptable and given the options of resampling and resubmitting a sample, having the test run with that sample but with a disclaimer attached for that sample, or dropping that sample from the analysis group.

IV. CHAIN-OF-CUSTODY PROCEDURES

All samples received by Northwestern Aquatic Sciences should be sent under chain-of-custody. A written chain-of custody procedure is available for use upon request. The purpose of this procedure is to maintain an accurate written record that can be used to trace the possession of the sample from the moment of its collection through its final analysis. In addition, this procedure insures that the samples are handled only by authorized and properly trained personnel.

A. GUIDELINES FOR SAMPLE COLLECTION

Each sample should be collected according to the established guidelines for the type of sample and the sampling location. Each sample must have a label attached to it containing the information specified in Section II.A. The sample collector is responsible for the care and custody of the samples until they are transferred to the appropriate laboratory or given to an assigned custodian.

B. TRANSFER OF CUSTODY

When turning over possession of samples, the transferor and the transferee sign, date, and record the time on the chain-of-custody sheet. This record sheet allows the transfer of a group of samples at a time. If the samples arrive at the laboratory when the designated personnel are not there to receive them, the samples must be put into a secure location and the transfer conducted when the appropriate personnel are present.

C. LABORATORY CUSTODY PROCEDURES

Samples are to be handled by a minimum number of people. For each project the designated sample custodian is the Project Manager (PM) assigned to that particular study. The PM may designate an alternate sample custodian to act in the PM's absence. Samples may be stored in designated refrigerators or stored in a locked cold-storage room at 4°C. Samples are discarded only under the direction of the Project Manager when it is certain that all tests have been properly performed and recorded.

V. DILUTION WATER

The dilution water used in toxicity tests will depend on the type of test, the objectives of the study, and logistical constraints. Individual test protocols and EPA manuals should be consulted for the proper dilution water to use and the proper pretreatment if any.

In general, marine tests employ Yaquina Bay seawater as the dilution water. Where salinity adjustment is called for, combinations of seawater, seawater brine and/or laboratory deionized water (Milli-Q) may be employed. In some instances artificial sea salts may also be used.

Most freshwater effluent testing is done with either receiving water collected upstream from the effluent outfall or synthetic water made from Milli-Q water and reagent grade chemicals.

In the case of sediment tests for discharge of dredge materials into disposal site waters, water from a reference sediment collection site may be used, depending on the objectives.

VI. TEST ORGANISMS

Test organisms that can be field collected by NAS include amphipods (*Rhepoxynius abronius* and *Eohaustorius estuarius*), clams (*Macoma nasuta*), worms (*Nephtyes caecoides*), shrimp (*Crangon franciscorum* and *Lissocrangon stylirostris*), Dungeness crab juveniles (*Cancer magister*), bay mussels (*Mytilus edulis*), and California or sea mussels (*Mytilus californianus*). Such organisms can be taxonomically identified from appropriate texts by staff members or taxonomists can be consulted at Oregon State University's Hatfield Marine Science Center in Newport, OR.

Many test organisms are purchased from commercial suppliers. Routinely purchased test organisms include blue mussel (*Mytilus* spp.), Pacific oyster (*Crassostrea gigas*), fathead minnow (*Pimephales promelas*), inland silverside (*Menidia beryllina*), sheepshead minnow (*Cyprinodon variegatus*), topsmelt (*Atherinops affinis*) mysid shrimp (*Mysidopsis bahia* and *Holmesimysis costata*), rainbow trout (*Oncorhynchus mykiss*), amphipods (*Ampelisca abdita*, *Leptocheirus plumulosus*, and *Hyalella azteca*), and juvenile polychaetes (*Neanthes* sp.).

NAS has adequate facilities for culturing both freshwater and marine organisms. Cultured species change in response to test needs. NAS currently maintains cultures of various organisms including the cladocerans *Daphnia magna*, *Daphnia pulex*, and *Ceriodaphnia dubia*, the freshwater amphipod, *Hyalella azteca* and the midge, *Chironomus tentans*.

A record is kept with information on the collection and care of test organisms. The location and date of each collection is recorded along with the species and number of organisms collected, field conditions (e.g., temperature and salinity), initials of persons collecting organisms, and any other pertinent information.

Organisms are transported to the laboratory and held in the laboratory under standard conditions as outlined in the SOPs for collections. Water quality data is kept on separate bench sheets. These data sheets are regularly transferred to a binder for all water quality data on organisms. Organisms are fed according to SOPs and protocols.

VII. FACILITIES, EQUIPMENT, AND ENVIRONMENTAL CONTROL

A. NEWPORT AQUATIC TOXICOLOGY LABORATORY

Northwestern Aquatic Sciences operates aquatic toxicology laboratory facilities in Newport, OR. The facilities include over 5000 ft^2 of offices, wet and dry biological laboratories, refrigerated and frozen storage, an analytical laboratory equipped for GC/MS and other analyses, and storage areas located on a 1.2-acre site adjacent to Yaquina Bay. NAS' Yaquina Bay location gives it direct access to high quality seawater, which may be filtered and UV sterilized as required. The laboratory is also supplied with oil-free compressed air, dechlorinated fresh water, and Milli-Q^{TM} deionized water. All bioassay water is supplied to the laboratory through non-toxic PVC or polyethylene pipe. Temperature-controlled rooms or fiberglass-lined water tables are available for organism maintenance or for the constant temperature control of test containers. Adequate electrical power, circulating water pumps, and heating and refrigeration equipment is available for maximum flexibility and support of aquatic studies.

Test vessels include glass aquaria ranging in size from 3.5 to 60 liters and borosilicate glass beakers ranging in size from 30 ml to 1000 ml. Special glassware is available for flow-through bioassays. Adequate electrical power, circulating water pumps, and heating and refrigeration equipment is available for maximum flexibility and support of aquatic studies and toxicity tests. Continuous flow Mount-Brungs proportional dilutors are available for use in short- and long-term continuous flow bioassays with toxic chemicals or complex wastes.

Instrumentation is available for measurement of conventional water quality parameters (i.e., dissolved oxygen, salinity, conductivity, pH, temperature, hardness, alkalinity, ammonia-N, and sulfide). Supporting laboratory space and equipment are available such as balances, ovens, inverted and compound microscopes, and a refrigerated centrifuge. The laboratory includes 4°C space for sample chilling and storage.

In addition, NAS has over 500 square feet of modern office space equipped with FAX, internet access, copy machines and a small reference library for support in the toxicity testing program. Offices are equipped to provide everything needed for data reduction and analysis, technical report writing, and other common laboratory and consulting support functions. Networked microcomputers are available for word processing, data analysis and data management. NAS currently has the following software available for data analysis: Microsoft EXCEL 2000, Biostat (U.S. Army Corps of Engineers, Seattle District), ToxCalc v.5.0.23N (Tidepool Scientific), and CETIS software, also by Tidepool Scientific. Efficient literature searching and retrieval is provided through in-house capabilities, as well as access to the Oregon State University library.

B. ENVIRONMENTAL CONTROL

Temperature control of bioassay and culture aquaria is achieved by using constant temperature rooms and by immersion of containers in water baths. There are four walk-in temperature-controlled rooms available, each with separate temperature and photoperiod control. Additional testing space is available in the form of temperature-controlled water baths. Water baths are circulated using submersible pumps or by employing a cascading principle for water movement to minimize temperature gradients and stratification. Chilling is accomplished with Blue M portable chillers. Heating employs either stainless steel or Vycor heaters controlled by YSI Model 63RC thermister controllers. Temperature control is easily within the ±1°C limit specified in most bioassay test protocols. Aeration is provided using oil-free air compressors. The laboratory lights are under photoperiod control.

VIII. CALIBRATION PROCEDURES AND FREQUENCY

Analytical measurements routinely used in aquatic toxicology consist of water quality (e.g. temperature, salinity, conductivity, pH, etc.), and weight and volume measurements. Instruments used are properly and frequently calibrated to ensure accurate and reliable measurements. A brief description of the calibration procedures for these instruments is given in this section. All measuring operations and testing equipment having an effect on the accuracy or validity of tests are calibrated and/or verified before putting into use and on a continuing basis.

Calibration frequency and procedures for balances, pH meters, thermometers, and other support equipment are included as part of the procedure manual and/or in standard operating procedures.

A. TRACEABILITY OF CALIBRATION

Wherever applicable, calibration of analytical support equipment and instruments is traceable to national standards of measurement.

B. REFERENCE STANDARDS

Reference standards of measurement (such as Class S or equivalent weights or traceable thermometers) are used for calibration only. Reference standards are subjected to in-service checks between calibrations and verifications. Northwestern Aquatic Sciences maintains a contract with Quality Control Services, Portland, Oregon for regular calibration of reference thermometers (every three years), Class S weights, and laboratory balances (annually).

Standards and other reagents are labeled when received. The label includes receipt date, date opened, and expiration date. Normally reagents expire one year after opening unless otherwise indicated.

C. WATER QUALITY

Temperature is measured to the nearest degree Centigrade using digital or mercury thermometers,. Laboratory thermometers are calibrated annually against a certified Standard Thermometer traceable to National Institute of Standards and Technology (NIST) standards.

Salinity is measured to the nearest 0.5 part per thousand (ppt) using a hand-held temperature-compensated refractometer. The refractometer is zeroed before use and after approximately every twenty measurements.

All pH measurements are made to the nearest 0.1 pH unit using Orion SA250 pH meters and probes. The meters and probes are maintained according to factory specifications. The instrument is calibrated before each day of use with standard buffer solutions that bracket the pH range of the samples (pH 7.0 and 10.0 for seawater samples and pH 7.0 and 4.0 buffers for freshwater samples unless the pH of the samples is expected to be above 7.0). Documentation of satisfactory operation is recorded daily when in use.

Dissolved oxygen is measured to the nearest 0.1 ppm using YSI Model 51B dissolved oxygen meters and probes. The instruments are maintained according to factory specifications and calibrated before each day of use using water-saturated air or air-saturated water as specified in the manufacturer's instructions. Documentation of satisfactory operation is recorded daily when in use.

Specific conductance is measured in umhos/cm using YSI Model 33 S-C-T Meters or an ICM Conductivity Meter. The instruments are maintained according to factory specifications. Operating conditions of the meters (electronic balance and battery condition) are checked prior to each use as specified by the manufacturer. Documentation of satisfactory operation is recorded monthly based on calibration against a NIST traceable standard.

Water hardness and alkalinity are measured in freshwater test solutions using Hach test kits accurate to within 10 mg/L as $CaCO_3$. The kits are based on EPA methods 130.2 and 310.1, respectively, except that the alkalinity measurement employs a chemical indicator rather than an electrometrically determined endpoint of pH 4.5. Measurements are expressed as mg/L of $CaCO_3$. Sulfide and total ammonia-N are also measured when required. These analyses are performed using Hach test kits that reference the methylene blue (EPA Method 376.2) and salicylate (Clin. Chim. Acta 14:403, 1966) colorimetric methods, respectively. Samples are not distilled prior to analysis.

D. WEIGHTS AND VOLUMES

Reference toxicants are weighed using an analytical balance accurate to the nearest 0.1 mg. Balances are checked before and after each use using weights traceable to NIST standards. Balances are serviced annually by QC Services, Portland, OR. Effluent and reference toxicant dilutions are made using Class A volumetric flasks and pipettes and graduated cylinders.

IX. TEST ACCEPTABILITY

Test acceptance criteria are specific to the type of toxicity test being performed. The actual test protocol should be consulted in each case. In general, mean mortality in dilution water controls cannot exceed 10-30% in effluent, sediment or reference toxicant tests, depending upon the type of test. Water temperature and other water quality parameters also have criteria for test acceptability which are defined in each test protocol. An individual test may be conditionally acceptable if temperature, DO, and other specified conditions fall outside specifications, depending on the degree of departure and the objectives of the tests. The acceptability of the test will ultimately depend on the best professional judgment of the investigator. Any deviations from test specifications are to be noted when reporting data from the test.

A. DOCUMENTING LABORATORY PERFORMANCE

Reference toxicant tests are used to demonstrate satisfactory laboratory performance. Reference toxicant tests for most test methods are conducted simultaneously with unknown sample toxicity tests. A few test methods have reference toxicant tests run on each batch of animals rather than concurrently with project tests. At a minimum, even for tests not requested as frequently, reference toxicant tests are performed at least semi-annually for each toxicity test method used in the laboratory. Control charts are constructed to monitor the performance of bioassay test results using reference toxicants. These charts are used to demonstrate satisfactory laboratory performance. See Section X , D for the use and construction of control charts to evaluate and document laboratory performance.

B. ACCEPTABILITY OF TEST ORGANISMS

In general, test organisms are considered acceptable if: 1) all organisms are from a single group spawned/hatched/released or field-collected at the same time from the same location; 2) their survival in the control of a test using its standard dilution water or control sediment is at least 80 or 90%; and 3) the toxicity values obtained from a reference toxicant test fall within the established intervals on the laboratory's control chart for that reference toxicant/organism combination. Actual individual test protocols should be consulted for individual test acceptability criteria since they are specific each type of test.

C. ACCEPTABILITY OF PHYSICAL/CHEMICAL PROPERTIES OF TEST SOLUTIONS

Conventional water quality parameters (e.g., temperature, dissolved oxygen, pH, salinity or conductivity, and alkalinity and hardness or sulfide and ammonia-N where applicable) are measured in at least one replicate of each test concentration at the beginning and end of a test at a minimum for most tests. The specific requirements are listed in individual test protocols. These data are recorded on the raw data sheets for that particular test and the raw data sheets become part of the archived file for that project.

In general, test temperature measurements should be within 1°C of the designated temperature. Dissolved oxygen should not fall below a specified level (usually a percentage of saturation or specific mg/L); if it does, aeration must then be employed (or increased, if already employed). The pH of test materials may need to be adjusted prior to testing depending on the type of test and organism employed. Also depending on the type of test, ammonia and sulfide may need to be monitored at the beginning and end of a test at a minimum and possibly during a test to evaluate their contribution to any test effect.

D. BRINE CONTROLS/SALT CONTROLS

Brine controls should be included in tests that use hypersaline brine to adjust the salinity of effluent dilutions. Brine controls are made using the same volume of brine as is used in the highest effluent concentration. Brines produced by freezing natural seawater are preferable to brines made by evaporation or by addition of commercial sea salt formulations, although any of these methods may be used. SOPs are available for brine preparation and for brine volume calculations.

Control and brine control results are compared using a t-test or ANOVA. The effluent toxicity test is acceptable if there is no significant difference at the $p=0.05$ level. It may be advisable to test the organism's response to specific brines before attempting their use in a full effluent toxicity test.

Similarly, salt controls are used when sea salts have been used to adjust the salinity of effluents. Salt controls are prepared by using the same amount of sea salts as is used in the highest effluent concentration.

X. LABORATORY DOCUMENTATION

Quality assurance programs are dependent on timely and accurate record keeping. Documentation of QA checks and procedures provide proof of performance and a reference to guide future work. This section describes the types of records to be kept and assigns responsibility for record keeping.

A. TEST METHODS AND STANDARD OPERATING PROCEDURES (SOPs)

SOPs are documents that contain detailed information on the requirements for the correct performance of laboratory and other procedures. NAS maintains a master list of SOPs that reflect its current and former programs, as well as allowing for growth. The QAO and Lab Director are responsible for developing, writing, and approving SOPs. The types of SOPs at NAS include administrative sops, QA sops, general data sops, microbiology sops, chemistry sops, and toxicology sops. Within the toxicology sops there are currently eight categories of SOPs:

- Laboratory Equipment, Supplies and Materials
- General Laboratory Practices
- QC Program
- Test Methods - WET Testing
- Test Methods - Freshwater Sediment
- Test Methods - Marine Sediment
- Animal Culture and Collection Methods
- Data Analysis, Management, and Reporting

The SOPs at NAS are considered proprietary and not to be given out. For each test method, there is a test protocol that is followed and appended to the final report. The test protocols may be provided to the client for review and/or signature prior to starting a project.

There are three controlled copies of all officially approved SOPs: QA, laboratory, and NAS library. All SOPs are approved by the QA Officer before being implemented. The distribution of current SOPs and archiving of outdated ones is controlled through the QA Officer. Each SOP has a cover sheet with its SOP number, inception date, current revision date, title, file name, references, distribution list, revision summary, and dates and signatures of the Laboratory Director/Technical Director and QAO. For all test method SOPs, and only test method SOPs, there is a signature page at the end of the SOP. All employees who have been trained to perform this test method sign and date this page and attest to the fact that they have read and understood the SOP and are currently using those procedures.

B. LABORATORY BENCH SHEETS

Laboratory bench sheets are used to document information from many routine laboratory operations. For example bench sheets are used to collect required data for all bioassay tests. Other examples of the use of bench sheets include: 1) the collection of culture data; 2) recording data for test animal collections; and 3) laboratory systems operations. Bench sheets are used to insure that the information is recorded in a complete and organized manner and that the test or other procedure can be reconstructed, if necessary.

All entries to laboratory bench sheets are made in black water-proof ink. No erasures are made on the original data sheets. Corrections are made by crossing one line through the incorrect entry and writing the correct entry, an explanation for the correction, the date, and the initials of the person correcting the error. Error correction codes may be used in explaining corrections.

C. LABORATORY NOTEBOOKS

Laboratory notebooks are used to document information that cannot easily be recorded on bench sheets. Laboratory notebooks are typically maintained for sample logbooks, reagent logbooks, and project notebooks for the collection of data not suitable for bench sheets. Each page in a laboratory notebook is initialed and dated as information is entered. Notebook pages are numbered prior to use. Notebook entries and corrections are made as with bench sheets (see Section IX. B.).

D. CONTROL CHARTS

Control charts are constructed to monitor the performance of bioassay test results using reference toxicants. These charts are used to demonstrate satisfactory laboratory performance. Control charts are prepared for each reference toxicant, test species, test condition, and endpoint combination, and successive toxicity values are plotted and examined to determine if the results are within prescribed
limits. A minimum of five reference toxicant tests is needed for constructing a control chart. After two years or a minimum of 20 data points, the control chart is maintained using only the 20 most recent points. The mean and upper and lower control limits (± 2 S) are recalculated with each successive test result. If the toxicity value from a given test with a reference toxicant does not fall in the expected range, this is not an automatic cause for rejection of the effluent or sediment test. Chance alone would cause one in 20 tests to fall outside the limits at the $P_{0.05}$ probability level. If more than one out of 20 reference toxicant test fall outside the control limits, the source of the variability is investigated, corrective actions are taken to reduce the variability and another reference toxicant test is performed as soon as possible. If the cause of the outlier can be documented, the outlier should be excluded from future calculations of the control limits. If two or more consecutive tests fall outside the control limits, the results must be explained and the reference toxicant test repeated.

E. PROJECT FILES

A project file is created for each project handled within the laboratory. The project file contains all documents associated with the project. This includes correspondence from the client, chain-of-custody records, raw data, test protocols, bench sheets, copies of laboratory notebook entries pertaining to the project, and a copy of the final report. When a project is complete, all records are passed to the QA Officer who inventories the file, checks for completeness, and puts the file into document archive.

F. CONFIDENTIALITY

Often during the course of business, NAS is privy to data or information that is considered confidential or proprietary to our clients. Such information includes results of their tests, origin of their samples, the fact that we are working with them, any procedures or processes that they conduct or investigate, any information about them or their business, our own laboratory procedures, and clients. All such information must be kept strictly confidential. The information should only be discussed with people designated as technical contacts or purchasing agents for the particular project or corporate officers for the clients' company. The information should not be discussed with anyone else, even people within a client's company not designated as a contact, without written permission from the client.

Access to laboratory records and data is limited to laboratory personnel except with the permission of the QA Officer or Laboratory Director.

Where clients require transmission of test results by telephone, telex, facsimile or other electronic or electromagnetic means, staff will ensure confidentiality is preserved

XI. REPORTING OF TEST RESULTS

The final report of the test results should include all of the following standard information at a minimum: name and identification of the test including a reference to the test protocol; the names of the investigator(s) and laboratory; information on the sample(s) including date of receipt, the type of sample, storage information and any chain-of-custody records; information on the test or dilution water; detailed information about the test organisms including source and acclimation or culture conditions; a description of the experimental design and test chambers and other test conditions including water quality; information about any aeration that may have been required; definition of the effect criteria and other observations; responses, if any, in the control treatment; tabulation and statistical analysis of measured responses; a description of the statistical methods used; any unusual information about the test or deviations from procedures; the results of associated reference toxicant tests.

XII. PERFORMANCE AND SYSTEM AUDITS

NAS participates in available federal and state accreditation and performance audit programs that subject the laboratory to stringent system and performance audits. A <u>system audit</u> is a review of laboratory operations conducted to verify that the laboratory has the necessary facilities, equipment, staff, and procedures in place to generate acceptable data. A <u>performance audit</u>

verifies the ability of the laboratory to provide accurate toxicity test results using samples submitted by the auditing agency.

NAS is currently accredited for toxicity testing with the State of Washington (Lab Accreditation No. C042). The State of Oregon does not currently require accreditation for toxicology laboratories although they are starting to implement NELAC on a voluntary basis. NAS has participated in the EPA DMR Performance Evaluation of aquatic toxicology laboratories since 1991. NAS has also qualified by performance evaluation testing to participate in Effluent Toxicity Characterization Program studies for the San Francisco Bay Region, California Regional Water Quality Control Board.

NAS performs annual internal audits of the aquatic toxicology laboratory and is occasionally required by specific projects to be audited by the client or a third party QA consultant.

XIII. CORRECTIVE ACTION

When errors, deficiencies, or out-of-control situations exist, the QA Program provides systematic procedures, called "Corrective Actions," to resolve problems and restore proper functioning to the analytical system.

Laboratory personnel are alerted that corrective actions may be necessary if:

1. Annual Performance Evaluation test results are unacceptable;
2. Control organism performance (survival, growth, etc.) does not meet acceptance criteria;
3. Reference toxicant test results exceed control chart limits;
4. Deficiencies are detected during routine QA reviews; or
5. Inquiries concerning data quality are received from clients.

Corrective action procedures are often handled at the bench level by the project toxicologist through review of the records of test water quality, test organism acclimation or culture conditions, preparation of test solutions and other applicable test records. If the problem persists or cannot be identified, the matter is referred to the project manager, laboratory supervisor, and/or QA Officer for further investigation. Once resolved, full documentation of the corrective action procedure, should be described in a formal Corrective Action Report and filed with the QA unit. Corrective action documentation is routinely reviewed by management. Further information on corrective action is available in SOPs.

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Attachment #2 Test Protocol for the 28-day chronic sediment toxicity test with *Hyalella azteca*

Northwestern Aquatic Science

Protocol No. NAS-686-HA4c

TEST PROTOCOL

FRESHWATER AMPHIPOD, *HYALELLA AZTECA*, 28-DAY SEDIMENT SURVIVAL AND GROWTH TEST

1. INTRODUCTION

1.1 Purpose of Study: The purpose of this study is to characterize the chronic toxicity of freshwater sediments using a 28-day exposure and survival and growth endpoints with the amphipod, *Hyalella azteca*.

1.2 Referenced Method: This protocol is based on ASTM Method E 1706-00 (ASTM 2003) and EPA Method 100.4 (EPA/600/R-99/064, 2000)

1.3 Summary of Method: A summary of test conditions for the amphipod 28-day sediment survival and growth test is tabulated below. The test with *Hyalella azteca* is conducted at 23 ± 1°C with a 16L:8D photoperiod at an illuminance of about 100-1000 lux. Test chambers are 300-mL high-form lipless beakers containing 100 mL of sediment and 175 mL of overlying water. Ten 7-8day old amphipods are used in each replicate. The test will be conducted with eight replicates/treatment. Amphipods in each test chamber are fed 1.0 mL of a YCT food daily. Each chamber receives two volume additions per day of overlying water. Overlying water will be dechlorinated city water with a hardness of approximately 30 mg/L. Test endpoints include survival and growth.

2. STUDY MANAGEMENT

2.1 Sponsor's Name and Address:
____
____
____
____

2.2 Sponsor's Study Monitor:

____

2.3 Name of Testing Laboratory:
Northwestern Aquatic Sciences
3814 Yaquina Bay Road, P.O. Box 1437
Newport, OR 97365.

2.4 Test Location:

____

2.5 Laboratory's Personnel to be Assigned to the Study:
Project Manager: ____________________________
Quality Assurance Unit: _____________________________
Aquatic Toxicologist: _______________________________
Aquatic Toxicologist: _______________________________

2.6 Proposed Testing Schedule: Tests are to begin within eight weeks, but preferably within two weeks. Reference toxicant test to be run concurrently.

2.7 Good Laboratory Practices: The test is conducted following the principles of Good Laboratory Practices (GLP) as defined in the EPA/TSCA Good Laboratory Practice regulations revised August 17, 1989 (40 CFR Part 792).

3. TEST MATERIAL

The test materials are freshwater sediments. The control and test sediments are placed in solvent cleaned 1 L glass jars fitted with PTFE-lined screw caps. At the laboratory the samples are stored at 4°C in the dark. The original sealed containers may be stored for up to 8 weeks prior to testing. Tests are to begin within 14 days of sample collection. Eight week holding times may apply in some circumstances. If jars are not not full when received or if sediment is removed for testing, headspaces should be filled with nitrogen to retard deterioration. A negative control sediment collected from Beaver Creek, Lincoln County will be used for the toxicity comparison.

4. TEST WATER

Test water (overlying water) at NAS is normally moderately hard synthetic water at a hardness of 80-100 mg/L as $CaCO_3$ and alkalinity of 60-70 mg/L as $CaCO_3$. Dilution water is prepared from Milli-Q reagent grade water and reagent grade chemicals. For the Portland Harbor study the water will be dechlorinated city water with hardness adjusted to approximately 30 mg/L.

5. TEST ORGANISMS

5.1 Species: amphipod, *Hyalella azteca*.

5.2 Source: Cultured at NAS. Alternatively, animals may be purchased from a reputable commercial supplier.

5.3 Age: 7-8 days old at start of test

5.4 Acclimation and Pretest Observation: Cultures are maintained at 23 ± 1°C under a 16:8 L:D photoperiod. Cultured amphipods are fed dried maple leaves with occasional Tetramin®, TetraFin® flake or rabbit chow supplements. Acclimation of test organisms to the test water of lower hardness (approximately 30 mg/L) may be necessary. If test organisms are to be acclimated, fifty percent of the holding water is changed daily with the addition of test water.

6. DESCRIPTION OF TEST SYSTEM

6.1 Test Chambers and Environmental Control: Test chambers used in the toxicity test are 300-mL high-form lipless glass beakers. Test chambers are maintained at constant temperature by partial immersion in a temperature-controlled water bath or by placement in a temperature-controlled room. Aeration is not employed unless dissolved oxygen drops below 2.5 mg/L. The test is conducted under an illuminance of 100-1000 lux with a 16L:8D photoperiod.

6.2 Cleaning: All laboratory glassware, including test chambers, is cleaned as described in EPA/600/4-90/027F. New glassware and test systems are soaked 15 minutes in tap water and scrubbed with detergent (or cleaned in automatic dishwasher); rinsed twice with tap water; carefully rinsed once with fresh, dilute (10%, V:V) hydrochloric or nitric acid to remove scale, metals, and bases; rinsed twice with deionized water; rinsed once with acetone to remove organic compounds (using a fume hood or canopy); and rinsed three times with deionized water. Test systems and chambers are rinsed again with dilution water just before use.

7. EXPERIMENTAL DESIGN AND TEST PROCEDURES

7.1 Experimental Design: The test involves exposure of amphipods to test and control sediments. The sediments are placed on the bottom of the test containers and are overlain with test water. The test exposure is for 28 days. The renewal of overlying water consists of two volume additions per day (static renewal). Each treatment consists of eight replicate test containers, each containing 10 organisms. Test chamber positions are completely randomized. Test organisms are randomly distributed to the test chambers. Blind testing will be used.

7.2 Setup of Test Containers: Sediments are homogenized and placed in test chambers on the day before addition of test organisms. Sediment (100 ml) is placed into each of eight replicate beakers. After addition of the sediment, 175 ml of test water is gently added to each beaker in a manner to prevent resuspension. The overlying water is replaced twice daily. The test begins when amphipods are introduced to the test chambers. Initial water quality measurements are taken prior to the addition of test organisms.

7.3 Effect Criterion: The effect criteria used in the 28-day amphipod bioassay are mortality and growth. Death is defined as the lack of movement of body or appendages on response to tactile stimulation. Growth is measured as change in dry weight.

7.4 Test Conditions: No aeration is employed unless dissolved oxygen falls below 2.5 mg/L. The test temperature employed is 23 ± 1 C. A 16:8, L:D photoperiod is used. Illumination is supplied by daylight fluorescent lamps at 100-1000lux. The overlying water is replaced twice daily.

7.5 Beginning the Test: On the day the test begins, amphipods are impartially counted into small containers of test water (10/container). The test is begun by rinsing test organisms into the equilibrated test containers. For the growth endpoint, time-zero weight data should be collected.

Feeding: Amphipods are fed 1.0 mL of YCT daily per test chamber. A feeding may be skipped if there is a build up of excess food. However, all beakers must be treated similarly.

Test Duration, Type and Frequency of Observations, and Methods: The duration of the toxicity test is 28 days. The type and frequency of observations to be made are summarized as follows:

TYPE OF OBSERVATION	TIMES OF OBSERVATION
BIOLOGICAL DATA	
Survival, growth	Day 28
PHYSICAL AND CHEMICAL DATA	
Hardness, alkalinity, conductivity, and ammonia-N	Beginning and end of test in overlying water of one replicate beaker from each treatment.
Temperature	Daily in overlying water of one replicate beaker from each treatment.
Conductivity	Weekly
Dissolved oxygen and pH	3X/week
Pore water ammonia	In test sediment taken from the bulk homogenized sediment prior to initiating the tests (day -1).

Dissolved oxygen is measured using a polarographic oxygen probe calibrated according to the manufacturer's recommendations. The pH is measured using a pH probe and a properly calibrated meter with scale divisions of 0.1 pH units. Temperature is measured with a calibrated mercury thermometer or telethermometer. Conductivity is measured with a conductivity meter. Hardness and alkalinity are measured using titrometric methods. Total ammonia-N is measured using Hach test kits based on the salicylate (Clin. Chim. Acta 14:403, 1996) colorimetric method.

Overlying water should be sampled just before water renewal from about 1 to 2 cm above the sediment surface using a pipet. It may be necessary to pool water samples from individual replicates. The pipet should be checked to make sure no organisms are removed during sampling of overlying water.

7.8 Test Termination: At test termination, the contents of each test container are sieved through a #35 (500 µm mesh) sieve to recover the amphipods. Amphipods from each replicate are put into a 30 mL plastic cup, rinsed with DI water, gently blotted and place into the appropriate tared aluminum weighing pan. The number of survivors for each container is recorded on the datasheet.

7.9 Growth Measurement: Growth is measured as average dry weight of animals in a test replicate at the end of the test on day 28. Pooled animals from each test replicate are gently blotted and placed into tared aluminum weigh pans. The pans are dried at 60-90°C to constant weight. The dried amphipods are placed into a dessicator and weighed as soon as possible to the nearest 0.01 mg (desirable to use 0.001 mg). The total weight of the dried amphipods in each pan is divided by the number of amphipods weighed to obtain an average dry weight per surviving amphipod per replicate.

8. CRITERIA OF TEST ACCEPTANCE

The test results are acceptable if the minimum survival of organisms in the control treatment at the end of the test is at least 80%.

9. DATA ANALYSIS

The endpoints of the toxicity test are survival and growth. Survival is obtained as a direct count of living organisms in each test container at the end of the test. Average amphipod dry weight is obtained as described above. The means and standard deviations are calculated for each endpoint and each treatment level. The statistical analysis will be performed by Windward.

10. REPORTING

The final report of the test results must include all of the following standard information at a minimum: name and identification of the test; the investigator and laboratory; date and time of test beginning and end; information on the test material; information on the source and quality of the overlying/test water; detailed information about the test organisms including acclimation conditions; a description of the experimental design and test chambers and other test conditions including feeding, if any, and water quality; definition of the effect criteria and other observations; responses, if any, in the control treatment; tabulation of measured responses from each replicate and a summary table of endpoints; any unusual information about the test or deviations from procedures; reference toxicant testing information.

11. STUDY DESIGN ALTERATION

Amendments made to the protocol must be approved by the sponsor and project manager and should include a description of the change, the reason for the change, the date the change took effect and the dated signatures of the project manager and sponsor. Any deviations in the protocol must be described and recorded in the study raw data.

12. REFERENCE TOXICANT

The reference toxicant test is a standard multi-concentration toxicity test using a specified chemical toxicant to evaluate the performance of test organisms used in the study. Reference toxicant tests are 96-hour, water only exposures, not 28-day sediment exposures. The reference toxicant test is run concurrently. Performance is evaluated by comparing the results of the reference toxicant test with historical results (e.g., control charts) obtained at the laboratory.

13. REFERENCED GUIDELINES

ASTM. 2003. Standard Test Methods for Measuring the Toxicity of Sediment-Associated Contaminants with Fresh Water Invertebrates. ASTM Standard Method No. E 1706-00. Am. Soc. Test. Mat., West Conshohocken, PA.

U.S. EPA. 2000. Section 11, Test Method 100.1, *Hyalella azteca* 10-d Survival and Growth Test for Sediments, pp. 47-54 In: Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates (Second Edition). EPA/600/R-99/064.

Weber, C.I. (Ed.) 1993. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms (Fourth Edition). EPA/600/4-90/027F.

14. APPROVALS

____________________________________for _________________________________
Name Date

____ for **Northwestern Aquatic Sciences**
Name Date

Test Conditions Summary

1. Test type	whole sediment toxicity test with renewal of overlying water
2. Test duration	28 days
3. Temperature	23 ± 1°C
4. Light quality	daylight flourescent light
5. Illuminance	100-1000 lux
6. Photoperiod	16L:8D
7. Test chamber size	300-mL high-form lipless beakers, (Pyrex® 1040 or equivalent)
8. Sediment volume	100 mL
9. Overlying water volume	175 mL
10. Renewal overlying water	2 volume additions/day (static renewal)
11. Age of test organisms	7-8 days old at test initiation
12. Organisms per test chamber	10
13. Replicates per treatment	8
14. Organisms per treatment	80
15. Feeding regime	YCT food, fed 1.0 mL daily/chamber
16. Cleaning	if screens are used, clean as needed
17. Aeration	None, unless DO falls below 2.5 mg/L
18. Overlying (test) water	Declorinated city water with hardness adjusted to approximately 30 mg/L
19. Overlying water quality	Hardness, alkalinity, conductivity, ammonia-N beginning and end; temperature daily; conductivity weekly; DO & pH 3X/wk
20. Endpoints	Survival & growth (based on weight)
21. Test acceptability criteria	Minimum control survival of 80%
22. Sample holding	≤ 8 weeks at 4°C in the dark, preferable 2 weeks
23. Sample volume required	1L (800 mL per sediment)
24. Reference toxicant	Concurrent testing required with cadmium as toxicant

Attachment #3 Test Protocol for the 10-day acute sediment toxicity test with *Chironomus tentans*

TEST PROTOCOL

FRESHWATER MIDGE, *CHIRONOMUS TENTANS*, 10-DAY SEDIMENT TOXICITY TEST

INTRODUCTION
Purpose of Study: The purpose of this study is to characterize the toxicity of freshwater sediments based on midge survival and growth using the midge, *Chironomus tentans*.

Referenced Method: This protocol is based on EPA Method 100.2 (EPA/600/R-99/064 2000) and ASTM Method E 1706-00 (ASTM 2003).

Summary of Method: A summary of test conditions for the midge 10-day sediment toxicity test is tabulated below. The 10-day sediment toxicity test with *Chironomus tentans* is conducted at 23°C with a 16L:8D photoperiod at an illuminance of about 100-1000 lux. Test chambers are 300-mL high-form lipless beakers containing 100 mL of sediment and 175 mL of overlying water. Ten second to third-instar midges are used in each replicate (all organisms must be third instar or younger and at least 50% of the larvae must be third instar). The test will be conducted with eight replicates/ treatment. Midges in each test chamber are fed 1.5 mL of a 4 g/L fish food flakes suspension daily. Each chamber receives two volume additions per day of overlying water. Overlying water will be dechlorinated city water with a hardness of as close to 30 mg/L as the organisms can tolerate. Test endpoints include survival and/or growth.

STUDY MANAGEMENT

Sponsor's Name and Address:
____
____
____
____

Sponsor's Study Monitor:
____

Name of Testing Laboratory:
Northwestern Aquatic Sciences
3814 Yaquina Bay Road, P.O. Box 1437
Newport, OR 97365.

Test Location: ___________________________________

Laboratory's Personnel to be Assigned to the Study:
Project Manager: ___________________________
Quality Assurance Unit: ______________________________
Aquatic Toxicologist: ________________________________
Aquatic Toxicologist: ________________________________

Proposed Testing Schedule: Tests are to begin withineight week, preferably within two weeks. Reference toxicant test to be run concurrently.

Good Laboratory Practices: The test is conducted following the principles of Good Laboratory Practices (GLP) as defined in the EPA/TSCA Good Laboratory Practice regulations revised August 17, 1989 (40 CFR Part 792).

TEST MATERIAL

The test materials are freshwater sediments. The control and test sediments are placed in solvent cleaned 1 L glass jars fitted with PTFE-lined screw caps. At the laboratory the samples are stored at 4°C in the dark. The original sealed containers may be stored for up to 14 days prior to testing. Tests are to begin within 14 days of sample collection. Eight week holding times may apply in some circumstances. If jars are not full when received or if sediment is removed for testing, headspaces should be filled with nitrogen to retard deterioration. A negative control sediment collected from Beaver Creek, Lincoln County, will be used for the toxicity comparison.

TEST WATER

Test water (overlying water) at NAS is normally *C. tentans* culture water, which is moderately hard synthetic water at a hardness of 80-100 mg/L as CaCO3 and alkalinity of 60-70 mg/L as CaCO3. Dilution water is prepared from Milli-Q reagent grade water and reagent grade chemicals. For the Portland Harbor study the water will be dechlorinated city water with hardness adjusted as close to 30 mg/L as the organisms can tolerate.

TEST ORGANISMS

Species: midge, *Chironomus tentans*.

Source: Cultured at NAS (Originally obtained from U.S. EPA Environmental Research Lab, Duluth, MN) or purchased from a reputable commercial supplier.

Age: Third instar or younger larvae (at least 50% of the larvae must be in the third instar at the start of the test). Third instar is normally 9 to 11 days after hatching; head capsule widths range from 0.33 to 0.45 mm; or length ranges from 4-6 mm; or dry weight ranges 0.08 to 0.23 mg/individual.

Acclimation and Pretest Observation: Cultures are maintained at 23 ± 1°C under a 16:8 L:D photoperiod. The culture water is moderately hard synthetic water. Acclimation of test organisms to the test water of lower hardness (approximately 30 mg/L) may be necessary. Midge are fed finely ground Tetrafin flakes in suspension (10g Tetrafin in 100 mL Milli-Q water). Mortality during the 48-hr prior to testing should not be excessive (<20%).

DESCRIPTION OF TEST SYSTEM

Test Chambers and Environmental Control: Test chambers used in the toxicity test are 300-mL high-form lipless glass beakers (Pyrex® 1040 or equivalent). Test chambers are maintained at constant temperature by partial immersion in a temperature-controlled water bath or by placement in a temperature-controlled room. Aeration is not employed unless dissolved oxygen drops below 2.5 mg/L. The test is conducted under an illuminance of 100 to 1000 lux with a 16L:8D photoperiod.

Cleaning: All laboratory glassware, including test chambers, is cleaned as described in EPA/600/4-90/027F. New glassware and test systems are soaked 15 minutes in tap water and scrubbed with detergent (or cleaned in automatic dishwasher); rinsed twice with tap water; carefully rinsed once with fresh, dilute (10%, V:V) hydrochloric or nitric acid to remove scale, metals, and bases; rinsed twice with deionized water; rinsed once with acetone to remove organic compounds (using a fume hood or canopy); and rinsed three times with deionized water. Test systems and chambers are rinsed again with dilution water just before use.

EXPERIMENTAL DESIGN AND TEST PROCEDURES

Experimental Design: The test involves exposure of midge larvae to test and control sediments. The sediments are placed on the bottom of the test containers and are overlain with test water. The test exposure is for 10 days. The renewal of overlying water consists of two volume additions per day (static renewal). Each treatment consists of eight replicate test containers, each containing 10 organisms. Test chamber positions are completely randomized. Test organisms are randomly distributed to the test chambers. Blind testing is normally used.

Setup of Test Containers: Sediments are homogenized and placed in test chambers on the day before addition of test organisms. Sediment (100 ml) is placed into each of eight replicate beakers. After addition of the sediment, 175 ml of test water is gently added to each beaker in a manner to prevent resuspension. The overlying water is replaced twice daily. The test begins when midges are introduced to the test chambers. Initial water quality measurements are taken prior to the addition of test organisms.

Effect Criterion: The effect criterion used in the midge bioassay is mortality, defined as the lack of movement of body or appendages on response to tactile stimulation. The chronic effect criterion is growth which is determined by using dry weight measurements.

Test Conditions: No aeration is employed unless dissolved oxygen falls below 2.5 mg/L. The test temperature employed is 23°C (range of ± 1°C). A 16:8, L:D photoperiod is used. Illumination is supplied by daylight fluorescent lamps at 100-1000 lux. The overlying water is replaced twice daily.

Beginning the Test: The test is begun by adding the organisms to the equilibrated test containers as previously described. Three extra replicates of midge larvae should be counted out and randomly selected for drying to determine initial average weight and instar data.

Feeding: Midge larvae are fed 1.5 mL daily per test chamber (1.5 mL contains 6.0 mg of dry solids). A feeding may be skipped if there is a build up of excess food. However, all beakers must be treated similarly.

Test Duration, Type and Frequency of Observations, and Methods: The duration of the acute toxicity test is 10 days. The type and frequency of observations to be made are summarized as follows:

Type Of Observation	Times Of Observation
Biological Data	
Survival, growth	Day 10
Physical And Chemical Data	
Hardness, alkalinity, ammonia-N, conductivity, pH, dissolved oxygen, and temperature	Beginning and end of test in overlying water of one replicate beaker from each treatment.
Dissolved oxygen, temperature	Daily in overlying water of one replicate beaker from each treatment.
Pore water ammonia	In test sediment taken from the bulk homogenized sediment prior to initiating the tests (day -1).

Dissolved oxygen is measured using a polarographic oxygen probe calibrated according to the manufacturer's recommendations. The pH is measured using a pH probe and a properly calibrated meter with scale divisions of 0.1 pH units. Temperature is measured with a calibrated mercury thermometer or telethermometer. Conductivity is measured with a conductivity meter. Hardness and alkalinity are measured using titrometric methods. Ammonia-nitrogen is measured using the salicylate colerimetric method (Clin. Chim. Acta 14:403, 1996).

Growth Measurement: Growth is measured as ash-free dry weight (AFDW) of animals in a test replicate at the end of the test on day 10. Pooled animals from each test replicate are rinsed with deionized water, gently blotted and placed into tared aluminum weigh pans. The pans are dried at 60-90°C to constant weight. The dried organisms are placed into a dessicator and weighed as soon as possible to the nearest 0.01 mg (desirable to use 0.001 mg). The total weight of the dried midge in each pan is divided by the number of midge weighed to obtain an average dry weight per midge. The dried larvae in the pan are then ashed at 550°C for two hours. The pan with the ashed larvae is then reweighed and the tissue mass of the larvae is determined as the difference between the weight of the dried larvae plus pan and the weight of the ashed larvae plus pan. Pupae or adult organisms are not included in the sample to estimate AFDW.

CRITERIA OF TEST ACCEPTANCE:

The test results are acceptable if the minimum survival of organisms in the control treatment at the end of the test is at least 70% and the average ash-free dry weight of *C. tentans* in the surviving controls is at least 0.48 mg.

DATA ANALYSIS

The endpoints of the toxicity test are survival and growth. Survival is obtained as a direct count of living organisms in each test container at the end of the test. Average midge ash-free dry weight is used to compare growth between treatment sediments and the control sediment. The means and standard deviations are calculated for each endpoint and for each treatment level. The statistical analysis will be performed by Windward.

REPORTING

The final report of the test results must include all of the following standard information at a minimum: name and identification of the test; the investigator and laboratory; date and time of test beginning and end; information on the test material; information on the source and quality of the overlying/test water; detailed information about the test organisms including acclimation conditions; a description of the experimental design and test chambers and other test conditions including feeding, if any, and water quality; definition of the effect criteria and other observations; responses, if any, in the control treatment; tabulation of measured responses from each replicate and a summary table of endpoints; any unusual information about the test or deviations from procedures; reference toxicant testing information.

STUDY DESIGN ALTERATION

Amendments made to the protocol must be approved by the sponsor and project manager and should include a description of the change, the reason for the change, the date the change took effect and the dated signatures of the project manager and sponsor. Any deviations in the protocol must be described and recorded in the study raw data.

REFERENCE TOXICANT

The reference toxicant test is a standard multi-concentration toxicity test using a specified chemical toxicant to evaluate the performance of test organisms used in the study. Reference toxicant tests are 96-hour, water only exposures, not 10-day sediment exposures. The reference toxicant test is run concurrently. Performance is evaluated by comparing the results of the reference toxicant test with historical results (e.g., control charts) obtained at the laboratory.

REFERENCED GUIDELINES

ASTM. 2003. Standard Test Methods for Measuring the Toxicity of Sediment-associated Contaminants with Fresh water Invertebrates. ASTM Standard Method No. E 1706-00. Am. Soc. Test. Mat., West Conshohocken, PA.

U.S. EPA. 2000. Section 12, Test Method 100.2, *Chironomus tentans* 10-d Survival and Growth Test for Sediments, pp. 55-62. In: Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates (Second Edition). EPA/600/R-99/064.

Weber, C.I. (Ed.) 1993. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms (Fourth Edition). EPA/600/4-90/027F.

APPROVALS

__________________________________for _____________________________________
Name Date

______________________________________ for Northwestern Aquatic Sciences
Name Date

Test Conditions Summary

1. Test type	whole sediment toxicity test with renewal of overlying water
2. Test duration	10 days
3. Temperature	23 ± 1°C
4. Light quality	daylight flourescent light
5. Illuminance	100-1000 lux
6. Photoperiod	16L:8D
7. Test chamber size	300-mL high-form lipless beakers (Pyrex® 1040 or equivalent)
8. Sediment volume	100 mL
9. Overlying water volume	175 mL
10. Renewal overlying water	2 volume additions/day (static renewal)
11. Age of test organisms	2nd to 3rd instar or younger larvae (≥ 50% of organisms must be 3rd instar)
12. Organisms per test chamber	10
13. Replicates per treatment	8
14. Organisms per treatment	80
15. Feeding regime	Fish food flakes, fed 1.5 mL chamber (1.5 mL contains 6.0 mg of dry solids) daily on days 0 - 9.
16. Aeration	None, unless DO falls below 2.5 mg/L.
17. Overlying (test) water	Dechlorinated city water with hardness adjusted as close to 30 mg/L as the organisms can tolerate
18. Overlying water quality	Hardness, alkalinity, conductivity, pH, ammonia-N beginning and end; temperature and DO daily
19. Endpoints	Survival and growth (dry weight)
20. Test acceptability criteria	Minimum control survival of 70%; mean weight of surviving control organisms 0.48 mg AFDW
21. Sample holding	≤ 8 weeks at 4°C in the dark, preferable 2 weeks
22. Sample volume required	1L (800 mL per sediment)
23. Reference toxicant	Concurrent testing required with potassium chloride as toxicant

Attachment #4 Examples of electronic data submittal for the toxicity tests

APPENDIX C

LABORATORY QUALITY ASSURANCE MANUALS

Columbia Analytical Services, Inc.
Northeast Analytical, Inc.
STL Seattle

(Provided on accompanying CD.)