



PORTLAND HARBOR RI/FS

ROUND 1

QUALITY ASSURANCE PROJECT PLAN

FINAL REPORT

November 22, 2002

Prepared for:
The Lower Willamette Group

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SECTION A
PROJECT MANAGEMENT

A1 TITLE AND APPROVAL SHEET

PORTLAND HARBOR SUPERFUND SITE LOWER WILLAMETTE RIVER RI/FS ROUND 1

Quality Assurance Plan Approvals

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SEA Chemistry QA Manager:	Janet Cloutier	_____	Date: _____
U.S. EPA Project Officer:	Wally Reid	_____	Date: _____
U.S. EPA Project QA Manager	Ginna Grepo-Grove	_____	Date: _____
ARI Project Manager:	Sue Dunnihoo	_____	Date: _____
ARI Laboratory QA Manager:	Dave Mitchell	_____	Date: _____
CAS Project Manager:	Abbie Spielman	_____	Date: _____
CAS Laboratory QA Manager:	Lee Wolf	_____	Date: _____
Axys Project Manager:	Coreen Hamilton	_____	Date: _____
Axys Laboratory QA Manager:	Dave Hoover	_____	Date: _____

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- Appendix G: Fish Tissue Compositing and Shipping SOP Round 1 Portland Harbor
RI/FS August 28, 2002
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RI/FS August 28, 2002

A2.4 LIST OF ACRONYMS

ACG	Analytical concentration goals
AG	Amber glass
AOC	Administrative Order on Consent
ARI	Analytical Resources Incorporated
ASTM	American Society for Testing and Materials
⁷ Be	Beryllium 7
BFB	Bromofluorobenzene
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
CRITFC	Columbia River Inter-Tribal Fish Commission
DFTPP	Decafluorotriphenylphosphine
DMMP	Dredge material management program
DQO	Data quality objective
EPA	U.S. Environmental Protection Agency
EQulS	Environmental Quality Information System
ERA	Ecological risk assessment
FSP	Field sampling plan
GC/ECD	Gas chromatography/electron capture detector
GC/MS	Gas chromatography/mass spectrometry
GFAA	Graphite furnace atomic absorption
G/p	Glass or plastic
GPC	Gel permeation chromatography
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
IDL	Instrument detection limit
IRP	Initial precision and recovery
ISA	Initial study area
LCS	Laboratory control sample
LCSD	Laboratory control sample duplicate
LOER	Laboratory for Oceanographic and Environmental Research, Texas A&M
LWG	Lower Willamette Group
LWR	Lower Willamette River
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
NRDA	Natural Resource Damage Assessment
OSHA	U.S. Occupational Safety and Health Administration
²¹⁰ Pb	Lead 210
PARCC	Precision, accuracy, representativeness, completeness, and comparability
PCB	Polychlorinated biphenyl
PCDE	poly-chlorinated diphenyl ether
PCP	Pentachlorophenol
pg/g	Picogram/gram
ppm	Parts per million
ppb	Parts per billion
ppt	Parts per trillion
PSEP	Puget Sound Estuary Program
PTFE	polytetrafluoroethylene
QA	Quality assurance
QA/QC	Quality assurance/quality control
QAPP	Quality assurance project plan
QC	Quality control
RA	Remedial action
RAO	Remedial Action Objective
RI/FS	Remedial Investigation/Feasibility Study
RM	River mile
RPD	Relative percent difference
RSD	Relative standard deviation
S	Sulfur
SDG	Sample delivery group
SEA	Striplin Environmental Associates
SICP	Selected ion current profile
SIM	Selected Ion Monitoring
SOP	Standard operating procedure
SOW	Statement of work
SRM	Standard reference material
SVOA	Semivolatile organic analysis
SVOC	Semivolatile organic compound
TBT	Tributyltin
TDS	Total dissolved solids
TOC	Total organic carbon
TSS	Total suspended solids
USGS	U.S. Geological Survey
VOA	Volatile organic analysis
VOC	Volatile organic compound
USC	Unified soil classification
WMG	Wide mouth glass

A3 DISTRIBUTION LIST

Oregon DEQ: Eric Blischke
US Fish & Wildlife Service: Ted Buerger
Columbia River Inter Tribal Fish Commission: Julie Carter
SEA Chemistry QA Manager: Janet Cloutier
Confederated Tribes of the Warm Springs Reservation of Oregon: Brian Cunningham
Confederated Tribes of the Siletz Indians: Tom Downey
ARI Project Manager: Sue Dunnihoo
Nez Perce Tribe: Rick Eichstaedt
Confederated Tribes of the Grand Ronde Community of Oregon: Kathleen Feehan
U.S. EPA Project QA Manager: Ginna Grepo-Grove
Axys Project Manager: Coreen Hamilton
Port of Portland: Trey Harbert
Yakima Nation: Lynn Hatcher
NOAA: Hellen Hillman
Axys Laboratory QA Manager: Dave Hoover
DNR Confederated Tribes of the Umatilla Indian Reservation: Audie Huber
EPA Oregon Operations Office: Chip Humphrey
Oregon Department of Fish & Wildlife: Rick Kepler
ARI Laboratory QA Manager: Dave Mitchell
U.S. EPA Project Officer: Wally Reid
Anchor Environmental LLC: Libby Smith
CAS Project Manager: Abbie Spielman
SEA Project Manager: Betsy Striplin
CAS Laboratory QA Manager: Lee Wolf
Northwest Natural: Robert Wyatt

A4 PROJECT/TASK

This section presents the organizational structure for sampling and analysis activities associated with the Portland Harbor Remedial Investigation and Feasibility Study (RI/FS) Round 1 investigation. The Lower Willamette Group (LWG) under the oversight of EPA Region 10 is conducting the RI/FS. The Round 1 investigation includes planning, fieldwork, laboratory analysis, data management and data evaluation. The Round 1 project organization and major task responsibilities are illustrated in Figure A4-1. Communications with EPA will be through these individuals. Contact information is listed in Table A4-1.

A4.1 EPA ORGANIZATION AND RESPONSIBILITIES

EPA is the lead agency for all Portland Harbor in-water RI/FS activities. EPA will oversee LWG activities associated with the Portland Harbor RI/FS, including the Round 1 investigation, as described in the RI/FS Work Plan (Striplin et al. 2002). EPA will coordinate all Trustee, tribe, and State input with respect to development of technical and decision documents. The site managers for EPA are Wallace Reid (Mr. Reid may be reached at Reid.Wallace@epamail.epa.gov and 206-553-1728), Chip Humphrey (Mr. Humphrey may be reached at Humphrey.Chip@epamail.epa.gov and 503-326-2678), and Tara Karamas (Ms. Karamas may be reached at Karamas.Tara-Ann@epamail.epa.gov and 206-553-0039).

EPA technical staff (with significant involvement in this RI/FS) includes Dana Davoli (human health risk assessment), Joe Goulet (ecological risk assessment), Julie Wroble (human health risk assessment), and Ginna Grepo-Grove (EPA QA-manager). Contact information for these individuals is located in Table A4-1. All communication with EPA should be through these managers and technical staff (see Table A4-1).

A4.2 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., Tosco Corporation, and Union Pacific Railroad. Each of these entities has signed the Administrative Order on Consent (AOC) for this Site.

The LWG is responsible for conducting the RI/FS and reporting the results in documents according to the Work Plan, AOC and referenced EPA guidance. The Lower Willamette Group is co-chaired by Mr. Trey Harbert of the Port of Portland and Mr. Bob Wyatt of Northwest Natural Gas. All official contact with the LWG should be through either Mr. Harbert or Mr. Wyatt (see Table A4-1).

A4.3 LWG TEAM ORGANIZATION AND RESPONSIBILITIES

Contractors retained by the LWG will undertake Round 1 sampling and analysis activities. The LWG consultant team is responsible for implementation of these tasks at the direction and oversight of the LWG. The organizational structure of the lead sampling and analysis personnel and associated laboratories is shown in Figure A4-1, and is described below.

A4.3.1 RI/FS Project Coordinator

Betsy Striplin of Striplin Environmental Associates (SEA) will be the RI/FS Project Coordinator and will manage the Portland Harbor RI and coordinate the overall RI/FS efforts. In this role, she will oversee the RI technical work and coordinate RI/FS activities with the LWG consultant team and other technical consultants. The Sampling and Analysis Coordinator will report directly to Ms. Striplin, along with other key team members to ensure that the objectives of the Round 1 field investigation are achieved. In the event that changes in the Field Sampling Plan (FSP) or Quality Assurance Project Plan (QAPP) are needed, Ms. Striplin will discuss proposed changes with the LWG and Environmental Protection Agency's (EPA) Project Manager 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. If this occurs, the LWG will notify the EPA Project Manager as soon as possible. Ms. Striplin may be reached at bstriplin@striplin.com and 206-241-5185.

A4.3.2 Sampling and Analysis Coordinator and Field QA Manager

Gene Revelas (SEA) will be the Sampling and Analysis Coordinator and will be responsible for all facets of the sampling and analysis program. His specific responsibilities include the following:

- Coordinate the field and laboratory analysis activities
- Provide technical direction and oversight of all contractors
- Ensure that laboratory capacity is sufficient to undertake the required analysis in a timely manner
- Ensure adherence to the schedule by tracking sampling, laboratory analysis, validation, and data management tasks
- Provide solutions to problems if they occur
- Inform the RI/FS Project Coordinator of required changes to the FSP and QAPP.

Mr. Revelas will also serve as Field QA Manager. In this role, he will oversee all aspects of the sampling events to ensure that the appropriate procedures and

methods are used. This may include, but is not limited to, field audits, review of field records and reports, and direct discussions with field personnel.

Mr. Revelas will report directly to the RI/FS Project Coordinator. Mr. Revelas may be reached at grevelas@striplin.com and 360-705-3534.

A4.3.3 LWG Common Consultants: Round 1 Managers

Each LWG consultant is responsible for major RI/FS tasks reflecting the firm's areas of expertise. In turn, each firm will support other consultant team members where appropriate. These consultants will provide direction and input in their areas of expertise during the Round 1 investigation. Ms. Laura Kennedy of Kennedy-Jenks Consultants is responsible for the Round 1 human health risk evaluation. Ms Kennedy may be reached at laurakennedy@kennedyjenks.com and 415-243-2150. Ms. Lisa Saban of Windward Environmental will conduct the Round 1 evaluation of ecological risks. Ms. Saban may be reached at lisas@windwardenv.com and 206-577-1288. Mr. Carl Stivers of Anchor Environmental will be responsible for the Round 1 FS activities. Mr. Stivers may be reached at cstivers@anchorenv.com and 206-287-9130. These managers will communicate directly with the Sampling and Analysis Coordinator or other staff as designated by the Sampling and Analysis Coordinator.

Qualifications of the consultant team project managers are described in the Work Plan (Striplin et al. 2002).

A4.3.4 Field Coordinator

Ian Stupakoff (SEA) will be the Field Coordinator and will be responsible for overall coordination of the field sampling tasks. Specifically, he will:

- Oversee the planning for each sampling event
- Coordinate field support between multiple sampling events scheduled for fall 2002
- Direct all aspects of the sampling events to ensure that the appropriate procedures and methods are used.

Mr. Stupakoff may be reached at stupakoff@striplin.com and 360-705-3534.

He 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.

A4.3.5 Field Staff

Field staff for the sampling events will be drawn from SEA and the consultant team. The operators of sampling vessels and equipment, as appropriate, will supply additional staff. Station positioning will generally be the responsibility of the vessel operator. In the event the vessel operator does not have station positioning capability or cannot meet the positioning requirements of the project, station-positioning services will be provided by a qualified subcontractor (e.g., David Evans and Associates, Portland, OR; Blue Water Engineering, Seattle, WA).

For all sampling tasks, the field crew will include the following individuals: site safety officer, cruise leader, and field staff. The organizational structure of the field cruise leaders for the various sampling events is shown in Figure A4-1. Collection of samples from the different matrices requires different sampling equipment, and different sampling vessels may also be used. Only one type of matrix will be sampled at a time from a given sampling vessel. Therefore, Round 1 sampling will be a series of sampling events, each addressing one type of matrix. For this reason, different cruise leaders have been selected for each sampling effort based on their experience and knowledge. Joe Thompson of SEA will lead the field sediment sampling effort. Mr. Thompson may be reached at jthompson@striplin.com and 360-705-3534. Ian Stupakoff of SEA will lead the fish tissue sampling efforts. Pam Sparks of SEA will lead the field benthic infauna sampling effort. Ms Sparks may be reached at psparks@striplin.com and 360-705-3534.

The Cruise Leader will be responsible for adherence to the FSP and QAPP, cruise preparation, mobilization, sample custody, storage, handling and shipping, and ensuring the correct completion of all field logs and chain-of-custody (COC) forms. Field decisions that involve changes to the FSP and QAPP will be coordinated with the Sampling and Analysis Coordinator, and other team members as appropriate. Recommended changes will be discussed with the EPA Project Manager.

The Site Safety Officer will have the following responsibilities:

- Correct any work practices/conditions that may result in personnel injury or exposure to hazardous materials
- Determine personal protection levels and necessary clothing/equipment, and oversee its proper use
- Verify that the field crew is aware of the provisions of the health and safety plan (HSP) and instructed in safe work practices
- Verify that the field crew has received the required safety training.

Various field staff from the consultant team will assist in sample collection, handling, and storage. Under the cruise leader's supervision they will maintain the field sampling logs and notebooks, and will be responsible for properly labeling containers for storage of chemical and biological samples.

A4.4 QUALITY ASSURANCE MANAGERS

Quality assurance (QA) managers have been assigned for all aspects of the Round 1 sampling and analysis. All quality assurance managers for Round 1 will report to the Sampling and Analysis Coordinator.

A4.4.1 Field QA Manager

Gene Revelas (SEA), the Sampling and Analysis Coordinator, will also serve as the Field Quality Assurance Manager for Round 1 sampling activities. He will oversee of the sampling events to ensure that the appropriate procedures and methods are used so that the QA objectives are met.

A4.4.2 Analytical Chemistry

Janet Cloutier (SEA) will be the QA Manager for analytical chemistry. She will perform laboratory oversight for the analytical laboratories and will direct the validation of chemical data. Ms. Cloutier may be reached at jcloutier@striplin.com and 360-705-3534.

A4.4.2.1 Analytical Laboratory Services

Three analytical chemistry laboratories were selected by the LWG for Round 1 analysis to ensure analytical capacity sufficient to maintain the schedule set forth in the AOC/SOW (EPA 2001a,b) and to take advantage of special analytical capabilities. One laboratory was selected to perform the benthic invertebrate sample sorting and taxonomic identification (QAPP section A4.4.3.1).

All laboratory SOPs will be provided to EPA under a separate cover. Due to confidentiality issues only one copy will be provided to EPA.

A4.4.2.1a Columbia Analytical Services (CAS)

Columbia Analytical Services (CAS) of Kelso, Washington, will perform tissue chemistry analysis (conventionals, metals, butyltins, polychlorinated biphenyl [PCB] Aroclors, and pesticides). CAS is a full-service chemical and analytical laboratory that is capable of performing work to Contract Laboratory Program (CLP) specifications. CAS staff has special expertise in the analysis of various complex matrices for inorganic and organic parameters. Project experience is demonstrated by participation in the EPA CLP, many RI/FS projects, and other projects supporting Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) activities. Abbie Spielman will be the CAS project manager. Ms. Spielman may be reached at 360-577-722. The QA manual for CAS is contained in Appendix A.

A4.4.2.1b Analytical Resources, Inc. (ARI)

Analytical Resources, Inc. (ARI) of Tukwila, Washington, will perform sediment chemical analyses and tissue semivolatile organic compound (SVOC) analyses. ARI is a full-service chemical analytical laboratory. ARI and its staff have special expertise in the analysis of various complex matrices for organic and inorganic parameters. Staff have participated in the development and review of methods found in the Puget Sound Estuary Program (PSEP) guidance. Project experience is demonstrated by participation in the EPA CLP, many RI/FS programs under CERCLA, and Dredged Material Management Program (DMMP) monitoring projects. Sue Dunninghoo will be the ARI project manager. Ms Dunninghoo may be reached at sue@arilabs.com and 206-695-6207. The QA manual for ARI is contained in Appendix B.

A4.4.2.1c Axys Analytical Services Ltd. (Axys)

Axys Analytical Services Ltd. (Axys) of Sidney, B.C., Canada, will perform analysis of dioxins/furans and PCB congeners in sediment and tissue samples. Axys is an ultra-trace organic laboratory that specializes in the analysis of these compounds. Axys will also perform all tissue homogenization. Dr. Coreen Hamilton will be the Axys project manager. Ms. Hamilton may be reached at chamilton@axys.com and 250-655-5800. The QA manual for Axys is contained in Appendix C.

These laboratories have demonstrated to the LWG that they have acceptable performance records and are capable of performing the analyses required. Back-up laboratories will be as follows: ARI and CAS will provide back up for each other due to the similarities in the chemical analyses being performed by these laboratories.

A4.4.3 Benthic Invertebrates

Pam Sparks (SEA) will be the QA Manager for benthic invertebrate analysis. She will perform laboratory oversight for the benthic laboratory and will direct the analysis of the benthic infauna samples.

A4.4.3 Benthic Laboratory

EcoAnalysts, Inc. of Moscow, ID will perform benthic invertebrate sample sorting and taxonomic identifications of both multiplate and sediment grab samples. The primary focus of EcoAnalysts is in the area of aquatic biology with an emphasis on benthic macroinvertebrates. Since their inception in 1994, EcoAnalysts has completed more than 200 projects from around the country and processed over 15,000 macroinvertebrate samples. Their project experience includes environmental monitoring programs and watershed assessments for EPA, U.S. Geological Survey (USGS), and the State of Idaho. Gary Lester will be the EcoAnalysts project manager. Mr. Lester may be reached at gglester@ecoanalysts.com and 208-882-2588. The benthic QA manual and the benthic community QAPP for the Round 1 Portland Harbor RI/FS are contained in Appendix D.

A4.4.4 Data Management

Tom Schulz (SEA) will have primary responsibility for data management. SEA will utilize the environmental quality information system (EQuIS) database as the primary repository of environmental data. Prior to the initiation of fieldwork, Mr. Schulz will work with the laboratories to ensure that they deliver data to SEA that is in the correct format for entry into the database. Use of this system will also ensure the easy transfer of data to EPA in the required format. Mr. Schulz may be reached at tschulz@striplin.com and 360-705-3534.

A4.4.5 Data Validation

Laboratory Data Consultants, Inc. (LDC) of Carlsbad, Ca, will perform the data validation for all matrices and analyses. LDC is an environmental chemistry and quality assurance company that focuses on data validation, data management and usability, and software development. Mr. Richard Amano will be the LDC project manager. Mr. Amano may be reached at richamano@aol.com and 760-634-0437.

A5 PROBLEM DEFINITION/BACKGROUND

A5.1 SITE BACKGROUND

The Initial Study Area (ISA) for the Portland Harbor Superfund Site extends from the southern tip of Sauvie Island at river mile (RM) 3.5 to the southern end of Swan Island at RM 9.2. 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 activities 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 (Striplin et al. 2002).

The purpose of the Portland Harbor RI/FS is to investigate the nature and extent of chemical concentrations for the in-water portion of the Portland Harbor Superfund Site (Site), to assess the potential risk to human health and ecological receptors, and to develop and evaluate potential remedial alternatives. A critical objective during the RI/FS will be 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 (benthic invertebrates, fish, shellfish, and piscivorous birds, and mammals, including those listed under the Endangered Species Act) and people that may eat fish or shellfish or come in contact with sediments from the Site.

This QAPP establishes the QA objectives for Round 1 sampling. The QA documentation provided in this QAPP will be followed by contractors for the LWG in conducting various sampling and data collection activities beginning in summer 2002.

The QAPP is designed to document the appropriate analytical methods and QA procedures for the collection and analysis of sediment and tissue. The goal of the QAPP is to ensure that data of sufficiently high quality are generated to support the project Data Quality Objectives (DQOs). DQOs are key components in the human health risk assessment (HHRA), ecological risk assessment (ERA), and nature and extent assessments for the Portland Harbor RI/FS.

The QAPP was written in accordance with *EPA Requirements for Quality Assurance Project Plans EPA QA/R5* (EPA 2001c), *EPA Guidance for Quality Assurance Project Plans EPA QA/G6* (EPA 2001d), *EPA Guidance for Quality Assurance Project Plans EPA QA/G5* (EPA 1998), and *Guidance for Conducting Remedial Investigations and Feasibility Studies under CERCLA* (EPA 1988). The analytical plan and associated quality assurance/quality control (QA/QC) procedures were also developed with consideration of the analytical protocols in the EPA CLP and PSEP (1986). The QAPP conforms to the requirements of the AOC (EPA 2001a) and the SOW (EPA 2001b).

A6 PROJECT/TASK DESCRIPTION

A6.1 ROUND 1

Round 1 sampling efforts will concentrate on the following field elements: sediment chemistry, tissue chemistry, and benthic infauna community structure. The rationale for the field elements is discussed in the Portland Harbor RI/FS Work Plan (Striplin et al. 2002). Most of the sampling activities will take place within the ISA; however, additional sampling will occur from about RM 2 to the ISA (RM 3.5) and beyond the ISA (from RM 9.2 to RM 10). The types of samples and objectives for each of the Round 1 data collection efforts are discussed below:

A6.1.1 Surface Sediment Chemistry

Surface sediment chemical data will be generated to support the following two objectives:

1. Surface sediment chemical data from areas where tissue samples of certain species are collected for ERA (i.e., species with small home ranges) will be used to help understand the relationships between sediment concentrations and those tissue concentrations.
2. Composite sediment samples will be collected from beach areas with known or potential human use to support the assessment of potential risks to human health associated with exposure to sediments.

A6.1.2 Tissue Chemistry

Tissue chemical data will be used in the HHRA and ERA to meet three objectives.

1. Tissue chemical residues in fish and crayfish consumed by people will be used in the HHRA to determine if there are unacceptable risks to human health associated with fish consumption.
2. Tissue residue levels for fish species and crayfish will be used in the ERA to assess risk to these species and wildlife species that consume them.
3. Limited benthic tissue samples, (i.e., clams) will be collected to assess risk to birds and fish through dietary exposure.

A6.1.3 Benthic Community

Benthic infauna community samples will be collected for the ERA. This information will be used to gain a better understanding of the types of benthic communities present in the ISA. The Round 1 benthic community assessment is intended to be exploratory in nature rather than highly quantitative.

A6.2 PROJECT SCHEDULE

Round 1 field sampling was initiated following EPA's conditional approval of the project FSP and QAPP and will be completed in fall 2002. Laboratory data will be delivered to EPA within 60 days of completing all Round 1 sampling, analysis, and validation. The draft Round 1 Site Characterization Summary will be delivered to EPA 120 days following completion of sampling, analysis and validation.

A7 QUALITY OBJECTIVES AND CRITERIA

A7.1 PURPOSE/BACKGROUND

Data needs for assessing the nature and extent of sediment chemical concentrations and human health and ecological risks, and for developing remedial alternatives for Portland Harbor sediments were identified based on a review of preliminary remedial action objectives (RAOs), historical data, and information developed as part of EPA's DQO process (EPA 2000a). A technical memorandum that presents preliminary RAOs for this site is found in Appendix A of the RI/FS Work Plan (Striplin et al. 2002).

The overall DQO for this project is to develop and implement procedures that will ensure the collection of representative data of known, acceptable, and defensible quality. The analytical and QA procedures described in this section are based on EPA guidance (EPA 1988, 1998, 2001c, and 2001d) and the project SOW (EPA 2001b) and reflect the intended use of data to be collected during Round 1. Tables A7-1, A7-2, and A7-3 show the QA/QC sample analysis requirements, frequency for calibration and quality control (QC), and corrective actions for Round 1 organics, metals, and conventional analyses, respectively. The DQOs, project specific method reporting limits (MRLs), EPA established analytical concentration goals (ACGs), and methodologies for each matrix are summarized in Tables A7-4 and A7-5.

Precision and accuracy will be determined by evaluating the results of matrix spikes, method blanks, and laboratory control samples. Acceptable limits for these measurements are found in Tables A7-6 and A7-7. Laboratories chosen for this project have demonstrated abilities in the form of previous projects with like matrices and detection limits. The ability to achieve stated MRLs is based on a combination of stated methodologies, laboratory standard operating procedures (SOPs), and MDL studies. All labs working on this RI/FS have internal, regulatory, and project QA requirements. Derivations from the QA requirements will prompt corrective action, which will be reported to the chemistry QA Manager. Deviations that may compromise the validity of the data, including exceedances of sample holding times and temperatures as well as QA samples that fall outside of established limits, will be reported to the chemistry QA Manager on the day of discovery.

A7.2 SPECIFYING DATA QUALITY OBJECTIVES

DQOs for the Portland Harbor RI/FS are generally described in this section. According to EPA (1998), DQOs are qualitative and quantitative statements that "clarify the intended use of the data, define the type of data needed to support the decision, identify the conditions under which the data should be collected, and

specify tolerable limits on the probability of making a decision error due to uncertainty in the data."

Preliminary RAOs that were used to identify the categories of data that would be needed to fulfill project objectives for the entire RI/FS include the following:

1. Reduce human health risks from direct contact with and incidental ingestion of contaminated sediments to acceptable levels.
2. Reduce human health risks from ingestion of contaminated fish to acceptable levels.
3. Reduce human health risks from direct contact and incidental ingestion of contaminated surface water to acceptable levels.
4. Reduce ecological health risks from ingestion of contaminated sediment to acceptable levels for fish, benthic invertebrates, birds, and mammals.
5. Reduce ecological health risks from ingestion of contaminated prey (fish and/or benthic invertebrates) to acceptable levels for fish, benthic invertebrates, birds, and mammals.
6. Reduce ecological health risks from ingestion of and contact with contaminated surface water to acceptable levels for fish, benthic invertebrates, birds, and mammals.
7. Promote remedial actions (RAs) that do not limit current or planned waterway, municipal, commercial, industrial, recreational, or tribal ceremonial uses of the river.
8. Promote RAs that are feasible for the physical system of Willamette River.
9. Integrate RAs with Natural Resource Damage Assessment (NRDA) findings and restoration plans.

It is anticipated that these preliminary RAOs will be refined throughout the data collection and evaluation phases of the project. The categories of data that will be required to complete the RI/FS include sediment and tissue chemistry, physical sediment characteristics, conventional parameters, habitat type and distribution, species occurrence, recreational and subsistence fishery use, hydrodynamic/sediment transport processes, sources (including upland and outside of the ISA), and source control status. This Round 1 QAPP addresses some but not all of the RAOs and data categories listed in the previous paragraph. The RI/FS is being implemented over multiple rounds of sampling.

Historical quantitative data of sufficient quality to support the Portland Harbor RI/FS were compiled in the project database and reviewed to identify specific data needs relative to the design of RI/FS field investigations and development of potential remedies. All data classified as Category 1 were considered appropriate for use as part of the RA process.

The quantitative historical data and general literature regarding aspects of the Site are summarized in Work Plan Sections 2.0 and 4.0 and include descriptions of regional geology, hydrology, topography, river morphology, sediment transport, dredging activities, ownership, land use, human development, and the results of various environmental investigations. Sediment chemistry data were obtained from site-specific investigations and dredging projects.

A significant amount of information, both quantitative and qualitative, exists for Portland Harbor, yet additional data are needed to support the RI/FS. Although numerous samples are available, not all analytes were measured in each sample, and there are areas of the river that have not been sampled or where data quality is not adequate to support an RI/FS. A subset (less than 20%) of the sediment chemistry data sets (primarily dredging studies) also had bioassay data. Only limited data (two surveys) were available to document the existing condition of macroinvertebrate communities in the ISA. Long-term monitoring data provided historical water chemistry for primarily conventional parameters. A few site-specific water quality data sets characterizing potential sources or releases were useable. Some industrial facilities undergoing site investigation under DEQ's oversight had groundwater data that were summarized as part of the historical database. Limited ecological studies, including tissue collection and analysis or habitat assessments, had been historically conducted in the ISA.

EPA's DQO process (EPA 2000a) was applied as part of the historical data evaluation to refine the specific data types needed to complete the RI/FS for Portland Harbor. The seven-step DQO process is designed to ensure that any data gaps, when filled, would meet the needs of the project. The seven-step DQO process documents the following:

1. Problems or issues that led to the investigation
2. Decisions to be made or questions to be answered
3. Inputs (i.e., types and source of data or information) to that decision
4. Spatial and temporal boundaries of the project
5. Decision rules or performance criteria used to evaluate the quality of the data and determine the outcome of the decision
6. Tolerable error relative to the decision rule
7. A sampling design and analysis plan that will collect the appropriate type and quality of data to meet the project objectives.

The following sections describe the issues, questions, and decisions associated with each data type necessary to determine chemical distributions, risks and remedies for the Site. Data needs that ensue from the DQO process form the basis

of the RI/FS sampling program. As noted above, the RI/FS will occur in multiple rounds and not all identified data needs will be pursued in the first round of sampling. The DQOs that were developed and presented in the project Work Plan (Striplin et al. 2002) are presented in Appendix F. These DQOs are considered draft DQOs and may be revised as the Work Plan is revised.

A7.3 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

Analytes, MRLs, and reference analytical methods are listed in Tables A7-4 and A7-5. Laboratories will use approved EPA, SW-846 and internal laboratory SOP methods for analysis. Analytical method modifications will be necessary to achieve MRLs. Any modification outside the stated methods or internal laboratory SOPs will be narrated in the final report. These low detection limits can also be expected to minimize reporting of non-detected values above more typical or routine quantification limits. The LWG understands there will be instances where high sample concentrations, non-homogeneity of samples, or sample matrix interferences preclude achieving the MRLs. As stated in EPA (1997) "current analytical methods may be unable to achieve detection limits at water quality criteria levels. These criteria levels should be considered target detection limits..." This statement is also applicable to sediment and tissues. Any limitation in data quality due to analytical problems will be clearly identified by the laboratory to the Chemistry QA Manager as soon as it is known. A copy of each analytical laboratory's digestion, extraction, cleanup and analysis SOPs have been provided to the EPA QA Manager and the LWG QA Manager. These are the stated SOPs that the laboratory will be using to achieve the MRLs listed in Tables A7-4 and A7-5. Due to confidentiality issues these documents will not be distributed more widely. The QA Managers will retain these copies until the end of the project.

A7.3.1 Specifying Measurement Performance Criteria

Tables A7-6 and A7-7 include project analytical goals for percent recoveries of spikes, surrogates, relative percent difference (RPD), and precision, accuracy, representativeness, completeness, and comparability (PARCC) parameters.

A7.3.2 Coordination with EPA for Lab Audits and Split Samples

Audits of chemical laboratories will be undertaken for each sampling round both prior to and during the analysis of samples. In the event that EPA or their designated representative wishes to accompany the LWG during these audits, the EPA Project Manager should make this request to the RI/FS Project Coordinator. Following this initial contact the appropriate QA Managers for the CERCLA team should interact directly with their counterparts at EPA.

Split and/or verification samples can be provided to EPA or their designated representative for chemical testing. EPA's Project Manager should contact the

RI/FS Project Coordinator to coordinate this activity and determine appropriate logistics. It is recommended that split samples be taken at those stations where blind duplicates and blind field replicates are taken so that EPA's comparison samples are evaluated relative to the field and analytical variability measured by the LWG.

A8 SPECIAL TRAINING/CERTIFICATION

The LWG has assembled a project team with the requisite experience and technical skills to successfully complete the Round 1 investigation. All consultant team personnel involved in sample collection have previous environmental sampling and analysis experience. Minimum training and certification requirements for laboratory personnel are described in the laboratory QA manuals (Appendices A through D).

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. 29 Code of Federal Regulation (CFR)§1910.120 requires training to provide employees with the knowledge and skills enabling them to perform their jobs safely and with minimum risk to their personal health. All sampling personnel will have completed the 40-hr HAZWOPER training course and 8-hour refresher courses, as necessary. The 40-hour course meets the U.S. Occupational Safety and Health Administration (OSHA) regulation 29CFR§1910.120(e)(3). Documentation of course completion will be required and copies will be maintained in personnel files.

A9 DOCUMENTS AND RECORDS

A9.1 FIELD OPERATIONS RECORDS

All field activities and observations will be noted in a field logbook during fieldwork. The field logbook will be a bound document containing individual field and sample log forms. Information will include personnel, date, time, station designation, sampler, types of samples collected, and general observations. Any changes that occur at the site (e.g., personnel, responsibilities, deviations from the Work Plan or FSP) and the reasons for these changes will be documented in the field logbook.

The field activities and observations will be clearly written with enough detail so that participants can reconstruct events later if necessary. Field logbooks will describe any changes that occur at the site, in particular personnel and responsibilities or deviations from the FSP as well as the reasons for the changes. Requirements for logbook entries will include the following:

- Logbooks will be bound, with consecutively numbered pages
- Removal of any pages, even if illegible, is prohibited
- Entries will be made legibly with black (or dark) waterproof ink
- Unbiased, accurate language will be used
- Entries will be made while activities are in progress, or as soon afterward as possible (the date and time that the notation is made should be noted, as well as the time of the observation itself)
- Each consecutive day's first entry will be made on a new, blank page
- The date and time, based on a 24-hour clock (e.g., 0900 a.m. for 9 a.m. and 2100 for 9 p.m.) will appear on each page
- When field activity is complete, the logbook will be entered into the permanent Portland Harbor project file.

In addition to the preceding requirements, the person recording the information must initial and date each page of the field logbook. If more than one individual makes entries on the same page, each recorder must initial and date each entry. The bottom of the page must be signed and dated by the individual who makes the last entry. The field team and task leader, after reading the day's entries, also must sign and date the last page of each daily entry in the field logbook.

Drawing a single line through the original entry, allowing the original entry to be read, will be the acceptable manner to make corrections to logbooks. The corrected entry will be written alongside the original. Corrections will be initialed and dated and may require a footnote for explanation.

The type of information that may be included in the field logbook and/or field data forms includes the following:

- Names of all field staff
- Sampling vessel
- A record of site health and safety meetings, updates and related monitoring
- Station name and location
- Date and collection time of each sample
- Observations made during sample collection, including weather conditions, complications, and other details associated with the sampling effort
- Sample description
- Depth of mudline below water surface
- River stage at the Morrison Street Bridge immediately prior to sampling
- Any deviation from the FSP.

A sample collection checklist will be produced prior to the initiation of the sampling program. It will be filled out after sampling operations have been completed at each station. The checklist will include station designations, types of samples to be collected (e.g., one jar for metals), and whether blind field replicates or additional sample volumes for laboratory QC analyses are to be collected.

Field data sheets and sample description forms (including core logs) will be completed for all samples and kept in the project file. Information such as habitat descriptions, sediment and biota sampling data will be noted on the field data sheets. Depending on the activity, the type of field data sheet and the information recorded on it may vary. Examples of the types of forms that may be used are provided in Appendix A of the FSP.

The cruise leader is responsible for ensuring that the field logbook and all field data forms are correct.

SECTION B

DATA GENERATION AND ACQUISITION

B1 SAMPLING PROCESS DESIGN AND SAMPLING METHODS

Complete sampling design and detailed sample collection and handling methods are described in the FSP. Station location maps are also contained in the FSP. The types and numbers of samples that will be collected, the rationale for collection, and the analysis that will be performed are discussed in the FSP.

In summary, sampling stations are located between RM 2 and RM 10 of the Willamette River. Numerous sample collection methods will be used. Surface sediments will be collected using a van Veen grab sampler or power grab. The upper 15 cm of sediment will be sampled and homogenized for chemical analysis (samples for analysis of volatile organic compounds will be jarred before the remainder of the sample is homogenized to prevent volatilization of compounds). Beach surface sediment will be collected using hand coring devices and analyzed for chemistry. Benthic infauna samples will be collected for community analysis and tissue chemistry. Lastly, eight fish species and crayfish will be collected using one or more of the following methods: electrofishing, beach seining, purse seining, hook and lines, trot lines, pots, or traps. Fish samples will be homogenized and analyzed for chemical concentrations. Detailed descriptions of sampling method requirements and field procedures, including QC procedures, are contained in the FSP.

B2 SAMPLING METHODS

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

Commercially available pre-cleaned jars will be used and the contractor will maintain a record of certification from the suppliers. The bottle shipment documentation will record 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 SEA project file. Field personnel are cautioned to not obstruct these stickers with sample labels.

Prior to shipment to the field, the project laboratories will add the required preservatives to the sample bottles and supply additional preservative in a transportable container. The laboratory will note on the bottle kit paperwork the lot number of the preservative placed in the bottles.

The MRLs for this project are notably lower than the certified levels to which manufacturers guarantee cleanliness. This drives the issue of possible contamination between MRLs and certified levels on the containers and reagent grade preservative chemicals. If this condition arises ($MRL \geq \text{analyte concentration} \leq \text{certified level}$) in the trip blank there will be a narration but no corrective action. If the analyte is noted in the sample and not in the blank then the assumption will be made that it is native to the sample and reported.

An appropriate amount of sample must be obtained in order for MRLs to be met, and precision and accuracy to be determined. Required sample volumes are listed in Table B2-1. All samples will be stored on ice in appropriate containers in the field. To achieve some of the low MRLs, sample volumes that exceed those normally required for SW-846 methods are necessary.

B3 SAMPLE HANDLING AND CUSTODY

B3.1 FIELD TO LABORATORY SAMPLE HANDLING AND CUSTODY

Detailed descriptions of sampling, documentation, custody procedures, and sampling locations for Round 1 are presented in Section 5 of the FSP.

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. Examples of laboratory COC forms are Figures B3-1, B3-2, and B3-3.

The field cruise leader, as the designated field sample custodian, will be responsible for all sample tracking and COC procedures for samples in the field. The sample custodian will be responsible for final sample inventory and will maintain sample custody documentation. The custodian will complete COC forms prior to removing samples from the sampling vessel. COC forms will be used for samples that are in route from the vessel to the sample processing or testing laboratories.

On completion of final inventory by the field sample custodian, each glass sample container will be placed into a "bubble wrap" plastic bag. Samples will then be placed into an ice chest. When the ice chest is full, the COC and the sample analysis request form will be placed into a zip-locked bag and taped onto the inside lid of the ice chest. Each ice chest will have three custody seals, one on the front of the chest and one on each side. On each side of the cooler a *This Side Up* arrow label will be attached; a *Handle with Care* label will be attached to the top of the cooler. Each ice chest will have COC seals and will be transported to the laboratory by car or commercial courier (e.g., FedEx). A commercial shipping invoice form will be used for international shipments (Figure B3-4).

Samples will have an air weigh bill that will follow the cooler while in the possession of a commercial carrier. This weigh bill will act as an intermediate COC. Shipment with signature requirements is mandatory. This process will allow the COC not to be interrupted. These packaging and shipping procedures are in accordance with U.S. Department of Transportation regulations as specified in 49 CFR 173.6 and 49 CFR 173.24. The laboratory sample custodian will establish the integrity of the seals at the laboratory. The coolers will be clearly labeled with sufficient information (name of project, time and date container was sealed, person sealing the cooler, and SEA's office name and address) to enable

positive identification. Copies of laboratory cooler receipts or sample log in sheets are provided in Figures B3-5, B3-6, and B3-7.

Each laboratory will have a stated process (SOP) for accepting custody and processing samples into the laboratory. The individual checking the samples into the laboratory system will ensure that the COC and sample tracking forms are properly completed, signed, and initialed on transfer of the samples. Each laboratory will deliver a copy of the COC and cooler 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.

The laboratory will check for physical integrity of the containers and seals and then inventory the samples by comparing sample labels to those on the COC document. The laboratory will enter the sample number into a laboratory tracking system by project code and sample designation. The laboratory will assign a unique laboratory number to each sample and will be responsible for distributing the samples to the appropriate analyst or for storing samples in an appropriate secure area. Specific laboratory COC procedures are described in the laboratory QA Plans for each of the designated labs (Appendices A-D).

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 that will need further analysis that is not performed by the initial contracted laboratory will be subject to all specifications in the previous section.

B3.3 ARCHIVED SAMPLES

All excess samples submitted to the chemical laboratory will be archived at $-20\pm 4^{\circ}\text{C}$. 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 excess samples for 6 months following completion of data validation. The laboratories will not dispose of the samples for this project until they are authorized to do so by the Sampling and Analysis Coordinator and EPA.

B4 ANALYTICAL METHODS

The analytical methods, QC measurements and criteria for all sample types are based on current SW-846, EPA method requirements, MRLs for the RI/FS, and PSEP guidance. Laboratory deliverables will be consistent with the requirements of the full CLP package. Electronic data will be provided by each laboratory in the format specified for entry into the EQUIS data management system. The laboratory QAPP and SOPs provide data quality procedures according to the protocols for the analytical method. Analytical methods are listed in Table B4-1 and are briefly described below.

B4.1 ANALYTICAL METHODS – SEDIMENTS AND TISSUE

B4.1.1 Physical Parameters - Sediment

B4.1.1a Total Solids

Total solids will be determined according to EPA-160.3/SM 2540B. These results will be used to back calculate and arrive at the amount of material needed in the extract to achieve the QAPP stated MRL.

B4.1.1b Grain Size

Grain-size analysis will be accomplished using guidance from PSEP protocols (EPA 1986). Eight class fractions and apparent grain size will be determined by not employing the peroxide oxidation option. Results will be expressed by class percentage (reportable to 0.01 percent) in the fractions listed below.

Particle size will be determined using PSEP (1986) which subdivide the silt-clay fraction by pipette and hydrometer. The following sieve series must be used: 4, 10, 18, 35, 60, 120, 230. The fine-grained fraction must be classified by phi size (+5, +6, +7, +8, >8). Results also will be presented as curves on semi-logarithmic graphs by plotting percent fines by weight versus grain size and in tabular formats [refer to U.S. Army Corps of Engineers manual EM-1110-2-1906, Appendix V(i), Presentation of Results Plate V-2].

B4.1.2 Physical Parameters - Lipids - Tissue

All lipid analysis will be performed at CAS. A mid-method lipid extraction will occur in the pesticide extraction. The EPA Manchester lab SOP (Appendix F) is a guidance document for the lipids extraction that is within the CAS SOP. The method describes the ability to sub-sample from an existing organic Soxhlet extraction. The laboratory producing percent lipid data will sub-sample from a PCB or pesticide extraction (Reimer 2002). An aliquot of 20-50% of the final extract volume, depending upon the residual volume needed to achieve MRLs of the intended analytes, will be taken and percent lipids determined. The laboratory SOP for mid-method percent lipid and explanation of solvent determination will be supplied to EPA.

B4.1.3 Tissue Homogenization Procedures for HHRA

Fish will be composited and homogenized according to methods presented in Appendices G and H.

B4.1.3a Scaling or Skinning

With the exception of bullhead, whole fish that will be used for fillet tissue samples will be scaled or skinned prior to filleting. To control contamination, separate sets of utensils and cutting boards will be used for skinning or scaling fish and for filleting fish. Fish with scales will be scaled and any adhering slime removed prior to filleting. Fish without scales (i.e., yellow or brown bullhead) will be skinned prior to filleting. Bullhead will be processed according to the following steps as outlined in the fish processing SOP:

- Whole body with skin.
- Fillet for all analysis, except Hg: no skin, but belly flap
- Fillet for Hg: no skin, no belly flap.

A fish will be scaled by laying it flat on a clean glass or polytetrafluoroethylene (PTFE) cutting board or on one that has been covered with heavy duty aluminum foil and removing the scales and adhering slime by scraping from the tail to the head using the blade edge of a clean stainless steel, knife. Cross-contamination will be controlled by rinsing the cutting board and knife with contaminant-free distilled water between individual fish. If an aluminum-foil-covered cutting board is used, the foil will be changed between fish. The skin will be removed from fish without scales by loosening the skin just behind the gills and pulling it off between knife blade and thumb or with pliers. Once the scales and slime have been scraped off or the skin removed, the outside of the fish will be washed with contaminant-free distilled water. The fish will then be placed on a second clean cutting board for filleting.

B4.1.3b Preparation of Fillets

Filleting will be conducted by or under the supervision of an experienced fisheries biologist. Gloves, if worn, will be talc- or dust-free, and of non-contaminating materials.

Prior to filleting, hands will be washed with Ivory soap and rinsed thoroughly in tap water, followed by distilled water (EPA 1991). Fish will be filleted on glass or PTFE cutting boards that are cleaned properly between fish or on cutting boards covered with heavy-duty aluminum foil that is changed between fish (EPA 1990a and EPA 1990b). Care will be taken to avoid contaminating fillet tissues with material released from inadvertent puncture of internal organs. If the fillet tissue becomes contaminated, during resection the fillet materials released from the inadvertent puncture of the internal organs may eliminate tissue eliminated as a sample or, alternatively, the fillet tissue may be rinsed in contaminant-free, deionized distilled water and blotted dry. The fisheries biologist will decide which procedure is appropriate. Regardless of the procedure selected, a notation will be made in the sample processing record.

Fish will be filleted prior to freezing.

Clean, high-quality stainless steel utensils will be used to remove one or both fillets from each fish, as necessary. The general procedure recommended for filleting fish, after scaling or skinning the fish, is as follows (EPA 1991):

- A shallow cut will be made through the skin on either side of the dorsal fin from the top of the head to the base of the tail.
- A cut will be made the entire length of the gill cover through the skin and flesh to the bone.
- A shallow cut will be made along the belly from the base of the pectoral fin to the tail. A single cut will then be made from behind the gill cover to the anus and then a cut will be made on both sides of the anal fin. While making the cuts, extreme care will be taken not to cut into the gut cavity as this could contaminate fillet tissues.
- The fillet will be removed.

The belly flap will be included in one fillet from each fish. Any dark muscle tissue in the vicinity of the lateral line will not be separated from the light muscle tissue that constitutes the rest of the muscle tissue mass.

Bones still present in the tissue after filleting will be removed carefully (EPA 1991).

Both fillets will be removed from a fish. One fillet, which contains the belly flap, will be used for organic analysis. The other fillet, which will be skinned and does

not contain the belly flap, will be used for the mercury analysis. Fillets will be weighed individually and the weight recorded to the nearest gram on the sample processing record.

Fillets will be wrapped and stored in aluminum foil inside a Ziploc bag.

B4.1.3c Preparation of Composites

Sample composites will be prepared using the guidance found in Appendix G.

The weights of fish composites need to yield an adequate size to perform all necessary analysis. The sample volume will be $\geq 300\text{g}$ if actual catch volumes provide this amount. The recommended sample size of $\geq 300\text{g}$ is intended to provide sufficient sample material to analyze for target analytes at appropriate detection limits. To meet minimum QA and QC requirements for the analysis of replicate, matrix spike (MS), and matrix spike duplicates (MSD), and allow for reanalysis if the QA and QC control limits are not met or if the sample is lost, more tissue will be necessary. It is noted that certain species are large and will yield substantially more tissue than their smaller counterparts. The larger species with extra tissue volume will be used to perform the QA/QC for the batches of tissue. If there is a limited volume of tissue for analysis then the QA Manager will be so notified and may, with concurrence from EPA, direct the laboratory to conduct co-extractions of dioxin/furans and PCB congeners.

B4.1.3d Preparation of Homogenates

To ensure even distribution of chemistry throughout tissue samples, whole fish must be ground and homogenized prior to analysis. Tissue will be homogenized as described in Appendix H.

B4.1.4 Tissue Homogenization Procedures for ERA

Fish will be composited and homogenized according to the methods presented in Appendices G and H.

B4.1.4a Preparation of Composites

Composites will be prepared using the guidance found in Appendix G.

B4.1.4b Preparation of Homogenates

To ensure even distribution of chemistry throughout tissue samples, whole fish must be ground and homogenized prior to analysis. Tissue will be homogenized as described in Appendix H.

The weights of fish composites need to yield an adequate size to perform all necessary analysis. The sample volume will be $\geq 300\text{g}$ if actual catch volumes provide. The recommended sample size of $\geq 300\text{g}$ is intended to provide sufficient sample material to analyze for target analytes at appropriate detection limits. To meet minimum QA and QC requirements for the analysis of replicate,

matrix spike (MS), and matrix spike duplicates (MSD), and allow for reanalysis if the QA and QC control limits are not met or if the sample is lost more tissue will be necessary. It is noted that certain species are large and will yield substantially more tissue than their smaller counterparts. The larger species with extra tissue volume will be used to perform the QA/QC for the batches of tissue. If there is a limited volume of tissue for analysis then the QA Manager will be so notified and may, with EPA concurrence, direct the laboratory to perform co-extractions of dioxin/furans and PCB congeners.

B4.1.5 Conventional Parameters

B4.1.5a Total Organic Carbon (TOC) Sediment Only

Total organic carbon (TOC) will be determined according to EPA (PSEP 1986) using Plumb, 1981. Sample pretreatment with HCl is required to liberate inorganic carbon (primarily carbonates) prior to carbon analysis. Carbon analysis is through sample oxidation at 850°C followed by CO₂ measurement by infrared spectrophotometry. Results are expressed in terms of carbon per dry weight of the un-acidified sample.

B4.1.6 Metals - Sediment and Tissue

Metals will be analyzed under clean laboratory conditions by a method requiring acid digestion prior to instrumental analysis. Instrumental analysis is accomplished using methods SW-846 6010B, 6020, 7761, 7060A, 7131A, 7421, 7041, and 7040. All samples run by GFAA will be double runs/injection except for the Method of Standard Additions. The %RSD between burns must be within the method's acceptable limits. If %RSDs are not met, samples will require re-analysis. The post digestion spikes will be performed on all samples, method blanks and laboratory control samples to determine if matrix effects are occurring during analysis. The spike recovery requirements are based on the concentration results and analysis protocol.

B4.1.6a Mercury (Hg) - Sediment and Tissue

Mercury will be analyzed using M7471A. In accordance with the SW-846, the QC reporting requirements will accompany all analyses. Per direction from Mr. Chip Humphrey of EPA Region 10, mercury hold-times have been extended to 6 months in accordance with EPA Guidance on Fish Sampling and Analysis. (EPA 2000c, Humphrey 2002).

B4.1.7 Organics - Sediment and Tissue

B4.1.7.1 Volatile Organics – Sediment only

The analyses will follow methodology found in SW-846-8260B and may employ modifications as the laboratories deem necessary to achieve the project DQOs. These modifications, if outside the laboratory SOP, will be narrated in the final report.

B4.1.7.2 Semi-volatile Organics - Sediment, and Tissue

Semivolatile organic compounds (SVOC) will be analyzed by the methodology found in SW-846-8270C full scan with certain modifications to achieve the projects DQOs. These modifications, if outside the laboratory SOP, will be narrated in the final report. These modifications may include the following:

- High volume injection followed by selective ion monitoring (SIM) analysis (see Tables A7-4 and A7-5)
- Gel permeation chromatography (GPC) cleanup for removal of high molecular weight hydrocarbons, lipids, and elemental sulfur (Sx). The laboratory will use GPC on sediments and tissue. Where lipid content is greater than 10% the laboratory will perform GPC clean-up twice.
- Final extract volumes adjusted to yield sufficient sensitivity and instrumental response without overloading
- Gas chromatography/mass spectrometry (GC/MS) initial calibration will be established with a 7 point calibration curve, of which at least one of the low standards must be at the project MRL. If the low standard is not at the project MRL then the laboratory will run a daily MRL check..
- Continuing calibration for all target analytes and surrogate spike compounds
- MS/MSD analysis for all of the listed spiking compounds
- Laboratory control spikes and spike duplicates (LCS/LCSD) analyses for all of the listed spiking compounds.

B4.1.7.2a SVOCs and PAHs – Sediment and Tissue

Samples for SVOC analyses will be initially analyzed using the full scan SW846-Method 8270C. If the ACG for any target compound is met with the full scan there need not be any further action. If there is not an ACG established then the laboratory stated level will be sufficient. If the ACG is not met the laboratory will analyze the sample with SIM in order to align the MRL closer to the ACG. If high concentrations of PAH or other stated target compounds (1,2,4-trichlorobenzene, 1,2-diphenyl hydrazine, 2,4-dinitrotoluene, 2,6-dinitrotoluene, 2-chloronaphthalene, 4-bromophenyl phenyl ether, 4-chlorophenyl phenyl ether, 1,2- 1,3- and 1,4-dichlorobenzene, hexachlorobenzene, hexachlorobutadiene, hexachloroethane, bis(2-chloroisopropyl)ether and nitrobenzene) are detected in the sample (concentrations greater than 5 times the MRL), the sample will not be further analyzed using the modified Method 8270C-SIM. In cases like these, the laboratory will report the results off the full scan Method 8270C. For PAHs and other stated target compounds that fall below 5x MRL, samples will be analyzed by 8270C-SIM. The data generated from the SIM run will be used in the risk assessment.

The MRLs listed in Table A7-5 may be updated in the near future resulting from ongoing research at Analytical Resources, Inc. If so, the LWG will provide EPA with the updated MRLs prior to initiation of sample analysis.

B4.1.7.2b Pentachlorophenol (PCP) and Hexachloroethane - Sediment and Tissue

Pentachlorophenol and hexachloroethane will be analyzed by method SW846-Method 8151A and SW846-Method 8270C SIM. This will allow the MRL to be at its lowest (8151A) and allow for matrix difficulties if there is an elevated concentration (8270C-SIMs).

B4.1.7.3 Chlorinated Pesticides and PCB-Aroclors – Sediment and Tissue

Chlorinated pesticides and PCB-Aroclors will be analyzed using methods SW-846-8081A & 8082. Modifications to sample size and methodology will depend upon project-specified MRL and laboratory SOPs. These modifications, if outside the laboratory SOP, will be narrated in the final report. However, the following modifications are recommended for cleanup of sample matrix:

- Elemental S_x is removed from the sample extract during GPC cleanup. Additional S_x removal using chemical agents may be required.
- Column chromatography (Florisil® and/or alumina) of extracts is required and not discretionary. A pre-screening of the samples is recommended to assure that the laboratory can adjust the sample size so there is no break through and chromatographic column overloading. The laboratory should also run Florisil® and/or alumina lot checks. The laboratory will maintain records detailing all screening steps and lot checks.

B4.1.7.4 PCB-congeners (Sediment and Tissue HHRA)

PCB-congeners will be analyzed by EPA method 1668A: Chlorinated Biphenyl Congeners in sediment, and tissue by high-resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS). Modifications to sample size and methodology will depend upon project-specified MRL and laboratory SOPs. These modifications, if outside the laboratory SOP, will be narrated in the final report.

B4.1.7.5 Herbicides (Sediment HHRA)

Herbicides will be analyzed SW-846-8151A. Modifications to sample size and methodology will depend upon project-specified MRL and laboratory SOPs. These modifications, if outside the laboratory SOP, will be narrated in the final report.

B4.1.7.6 Dioxin and Furan (Sediment and Tissue HHRA)

Dioxin and furan will be analyzed EPA M1613 rev. B employing some modifications as the laboratories deem necessary to achieve the project DQOs. These modifications, if outside the laboratory SOP, will be narrated in the final report.

B4.1.7.7 Butyl Tins (Sediment and Tissue HHRA)

Butyl Tins will be analyzed using independent laboratory-identified methods employing some modifications, as the laboratories deem necessary to achieve the project DQOs. These modifications, if outside the laboratory SOP, will be narrated in the final report.

B5 QUALITY CONTROL

B5.1 FIELD QUALITY CONTROL

QC samples are collected in the field and used to evaluate the validity of the field sampling effort. Field QC samples are collected for laboratory analysis to check sampling and analytical precision, accuracy, and representativeness. The following section discusses the types and purpose of field QC samples that will be collected for this project. Tables A7-1, A7-2, A7-3, and B5-1 provide a summary of the types and frequency of collections of field QC samples.

B5.1.1 Field-Audits

Audits for field performance will be conducted at least once during each field program. The audits will involve evaluating the sample collection and processing procedures relative to the procedures described in the project FSP and QAPP and relative to standard procedures for collection of sediment and tissue samples. Data recording procedures will be reviewed for completeness.

Results of the field performance audit may identify the need for corrective actions. If this occurs, the Field QA Manager will immediately institute the necessary corrective actions (see Figure B5-1 for field corrective action form) and will conduct an additional audit to ensure that the correct procedures continue to be followed.

B5.1.2 Field Quality Control Samples

Field QC samples are used to assess sample variability (e.g., replicates), evaluate potential sources of contamination (e.g., rinsate and trip blanks), or confirm proper storage conditions (e.g., temperature blanks). The types of QC samples that will be collected in Round 1 are described in this section and summarized in Table 5-3 of the FSP. The types of field QC samples that will be collected in Round 1 are described in the following sections

B5.1.3 Replicate Samples for Sediment

Field replicates are additional samples collected at a station to enable statistical analysis of the resulting data. Their origin is not revealed to the laboratory (hence the term blind). Collecting new sediment at the sampling location will generate replicate samples. These data will be used to determine natural variability associated with the environment and laboratory operations.

At approximately 5% of the sediment sampling stations, replicate samples will be collected. All replicates will be submitted to the laboratory blind. The frequency of replicate sediment samples at stations with co-located sediment chemistry and tissue chemistry will be 10 %.

B5.1.4 Field Splits

Blind field splits will also be generated for sediment samples at the same stations as the blind field replicates. These sediment samples will be taken from the same composite sample as the field sample. The resulting data will provide information on the variability associated with sample handling and laboratory analysis operations. Blind field samples will be generated at approximately 10% of the sediment chemistry stations.

B5.1.5 Replicate Samples for Tissue

For the ERA, duplicate tissue samples will be collected for a minimum of 10% of the samples.

For the HHRA, triplicate samples will be collected at all of the sampling locations. EPA guidance for fish advisories recommends that duplicate composite samples should be collected at a minimum of 10% of the sampling locations (EPA 2000b). To provide a more robust database, three composite samples will be collected as replicates at all sampling locations. By collecting triplicate samples at all locations, the sampling design for the HHRA far exceeds EPA's minimum recommendations.

Additional information on procedures for collecting and compositing replicate tissue samples for the HHRA is provided in the FSP Section 5.6.6.

B5.1.6 Temperature Blanks

Temperature blanks are used to measure and ensure cooler temperature upon receipt at the laboratory. One temperature blank per cooler will be prepared and submitted to the project laboratory. The temperature blank will consist of sample jar containing de-ionized water, which will be packed into the cooler in the same manner as the rest of the samples and labeled "temp blank". This temperature will be noted on the cooler receipt form.

B5.1.7 Field Blanks

B5.1.7a Field Equipment Blanks

Field equipment blanks will be used to assess the introduction of chemical contaminants during sampling and field processing activities. Field equipment blanks will consist of rinsate blanks collected by pouring anywhere from 3-6L of de-ionized water over or through decontaminated sampling equipment and collected in the appropriate sample containers (1L amber glass). Equipment surfaces exposed during actual sampling will be rinsed. These samples will be analyzed along with the field samples. No rinsate blanks will be collected from disposable field equipment. Field equipment rinsate blanks will be generated for all chemical parameter groups at approximately 5 % of the stations and submitted for analysis to the laboratory.

B5.1.7b Field Laboratory Equipment Blanks

EPA Region 10 has approved Axys Analytical Services Ltd SOP for homogenization and decontamination of equipment. This lab is the current lab performing this same routine for the EPA's National Fish Study. Due to the clean lab procedures and adherence to internal SOPs routinely employed by Axys, the LWG will not be collecting equipment blanks from the tissue homogenizers for the remainder of this project.

B5.1.8 Field Trip Blanks

Field trip blanks will be used to determine if VOCs are introduced to samples during holding, shipping, or storage prior to analysis. Field trip blanks will consist of deionized water sealed in a sample container by the analytical laboratory. The trip blank will be generated and transported to and from the field and then returned to the laboratory unopened for analysis. One trip blank will be included with each cooler containing sample for VOA.

B5.2 ANALYTICAL LABORATORY QUALITY CONTROL

QC procedures for laboratory analysis will be consistent with the requirements described in each laboratory's protocols and methods. These requirements are also presented in SOPs (that are being submitted to the EPA QA Manager) as part of the laboratory's QA program (see Appendices A-D). Methods for establishing the quality of laboratory measurements and sample results will generally conform to limits set in Tables A7-6 and A7-7. Additional QC measurements will be made and reported for purposes of evaluating data quality specific to this project. *Some modifications have been made to 1) expand the range of instrumental calibrations, 2) reduce quantification limits, and 3) establish precision at quantification levels below those of CLP. These changes are necessary to meet the DQOs for this project.* Data validation and reporting of data quality will use the guidance of the EPA data validation functional guidelines for in-organics, organics, and dioxins (EPA 1994, 1999, 2001e, 2002). All QC measurements and data assessment for this project will be conducted on samples from this project alone; samples from other projects will not be mixed with samples from this project for assessment of data quality.

The Chemistry QA Manager will oversee the activities of all analytical chemistry support employed in this project. Oversight will be achieved through on-site inspections and reviews of analytical facilities prior to and during analysis of project samples.

Prior to initiating laboratory analysis, a QA evaluation and evidentiary audit of the laboratories will be performed in a manner similar to those procedures used for a CLP-type systems audit. CLP guidance and the laboratory QAPP and SOPs will be used as references for performing on-site laboratory evaluations. Continuing performance audits will be conducted on a regular basis to ensure the

laboratories are providing data of known and sufficient quality. As an independent assessment of the analytical process, independent commercial analytical reference materials (where available for the analytes of concern at appropriate concentrations) will be used, at a minimum, at the beginning and end of each task or phase of the project. The frequency of on-site audits depends on the type of interaction and communications the Chemistry QA Manager experiences with the laboratory staff and on the frequency of observations of noncompliance with QC criteria and SOPs. The Chemistry QA Manager's interaction with the laboratories will be focused on coordination, management, and assessment of performance, and on the rapid institution of corrective actions, if required.

B5.2.1 Internal Quality Control Samples

QC samples are used to evaluate PARCC parameters for analytical results. Analytical methods specify routine procedures that are required to evaluate if data are within proper QC limits. Additional internal QC includes collection and analysis of field and laboratory QC samples. These are described in the sections below and summarized in Tables A7-1, A7-2, A7-3, and B5-1.

B5.2.2 Method Reporting Limit Check

To ensure that laboratory instrumentation can achieve the required MRL from DQOs that are set forth in Tables A7-4 and A7-5. If the initial calibration curve contains a standard at the MRL the laboratory may forgo analyzing a daily MRL check standard. If not, the laboratory will run a MRL check standard per analytical sequence. This sample will be after the instrument blank check sample and prior to analyzing samples from this group. The instrument must be able to achieve the requested MRLs without interference. If the instrument cannot achieve these levels the samples must be analyzed on a different instrument that is able to achieve the required MRLs for this project.

B5.2.3 Method Blanks

Introduction of contaminants during sampling and analytical activities will be assessed by the analysis of blanks. Method blanks are used to check for laboratory contamination and instrument bias. Laboratory method blanks will be analyzed at a minimum of frequency of 5% or one per analytical batch for all chemical parameter groups.

B5.2.4 Laboratory Duplicates

Sample analytical variability and laboratory precision and accuracy will be determined by the analysis of laboratory generated sample splits at a frequency of 5% or once per batch of 20 samples. The duplicate results will be used for determination of RPDs. Variabilities in organic compound analysis will be evaluated by analysis of MS and MSD samples. Duplicate samples for inorganic

analysis will be analyzed at a frequency of 5%. Conventional parameters also will be analyzed in duplicate at a frequency of 5%. Precision and accuracy information will be generated for dioxins and furans using the on-going precision and recovery samples run per the method. See Tables A7-1, A7-2, and A7-3 for associated sample QC requirements and Tables A7-6 and A7-7 for QC objectives and limits for analysis of laboratory duplicates.

B5.2.5 Surrogate Spikes

Surrogate compound analysis for organics also will follow the guidance in the laboratories SOPs and will evaluate the laboratories ability to recover the analytes of interest. If data fall outside the established limits for the surrogates a corrective action must be implemented, and the Chemistry QA Manager notified. The corrective action can range from re-analysis to re-extraction/re-analysis of the sample. If after these actions the surrogates are still outside of established limits it will be considered matrix effects and narrated in the final report.

Qualification of data will occur when organic compound surrogate recoveries fall outside acceptance limits and will be noted in the laboratory case narrative. Criteria and requirements, summarized in Tables A7-6 and A7-7, will be employed for the analysis conducted in this program and will be used to support the evaluation of laboratory results during data validation.

B5.2.6 Laboratory Control Samples

Laboratory control samples (LCS) are used to monitor the laboratory's day-to-day performance of routine analytical methods independent of matrix effects. In this Round 1 sampling effort an LCS/LCSD will be analyzed with each batch of organic and inorganic analysis. This should provide usable precision and accuracy measurements for each batch. For inorganic samples a standard reference material (SRM) will also be run. If the laboratory runs a blank spike and blank spike duplicate for organics then they will also run an appropriate SRM.

B5.2.7 Matrix Spike and Matrix Spike Duplicates

Matrix Spike (MS) and matrix spike duplicates (MSD) samples provide information to assess precision and accuracy. The laboratory will follow EPA guidance for MS/MSD sample analysis. Percent recoveries, including relative percent difference (RPD) will be assessed for organics from the MS/MSD and for inorganics from the MS. MS/MSD recovery will be measured at a minimum frequency of 5% or one per batch of up to 20 samples.

B6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

B6.1 PREVENTATIVE MAINTENANCE AND CALIBRATION PROCEDURES

Analytical instrument testing, inspection, maintenance, setup, and calibration will be conducted in accordance with the QC requirements identified in each laboratory's SOPs. In addition, each of the specified analytical methods provides protocols for proper instrument calibration, setup, and critical operating parameters.

Preventive maintenance in the laboratory will be the responsibility of the laboratory personnel and analysts. At a minimum, the preventative maintenance schedules contained in the EPA methods and laboratory SOPs and in the equipment manufacturer's instructions will be followed. This maintenance includes routine care and cleaning of instruments, and inspection and monitoring of carrier gases, reagents, solvents, reference materials, and glassware used in analysis. All maintenance of instruments and procedures will be documented in maintenance log/record books. Each of the laboratories has SOPs for preventive maintenance that is contained in their individual QA manuals.

B7 INSTRUMENT/EQUIPMENT CALIBRATION AND FREQUENCY

B7.1 CALIBRATION 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 (Tables A7-1, A7-2 and A7-3). Instrument calibration procedures and schedules will conform to analytical protocol requirements and are described in the laboratories' QA manuals.

B7.1a Calibration Standards

All calibration standards will be obtained from either the EPA repository or a commercial vendor and traceability back to NIST will be provided by the labs. Stock solutions for surrogate parameters and other inorganic mixes will be made from reagent-grade chemicals or as specified in the method SOP. Stock standards will also be used to make intermediate standards from which calibration standards are made. Special attention will be given to expiration dating, proper labeling, proper refrigeration, and freedom from contamination. Documentation relating to the receipt, mixing, and use of standards will be recorded in the appropriate laboratory logbook. Logbooks must be bound. Specific handling and documentation requirements for the use of standards will be provided in the selected laboratory's QA manual (Appendices A-D).

B8 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

Sample container requirements are discussed in Section B2. Other supplies include deionized water, chemicals for decontamination, and personal protective equipment. All 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 kept in project files

B9 NON-DIRECT MEASUREMENTS

Existing chemical and biological data from previous investigations in the Lower Willamette River (LWR) were compiled from historical databases, scientific literature, and technical reports. All data were reviewed for quality assurance prior to entry in the project database. The historic data, results of the QA review, and acceptance criteria for use, are described in the Work Plan (Striplin et al. 2002). The historic data meeting QA requirements were one of several elements considered in developing the Round 1 sampling plan and identifying target analytes.

B10 DATA MANAGEMENT

B10.1 DATA REPORTING

Analytical reports from the laboratory will include QC results and any other necessary analytical information to enable reviewers to determine the quality of the data. Initial data reduction, evaluation, and reporting performed at the laboratory will be in conformance with this QAPP for organic and inorganic analysis. Analytical data will be reported in the units specified in Tables A7-4 and A7-5.

Data will be delivered in both hardcopy and electronic format to the Chemistry QA Manager; who will be responsible for distributing it to the data validator and for permanent archival. Hard copy deliverables will be similar in format and content to those required for CLP. Electronic data deliverables must be compatible with SEA's EQuIS database (Figure B10-1 describes SEA EQuIS format). EPA will also receive electronic data compatible with NOAA Query Manager format. Hardcopy data deliverables and documentation will be archived for all laboratory results and procedures and will be made available to EPA upon request.

Reporting requirements will include at least the following:

For inorganic and organic analytes (where applicable for each analysis):

- Tabulated results for samples and QC samples
- Narrative referencing or describing the procedure used and discussing any analytical problems
- Reconstructed ion chromatograms for GC/MS analysis for each sample, mass spectra of detected target compounds for each sample, and associated library spectra
- Enhanced and un-enhanced mass spectra of detected target compounds for each sample and associated library spectra
- Internal standard and surrogate compound performances
- Gas chromatograph/electron capture detector (GC/ECD) chromatograms for each sample
- Raw data quantification reports for each sample
- Sample extraction, dilution, and cleanup logs
- A calibration data summary reporting calibration range used [and decafluorotriphenylphosphine (DFTPP) and bromofluorbenzene (BFB) –define acronyms spectra and quantification report for GC/MS analysis]
- GC/MS instrument data files in binary code (on CD-ROM)
- Second source calibration verification data

- Initial Calibration Summary
- Continuing Calibration Standard Summary
- Initial Precision and Recovery (IPR) Summary
- On-going Precision and Recovery Summary
- GC instrument data files on CD-ROM
- Method Blank Summary
- Selected Ion Current Profile (SICP) of each PCDD/PCDF isomer with the corresponding polychlorinated diphenyl ether (PCDE) isomer as listed in Table 8 of Method 1613B.

For inorganic analytes

- Narrative referencing or describing the procedure used and discussing any analytical problems
- Tabulated results for each sample in units as specified for each matrix according to the analytical protocol, approved and signed by the section manager
- Any data qualifications and explanation for any variance from the specified analytical protocols
- Results for all of the QA/QC checks performed.

The remainder of the deliverable requirements for both organics and in-organics are addressed within the electronic deliverable requirement for compatibility with EQUIS.

The laboratory will assign data flags, or qualifiers, following independent laboratory defined flags for organic and inorganic analysis. The laboratories are required to immediately notify the Chemistry QA Manager when any QC measurements are consistently outside of project QC criteria or DQOs. The problem will be reviewed to determine the causes and to implement a remedy.

An independent third party data validator will be responsible for data validation. Data validation and reporting will be accomplished for all analytical parameters including conventional analytes. The organics data will be evaluated in general accordance with EPA's *Laboratory Data Validation Functional Guidelines for Evaluating Organics Analysis* (EPA 1999). CLP inorganics data will be validated in general accordance with EPA's *Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analysis* (EPA 1994). Dioxin/furan data will be evaluated in general accordance with EPA's *National Functional Guidelines for Chlorinated Dioxin/Furan Data Review* (EPA 2002). Modifications will be made to the functional guidelines to accommodate QA/QC requirements of the non-CLP methods that will be used for this project.

A tiered data validation using both partial and full validation will occur. The first 5% of the data per suite of parameters will be fully validated by EPA's QA office. The LWG will be submitting the following laboratory data deliverables to EPA's QA office:

- QC Summary Forms
 - surrogate recovery forms
 - matrix spike and matrix spike duplicate forms
 - method blanks summary forms
 - Instrument performance check forms
 - initial calibration forms
 - continuing calibration check forms
 - internal standard area and retention times forms
- Target Compound Results and sample data
 - reconstructed ion chromatograms
 - quantitation list, EICP for manual integrations
- Enhanced and un-enhanced mass spectra of detected compounds
- Standards data
- QC Data for the instrument
 - performance checks
 - method blanks
 - laboratory control samples
 - matrix spike and duplicate analytical runs
- COC, shipment and other sample control documentation
- Statement of Work or SOPs.

The next 25% of the data is recommended to be fully validated by LWG's third party data reviewer.

The remainder of the sample delivery groups will be partially validated. Five percent of the partially validated data will be peer reviewed by EPA utilizing the full data package (summary forms and raw data) submitted by the laboratories.

Chemical data will be reviewed with regard to the following, as appropriate to the particular analysis:

- COC records (including cooler receipt forms)
- Holding times and conditions
- Conformance with required analytical protocol(s)
- Instrument calibration
- Blanks
- Detection/quantification and quantification limits

- Recoveries of surrogates and/or spikes (LCS/LCSD and MS/MSD)
- Variability for duplicate analysis (RPD)
- Corrective Action Records
- Completeness
- Data report formats.

If the data are outside the PARCC parameters, project specified MRLs, project QA/QC limits, or if sample collection, handling, or documentation are lacking, then corrective action(s) will be initiated.

B10.2 DATA ARCHIVING

All laboratories will maintain all data, forms, communications, and electronic data pertaining to this project for a minimum of seven years. After seven years the laboratories will notify the LWG about removal of the data from the laboratory site. The LWG has an established library and records center. All data, communications, and electronic data will be archived and maintained for seven years, after this time the data will be digitally archived and maintained by the LWG.

SECTION C

DATA QUALITY ASSESSMENT

C1 DATA QUALITY ASSESSMENT

C1.1 PARCC PARAMETERS

The data quality assessment will include an overall evaluation of the data based on the validation results and the project DQOs. The usability of the data will be evaluated to determine how results of the data validation will be reconciled with the data uses. PARCC parameters (see Table C1-1) are the specific procedures to be used to assess, on a routine basis, the precision, accuracy, completeness, representativeness, and comparability characteristic of each type of critical measurement for each type of sample matrix. The goal for this project for PARCC parameters is 90 percent. Determination of PARCC parameters is outlined in Tables A7-6 and A7-7.

C1.1.1 Precision

Precision is the measure of the reproducibility between individual measurements of the same property, usually under similar conditions, such as multiple measurements of the same sample. Precision is assessed by performing multiple analysis on a sample and is expressed as RPD when duplicate analysis are performed and as percent relative standard deviation (%RSD) when more than two analysis are performed on the same sample (e.g., triplicates).

Precision measurements can be affected by the nearness of a chemical concentration to the method detection limit (MDL), where the percent error (expressed as either RSD or RPD) increases. The equations used to express precision are as follows:

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

- C₁ larger of the two observed values
C₂ smaller of the two observed values
RPD Relative percent difference

$$\%RSD = (SD / D_{avg}) \times 100$$

$$\text{Where: } SD = \sqrt{\left(\frac{\sum_{i=1}^n (D_i - D_{avg})^2}{(n - 1)} \right)}$$

- RPD Relative percent difference
D_i ith sample value
D_{avg} average sample value
n number of samples

C1.1.2 Accuracy

Accuracy is an expression of the degree to which a measured or computed value represents the true value. Accuracy may be expressed as a percentage of the true or reference value or as a percent recovery in those analyses where spiked samples are analyzed. Accuracy of MS is measured by calculating the percentage of recovery of spiked compounds as follows:

$$\%R = \frac{S - U}{C_{sa}} \times 100$$

%R	percent recovery
S	measured concentration in spiked aliquot
U	measured concentration in un-spiked aliquot
C _{sa}	actual concentration of spike added

C1.1.3 Representativeness

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. In the field, representativeness will be addressed primarily in the sample design, through the selection of sampling sites and procedures. In the laboratory, representativeness will be ensured by the proper handling and storage of samples and analysis within the specified holding times, so that the material analyzed reflects the material collected as accurately as possible.

C1.1.4 Comparability

Comparability expresses the confidence with which one data set can be compared with another. Comparability for this project will not be quantified, but will be addressed through the use of SOPs, field and laboratory methods that are based on the EPA recognized methods and procedures for physical and chemical analysis of environmental samples. The use of standard reporting units also will facilitate comparability with other data sets. Units provided in Tables A7-4 and A7-5 will be used as the reporting units for this program. Comparability of data generated by this project with other data will be discussed, when appropriate, in deliverable reports.

C1.1.5 Completeness

Completeness is a measure of the amount of data that is determined to be valid in proportion to the amount of data collected.

Completeness is expressed as a percentage and is calculated as follows:

$$\%C = \frac{V}{N} \times 100$$

- %C percent completeness
V number of measurements judged valid
N total number of measurements necessary to achieve a specified statistical level of confidence in decision-making

The target for completeness for all components of this project is 90 percent. Data that have been qualified as estimated because the QC criteria were not met will be considered valid for the purpose of assessing completeness. Data that have been qualified as rejected will not be considered valid for the purpose of assessing completeness.

C1.2 DETECTION LIMITS

The MDL, as defined in 40 CFR 136.2(f), is the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. MDLs are determined from analysis of a sample in a given matrix containing the analyte. The MRLs for all analytes, which have been set at or above MDL, are listed in Tables A7-4 and A7-5. When an analyte falls between the MRL and lab stated MDL the result will be flagged "J", which indicates an estimated value.

The MDL is defined as follows:

$$\text{MDL} = t_{(n-1, 1-\alpha = 0.99)} \times s$$

s = standard deviation of the replicate analysis

$t_{(n-1, 1-\alpha = 0.99)}$ = students' *t*-value at a 99 percent confidence level (= 0.01) and *n*-1 degrees of freedom

The achieved MRL should support the stated ACGs. The achieved MRL may vary as a result of sample size. Laboratories should attempt to adjust sample size to meet the ACG.

C1.3 REPORTS TO MANAGEMENT

C1.3.1 Corrective Actions

Corrective actions will be required if there are deviations from the methods or QA requirements established in this QAPP. 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 selected will be appropriate to the severity of the non-conformance and realistic in terms of the resources required for implementation.

The Chemistry QA Manager, the Sampling and Analysis Coordinator, or any project team member who discovers or suspects a non-conformance is responsible for reporting the non-conformance. The Sampling and Analysis Coordinator will ensure that no additional work dependent on the non-conforming activity is performed until a confirmed non-conformance is corrected.

Corrective action reports (Figure C1-1) will be used to document non-conformances and subsequent corrective actions. The Chemistry QA Manager and Sampling and Analysis Coordinator will review these reports and approve the corrective action. The RI/FS Coordinator may also submit the corrective action reports to the LWG or EPA, as appropriate. The Chemistry QA Manager is ultimately responsible for implementation of appropriate corrective action and maintenance of a complete record of QC issues and corrective actions. The Chemistry QA Manager will inform the Sampling and Analysis Coordinator of any significant deviations from the QAPP and any corrective action reports prepared for this project. The Sampling and Analysis Coordinator will be responsible for evaluating all reported non-conformances, conferring with the RI/FS Coordinator, and executing the corrective action as developed and scheduled.

C1.3.1a Field Corrective Action

The initial responsibility for monitoring the quality of field measurements and sample collection lies with the field personnel. Each technical staff member is responsible for verifying that all QC procedures are followed. A description of any corrective action taken will be entered in the field logbook. If conditions do not allow for conformance with the FSP or QAPP, then the Sampling and Analysis Coordinator will be immediately consulted. The Sampling and Analysis Coordinator must authorize any corrective action or field condition resulting in a revision to the FSP or QAPP. If corrective action requires a departure from FSP or QAPP, these changes will be documented on a Field Change Request Form (Figure B5-1). In circumstances where conditions are unexpected, the appropriate sampling actions consistent with project objectives will be conducted after the Field Supervisor confers with the Sampling and Analysis Coordinator. This change will be noted in the field log and a change request form completed for the project files.

C1.3.1b Laboratory Corrective Action

There will be continuous data assessment and comparison of data precision, accuracy, and completeness to the data acceptance criteria and project DQOs. The Laboratory QA Coordinator or Project Manager will be responsible for

keeping the Chemistry QA Manager apprised of the laboratory's QC status during all analytical events. Assessing the problem and implementing corrective action will follow any significant or consistent deviation from acceptance criteria and analytical goals.

The need for corrective action in the analytical laboratory may come from several sources: equipment malfunction, failure of internal QA/QC checks, method blank contamination, noncompliance with QA requirements, or failure of performance or system audits. In accordance with the laboratory's SOP, laboratory QA/QC failures will immediately be brought to the attention of the appropriate persons in the laboratory. If analytical conditions are such that non-conformance with this QAPP is indicated, the Chemistry QA Manager will be notified within 24 hours so that any additional corrective actions can be taken.

Specific corrective actions are outlined in the laboratory SOPs and include but are not limited to the following:

- Identify the source of the nonconformance
- Reanalyze sample(s) if holding time criteria permit
- Retrieve archived sample(s) for analysis (each sample collected has an associated archived sample for use as sample backup, primarily for extractable organics and/or metals analysis)
- Reanalyze sample(s) following re-sampling
- Evaluate and/or amend sampling and analytical procedures
- Accept the data and apply qualifier(s) to indicate level of uncertainty
- Reject data as unusable.

As soon as sufficient time has elapsed for the corrective action to be implemented, evidence of correction of deficiencies will be presented.

C2 DATA REPORTING

Effective communication between all personnel is an integral part of a quality system. Planned reports provide a structure for apprising management of the project schedule, the deviations from approved QA and test plans, the impact of these deviations on data quality, and the potential uncertainties in decisions based on the data.

C2.1 LABORATORY REPORTS

QA reports will include analysis reports from the laboratory and corrective action reports from the Chemistry QA Manager. All reports required under this QAPP will be submitted to the Chemistry QA Manager, who reports to the Sampling and Analysis Coordinator. All labs will be required to submit the following information in the final data package/report:

C2.1.1 Sample Data

Sample data reports including sample analysis time, number of samples, cross reference of laboratory ID and sample ID, sample location information, deviation from SOPs, time of day, and date.

C2.1.2 Sample Management Records

C2.1.2a Test Methods

Test methods are specified in Table B4-1. Associated QC and frequency are listed in Tables A7-1, A7-2, A7-3, and Table B5-1. Project specific ACGs are listed in Tables A7-4 and A7-5 and QA/QC limits are listed in Tables A7-6 and A7-7. Laboratories will only prepare LWG samples with samples from the same project. Since samples will be stored frozen (considered to be in a state of stasis) the laboratory will be able to pull batches of 20 for extractions.

C2.1.3 QA/QC Reports

The QA report needs to summarize the QC results and present the information that the data user needs to assess the impact of the bias or imprecision of the data on the usability of that data. This includes percent recoveries for surrogates and spikes, RPD for duplicated analysis, tunes, initial calibration, continuing calibration, instrument blanks, daily low level MRL sample, second source calibration verification samples, project blanks (field, reagent, rinsate, and method), replicates, duplicates, and spikes (surrogate and matrix).

C2.2 MONTHLY REPORTS

At the end of every month during which project samples are received by the laboratory, the laboratory will prepare and deliver to the Chemistry QA Manager a QA status report that includes:

- Inventory and status of samples held at the laboratory
- Summaries of out-of-control laboratory QC data and any corrective actions implemented
- 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.

C2.3 QUALITY ASSURANCE REPORTS

Reports of significant QA deficiencies will be provided immediately to the Chemistry QA Manager. Verbal notice will be followed with written documentation in a memorandum and a corrective action report. The Chemistry QA Manager will be responsible for reporting QA problems to the Sampling and Analysis Coordinator.

All reported data will include results of the QA data validation review and conclusions regarding data accuracy, precision, completeness, and any corrective actions and sampling procedure alteration documentation. Data validation results will be provided to EPA in a technical memorandum.

SECTION D

DATA VALIDATION AND USABILITY

D1 DATA REVIEW, VERIFICATION, AND VALIDATION

The first 5% of the data for each suite of parameters will be submitted to EPA for validation by EPA's QA Office. After sample analysis the following laboratory data deliverables will be sent to EPA for validation:

- QC Summary Forms which include surrogate recovery forms, matrix spike and matrix spike duplicate forms, method blanks summary forms, instrument performance check forms, initial calibration forms, continuing calibration check forms, and internal standard area and retention times forms;
- Target compound results and sample data which includes reconstructed ion chromatograms, quantification list, EICP for manual integrations and enhanced and un-enhanced mass spectra of detected compounds
- Standards data
- QC data for the instrument performance checks, method blanks, laboratory control samples (if required), matrix spike and duplicate analytical runs
- COC, shipment and other sample control documentation
- Statement of Work or SOPs.

The next 10-20% of the data will be fully validated by LWG's third party data reviewer. This percentage will be determined based on the results of the EPA data validation. This data validation will involve the same process as EPA's.

Depending on the validation results of the first 5% of the samples, the rest of the samples may undergo a third party partial data validation. This process would involve the assessment and evaluation of analytical QC and sample results summary forms. Five percent of the partially validated data will be peer reviewed by EPA utilizing the full data package (summary forms and raw data) submitted by the laboratories.

D2 VERIFICATION AND VALIDATION METHODS

Chemistry data quality will be evaluated by comparisons to QC criteria. When data fall outside of the QC criteria they will be flagged. The PARCC parameters for this project include the goal of at least 90% data completeness. Data that are rejected will not be used for RI/FS decision-making. The sampling design is considered robust enough that if the 90% data completeness goal is achieved then the LWG should have sufficient data to move forward with evaluations of ecological and human health risks as well as nature and extent evaluations.

Through discussions that occurred between EPA and the LWG prior to completion of the Work Plan, ACGs were established. These goals are exceptionally low, and in a variety of instances are below levels that the laboratories are capable of attaining when the sample matrices are anything but clean. Therefore the LWG, working with the analytical laboratories and EPA, have established project specific MRLs. When ACGs are not attained, but MRLs are met, the data will continue to be used for RI/FS decision-making provided that the data meet QC requirements. These data will be discussed relative to uncertainty in the RA, and ultimately risk management decisions will be made that will consider the level of confidence associated with all analytical results.

D3 RECONCILIATION WITH USER REQUIREMENTS

Validated data will be analyzed to support multiple DQOs. Sediment and tissue chemical distributions will be assessed to describe the relationships between these parameters as well as human and ecological risks. Other sediment data will be used to assess risk to humans from exposures at beaches. If anomalous trends are evident, the laboratory data will be immediately reviewed to reconfirm that the data have been appropriately reported. Anomalous distributions could suggest the presence of unknown sources or transport mechanisms that would require additional study in Round 2.

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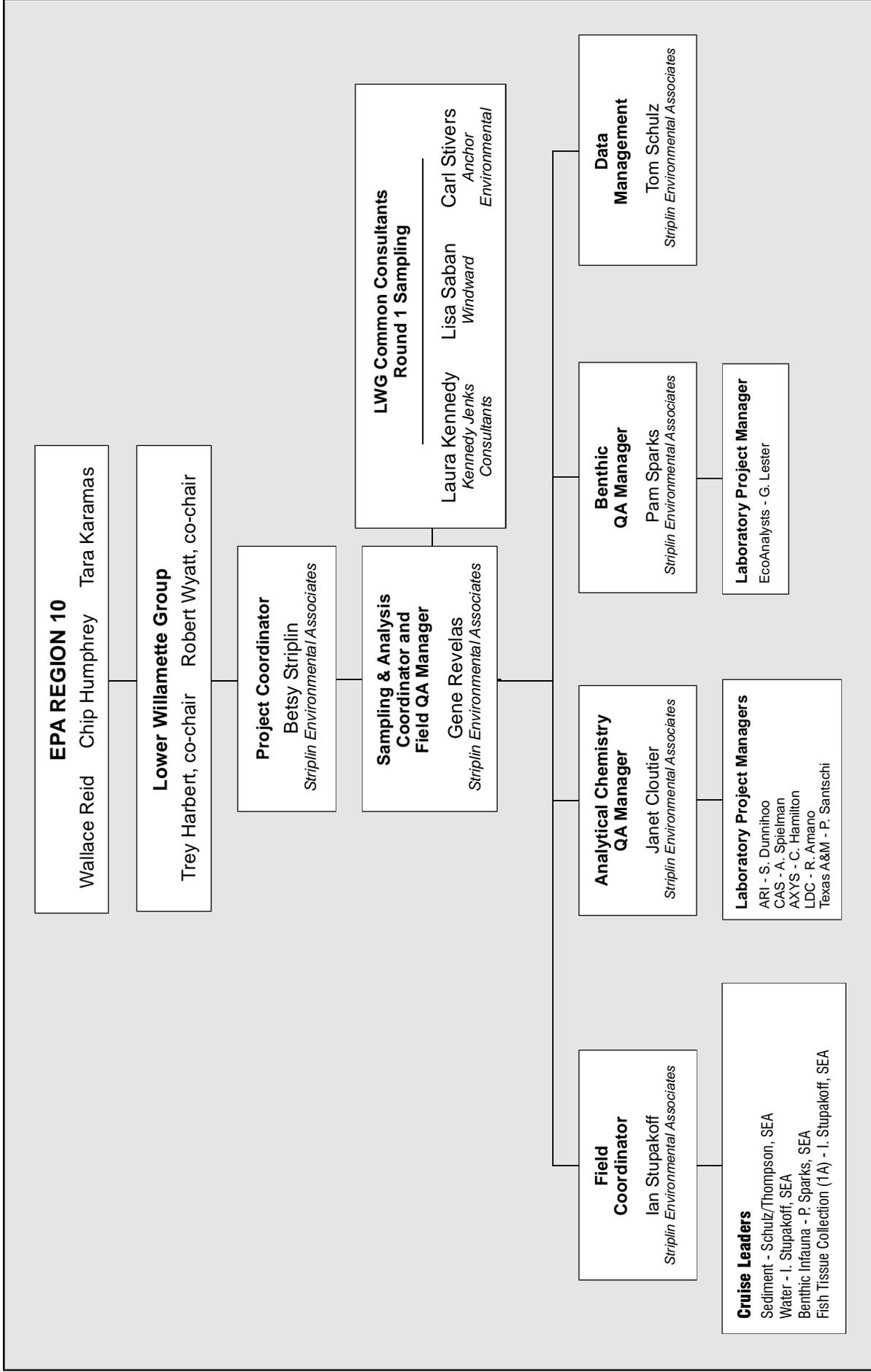


Figure A4-1. Round 1 Project Organization.



An Employee-Owned Company

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CHAIN OF CUSTODY
Sediment and Tissue Chemistry

PAGE _____ OF _____ SR# _____
COC # _____

PROJECT NAME		PROJECT NUMBER		PROJECT MANAGER		COMPANY/ADDRESS		PHONE #		FAX #		SAMPLER'S SIGNATURE		SAMPLE I.D.		DATE	TIME	LAB I.D.	MATRIX	NUMBER OF CONTAINERS Metals (list below) <input type="checkbox"/> Total Volatile Solids <input type="checkbox"/> Total Solids <input type="checkbox"/> Lipids TOC (ASTM D4129M) Grain size - PSEP / ASTM D422 Sulfide <input type="checkbox"/> Total (9030M) <input type="checkbox"/> Water Soluble AVS / SEM Ammonia <input type="checkbox"/> Total (350.1m) <input type="checkbox"/> Pore water Pesticides (8081) PCBs (8082) <input type="checkbox"/> Aroclors <input type="checkbox"/> Congeners Semivolatiles (GC/MS SIM) <input type="checkbox"/> PAHs <input type="checkbox"/> Phthalates <input type="checkbox"/> Phenols <input type="checkbox"/> PSEP Organotin - Sediment <input type="checkbox"/> Mono <input type="checkbox"/> Di <input type="checkbox"/> Tri <input type="checkbox"/> Tetra Organotin - Pore water <input type="checkbox"/> Mono <input type="checkbox"/> Di <input type="checkbox"/> Tri <input type="checkbox"/> Tetra Volatiles (8260) TRPH 8015 / 418.1 REMARKS	
Signature _____		Date/Time _____		Signature _____		Date/Time _____		Signature _____		Date/Time _____		Signature _____		Date/Time _____		Signature _____		Date/Time _____			
Printed Name _____		Firm _____		Printed Name _____		Firm _____		Printed Name _____		Firm _____		Printed Name _____		Firm _____		Printed Name _____		Firm _____			
REPORT REQUIREMENTS I. Routine Report: Method Blank, Surrogate, as required II. Report Dup., MS, MSD as required III. Data Validation Report (includes all raw data) IV. CLP Deliverable Report V. EDD				INVOICE INFORMATION P.O. # _____ Bill To: _____ TURNAROUND REQUIREMENTS 24 hr. _____ 48 hr. _____ 5 Day _____ Standard (10-15 working days) Provide FAX Results Requested Report Date _____				Circle which metals are to be analyzed: SMS Metals: As Cd Cr Cu Pb Hg Ag Zn CA Metals: Ag As Cd Cr Cu Hg Ni Pb Se Zn				SPECIAL INSTRUCTIONS/COMMENTS:									
RELINQUISHED BY: Signature _____ Date/Time _____ Printed Name _____ Firm _____				RECEIVED BY: Signature _____ Date/Time _____ Printed Name _____ Firm _____				RELINQUISHED BY: Signature _____ Date/Time _____ Printed Name _____ Firm _____				RECEIVED BY: Signature _____ Date/Time _____ Printed Name _____ Firm _____									

Figure B3-1. CAS Chain-of-Custody Form.

Figure B3-3. Axys Chain-of-Custody Form.

 AXYS Axys Analytical Services Ltd POST OFFICE BOX 2219, 2045 MILLS ROAD WEST TEL (250) 655-5800 SIDNEY, BRITISH COLUMBIA, CANADA V8L 3S8 FAX (250) 655-5811			ANALYSIS REQUESTED							Sample Received (Y or N)
			Preservative Added (Y or N)							
Date Submitted: _____ Date Required: _____										
Sample ID	Date/Time Sample	Sample Type							Lab Sample No.	
Client		No. Samples Submitted:		Relinquished by:		Date:		Received by:		Date:
Contact:		No Coolers/Boxes		Relinquished by:		Time:		Received by:		Time:
Address: _____		Instruction to lab: (include quote #, if applicable)				Date:				Received by:
_____				Date:		Received by:		Time:		
_____				_____		Time:		_____		_____
Postal Code:		_____		Sample Condition Upon Receipt:						
FAX No:		_____		Frozen: _____ Cold: _____ Ambient: _____						
Tel. No:		_____		Other (Breakage, Leakage, etc.) _____						
P.O. No:		Job No:		_____						

Figure B3-5. CAS Cooler Receipt Form.

**Columbia Analytical Services Inc.
Cooler Receipt And Preservation Form**

Project/Client _____ Work Order K22 _____

Cooler received on _____ and opened on _____ by _____

1.	Were custody seals on outside of cooler? If yes, how many and where? _____	Y	N
2.	Were seals intact and signature & date correct?	Y	N
3.	COC # _____		
	Temperature of cooler(s) upon receipt: _____		
	Temperature Blank: _____		
4.	Were custody papers properly filled out (ink, signed, etc.)?	Y	N
5.	Type of packing material present _____		
6.	Did all bottles arrive in good condition (unbroken)?	Y	N
7.	Were all bottle labels complete (<i>i.e.</i> analysis, preservation, etc.)?	Y	N
8.	Did all bottle labels and tags agree with custody papers?	Y	N
9.	Were the correct types of bottles used for the tests indicated?	Y	N
10.	Were all of the preserved bottles received at the lab with the appropriate pH?	Y	N
11.	Were VOA vials checked for absence of air bubbles, and if present, noted below?	Y	N
12.	Did the bottles originate from CAS/K or a branch laboratory?	Y	N
13.	Are CWA Microbiology samples received with > 1/2 the 24 hr. hold time remaining from collection?	Y	N
14.	Was CL2/Residual negative?	Y	N

Explain any discrepancies: _____

RESOLUTION: _____

Samples that required preservation or received out of temperature:

Sample ID	Reagent	Volume	Lot Number	Bottle Type	Rec'd out of Temperature	Initials

Login person has verified bottle id's to COC and labels: _____

Figure B3-6. ARI Cooler Receipt Form.

Cooler Receipt Form



ARI Client: _____ Project Name: _____
 COC NO.: _____ Delivered By: _____
 Tracking NO.: _____ Date: _____
 ARI Job No.: _____ Lims NO.: _____

Preliminary Examination Phase:

1. Were intact, properly signed and dated custody seals attached
 To the outside of the cooler? YES NO
2. Were custody papers included with the cooler YES NO
3. Were custody papers properly filled out (ink, signed etc.)? YES NO
4. Complete custody forms and attach all shipping documents OK NA

Cooler Accepted BY: _____ Date: _____ Time: _____

Log-IN Phase:

5. Was a temperature blank include in the cooler? YES NO
6. Record Cooler Temperature °C
7. What kind of packing material was used?
8. Was sufficient ice used (if appropriate)? YES NO
9. Were all bottles sealed in separate plastic bags? YES NO
10. Did all bottles arrive in good condition (unbroken)? YES NO
11. Were all bottle labels complete and legible? YES NO
12. Did all bottle labels and tags agree with custody papers? YES NO
13. Were all bottles used correct for the requested analyses? YES NO
14. Do any of the analyses (bottles) require preservative?
 (if so, Preservation checklist must be attached) YES NO
15. Were all VOA vials free of air bubbles? YES NO
16. Was sufficient amount of sample sent in each bottle? YES NO
17. Notify Project Manager of any discrepancies or concerns..... OK NA

Cooler Opened By: _____ Date: _____ Time: _____

Explain any discrepancies or negative responses:

Figure B3-7. Axys Cooler Receipt Form (1 of 2).

SAMPLE RECEIVING RECORD

Waybill : Present _____ Absent _____		Waybill #:	
Date Shipped:	Date Received:	Time Received:	
Received By (print):		Signature:	
AXYS Client and Contract #:		Client Reference #:	
Condition of Shipping Container:			
Temperature of Shipping Container on Receipt:			
Custody Seals: Absent : _____ Present : ___ Intact ___ Broken _____ On: Shipping Container: ___ Sample Container: _____		Custody Seal Numbers:	
AXYS Sample IDs:			
Log-in by (print):		Signature:	
Chain of Custody or Documents: Present: _____ Absent: _____ Traffic Report/Packing List: Y / N			
N	Sample IDs	Y /	Sample Tag Numbers Y / N
N	Location	Y /	Sample Type Y / N
N	Date & Time of Collection	Y /	Preservative Added Y / N _____ (details)
N	Collector's Name	Y /	Preservation Requested Y / N _____ (details)
Sample Acceptance Criteria:			
N	Appropriate Container	Y / N	Matrix Type : _____ Correct Labelling Y /
N	Damaged Container	Y / N	Holding Time Exceeded Y /
N	Adequate Sample Size	Y / N	Appropriate Temperature Y /
Aqueous Samples: pH adjustment required Y / N			
Residual Cl required Y /		N	
Sample Tags: Present: _____ Absent: _____		Sample Labels: Present: _____ Absent: _____	
Sample Labels Cross Referenced to Chain of Custody	Y / N	Information Agrees	Y / N
Sample Tags Cross Referenced to Sample Labels	Y / N	Information Agrees	Y / N
Sample Tags Cross-Referenced to Chain of Custody	Y / N	Information Agrees	Y / N
Problems or Discrepancies:			
Action Taken:			

Figure B5-1. Field Change Request Form.

	FIELD CHANGE REQUEST	Project Number:
Project Number: Project Name:		Field Change No. Page _____ to
<p>CHANGE REQUEST Applicable Reference: Description of Change:</p> <p>Reason for Change:</p> <p>Impact on Present and Completed Work:</p> <div style="display: flex; justify-content: space-between; margin-top: 20px;"> <div style="width: 60%;"> <p style="text-align: center;">(Field Scientist)</p> <p style="text-align: center;">(Field Task Leader)</p> </div> <div style="width: 35%; text-align: right;"> <p>Requested by: Date: ____/____/</p> <p>Acknowledged by: Date: ____/____/</p> </div> </div>		
<p style="text-align: center;">FIELD OPERATIONS MANAGER RECOMMENDATION</p> <p>Recommended Disposition:</p> <div style="display: flex; justify-content: flex-end; margin-top: 20px;"> <p>Recommendation by: Date: ____/____/</p> </div> <p style="text-align: center;">(Sampling and Analysis Coordinator)</p>		
<p style="text-align: center;">PROJECT MANAGER APPROVAL</p> <p>Final Deposition:</p> <div style="display: flex; justify-content: flex-end; margin-top: 20px;"> <p>Approved/Disapproved by: Date: ____/____/</p> </div> <p style="text-align: center;">(CERCLA Coordinator)</p>		

Figure B10-1. SEA EQUiS Format.

Field Name	Field Type	Comments
sys_sample_code	Text [40]	filled in by lab
sample_name	Text [30]	filled in by lab
sample_matrix_code	Text [10]	filled in by lab
sample_type_code	Text [20]	filled in by lab
sample_source	Text [10]	filled in by lab
parent_sample_code	Text [40]	filled in by lab
sample_delivery_group	Text [10]	filled in by lab
sample_date	Date	filled in by lab
sample_time	Text [5]	filled in by lab
chain_of_custody	Text [15]	filled in by lab
sample_receipt_date	Date	filled in by lab
sample_receipt_time	Text [5]	filled in by lab
sys_loc_code	Text [20]	filled in by SEA
equipment_code	Text [60]	filled in by SEA
start_depth	Double	filled in by SEA
end_depth	Double	filled in by SEA
depth_unit	Text [15]	filled in by SEA
sent_to_lab_date	Date	filled in by SEA
sampler	Text [30]	filled in by SEA
data_provider	Text [20]	filled in by SEA
sampling_reason	Text [30]	filled in by SEA
task_code	Text [10]	filled in by SEA
collection_quarter	Text [5]	filled in by SEA
composite_yn	Text [1]	filled in by SEA
composite_desc	Text [255]	filled in by SEA
sample_class	Text [10]	filled in by SEA
custom_field_1	Text [255]	filled in by SEA
custom_field_2	Text [255]	filled in by SEA
custom_field_3	Text [255]	filled in by SEA

Figure C1-1. Corrective Action Record.

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:

Table A4-1. Project Team Contact Information.

Organization	Person	Project Role	Phone	Fax	Email
EPA	Wallace Reid	Site Manager	206-553-1728	206-553-0124	reid.wallace@epa.gov
	Chip Humphrey	Project Manager	503-326-2678	503-326-3399	humphrey.chip@epa.gov
	Tara Karamas	Project Manager	206-553-0039	206-553-0124	karamas.tara-ann@epa.gov
LWG	Trey Harbert, Port of Portland	Co-Chair	503-944-7326	503-944-7353	harbert@portptld.com
	Bob Wyatt, Northwest Natural	Co-Chair	503-226-4211 ext. 5425	503-273-4815	rjw@nwnnatural.com
LWG Common Consultants	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
	Betsy Striplin (SEA)	CERCLA Coordinator	206-241-5185	206-241-5159	bstriplin@striplin.com
	Gene Revelas (SEA)	Sampling and Analysis Coordinator	360-705-3534	360-705-3669	grevelas@striplin.com
	Janet Cloutier (SEA)	Analytical Chemistry QA Manager	360-705-3534	360-705-3669	jcloutier@striplin.com
	Ian Stupakoff (SEA)	Field Coordinator	360-705-3534	360-705-3669	stupakoff@striplin.com
	Pam Sparks (SEA)	Benthic QA Manager	360-705-3534	360-705-3669	psparks@striplin.com
Laboratories	Tom Schulz (SEA)	Data Management	360-705-3534	360-705-3669	tschulz@striplin.com
	Sue Dunnihoo (ARI)	Laboratory Project Manager	206-695-6207	206-695-6201	sue@arilabs.com
	Abbie Spielman (CAS)	Laboratory Project Manager	360-577-7222	360-636-1068	aspelman@kelso.caslab.com
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Table A7-1. Organic QA/QC Sample Analyses Procedures.*

	Blind Field Duplicates Sediment	Blind Field Replicates Sediment	Field Triplicates Tissue	Instrument Blanks	TUNE ⁷	ICAL ⁴	CCV ⁵	Method Blanks	LCS/ LCS/D	OPR	MS/MSD	Surrogates	Check STD	Internal STD
Volatile Organics	X	X			X	X ¹³	X ¹⁴	X ¹	X ^{1,6}		X ^{1,6}	X ³	X ¹⁰	X ¹⁵
Semivolatiles	X	X	X ¹²		X	X ¹³	X ¹⁴	X ¹	X ^{1,2,6}		X ^{1,6}	X ³	X ¹⁰	X ¹⁵
Pesticides/PCBs	X	X	X ¹²	X ⁸		X ¹³	X ¹⁴	X ¹	X ^{1,2,6}		X ^{1,6}	X ³	X ¹⁰	
Herbicides	X	X	X ¹²	X ⁸		X ¹³	X ¹⁴	X ¹	X ^{1,6}		X ^{1,6}	X ³	X ¹⁰	
PCDDs/PCDFs/HRPCBs	X	X	X ¹²	X ⁸	X	X	X	X ¹		X ¹¹		X ³	X ¹⁰	X ¹⁵
TBT	X ⁹	X ⁹	X ¹²		X	X	X	X ¹	X ^{1,6}		X ^{1,6}	X ³	X ¹⁰	X ¹⁵

¹ = Frequency of Analysis (FOA) = 5% or one per extraction batch, whichever is more frequent. VOCs every 12 hours. For method blank contamination: Corrective action: reduce contamination or reextract/reanalyses.

² = Certified Reference Material. Control Limits: organic: within 95% confidence interval of true value metals: 80-120% recovery. Corrective action: PM discretion: discuss results with laboratory; qualify sample results.

³ = Surrogate spikes required for every sample, including matrix spiked samples, blanks, PE and reference materials. %recovery limits are listed in Tables A7-7, A7-8 and A7-9, if surrogates are outside of stated limits reanalyses or reextraction may be required, chemistry QA manager must be notified immediately.

⁴ = Initial calibration required before any samples are analyzed, after each major disruption of equipment, and when ongoing calibration fails to meet criteria.

⁵ = Ongoing calibration required at the beginning of each work shift, every 10 samples or every 12 hours.

⁶ = %recovery limits are listed in Tables A7-7, A7-8, and A7-9 if outside of stated limits reanalyses or reextraction may be required, chemistry QA manager must be notified immediately.

⁷ = This is done every 12 hours to check instrument ability to generate valid data.

⁸ = Analyzed at a frequency specified in the method or SOP or after a sample with high concentrations of target analyte to avoid carry-over.

⁹ = Bulk sediment analysis

¹⁰ = This standard is made at the QAPP listed MRLs for those analyses that do not include a standard at the MRL in the initial calibration curve. It is run daily to assure that the instrument can achieve the analytical concentration goals listed in the QAPP. In the case of PCDDs/PCDFs this will be at the same level as CS 0.2.

¹¹ = Ongoing Precision and Recovery standard (OPR): a laboratory blank spike with known quantities of analytes. The OPR is analyzed exactly like a sample. Its purpose is to assure that the results produced by the laboratory remain within the limits specified in this method for precision and recovery.

¹² = Refer to section 3.1.5 Field Triplicates - Tissue Only for explanation.

¹³ = Frequency specified in analytical protocol. Control Limits: <30%RSD for SVOCs, Pesticides, Herbicides, PCB-Ar and VOCs; relative response factors >0.05 for SVOCs and VOCs, >20% breakdown of endrin and DDT (30% combined) for Pesticide. If these limits are not met then the laboratory must recalibrate and reanalyze affected samples.

¹⁴ = Frequency specified in analytical protocol: after every 10-12 samples or every 12 hours whichever is more frequent, and after the last sample of each work shift. Control Limits: <25%D for SVOCs, and VOCs; <15%D (average) for Pesticides, Herbicides, and PCB-Ar, relative response factor >0.05 for SVOCs and VOCs. If these limits are not met then the laboratory must recalibrate and reanalyze affected samples.

¹⁵ = Internal standards are added to every sample as specified in analytical protocol. Area response and retention time as indicated in each method. Corrective action: Laboratory to correct problem and reanalyze affected samples.

* = These frequency, control limits, and corrective actions follow method specific guidance.

Table A7-2. Metals QA/QC Sample Analyses Procedures.*

	Blind Field Duplicates Sediment	Blind Field Replicates Sediment	Field Triplicates Tissue	Instrument Blanks	ICAL	CCV	IB	Method Blanks	Duplicates	LCS/LCSD	MS	SRM	Check STD
Metals	X	X	X ⁴	X ⁷	X ⁵	X ⁶	X ¹⁰	X ¹	X ¹	X ^{1,2,3}	X ^{1,3}	X ⁹	X ⁸
Mercury	X	X	X ⁴	X ⁷	X ⁵	X ⁶	X ¹⁰	X ^{1,3}	X ¹	X ^{1,3}	X	X ⁹	

¹ = Frequency of Analysis (FOA) = 5% or one per extraction batch, whichever is more frequent. For method blank contamination: Corrective action: redigest and reanalyze with analyte conc > 10 times the highest method blank.

² = Certified Reference Material. Control Limits: organic: within 95% confidence interval of true value; metals: 80-120% recovery. Corrective action: PM discretion: discuss results with laboratory; qualify sample results.

³ = %recovery limits are listed in Tables A7-7, A7-8 and A7-9, if analytes are outside of stated limits reanalyses or reextraction may be required, chemistry QA manager must be notified immediately.

⁴ = Refer to section 3.1.5 Field Triplicates - Tissue Only for explanation.

⁵ = Frequency: Daily; Control Limits: Correlation coefficient ³ 0.995; Corrective Action: Lab to recalibrate the instrument and reanalyze any affected samples. The initial calibration verification must come immediately after initial calibration and must be 90-110% (80-120% for Hg) Lab to recalibrate the instrument and reanalyze any affected samples. For GFAA the +/- 10%.

⁶ = Frequency: After every 10 samples or as specified in each method, and after the last sample. Control Limits: must be 90-110% (80-120% for Hg) Lab to recalibrate the instrument and reanalyze any affected samples. For GFAA the +/- 10%

⁷ = Frequency: After initial calibration, then 10% of samples or as specified in each method, and after the last sample. Control Limits: analyte conc £ ACG; Corrective Action: Lab to recalibrate the instrument and reanalyze any affected samples.

⁸ = ICP Interelement Interference Check Sample Frequency: beginning and end of every analytical sequence or 2/8 hour shift. Control Limits: 80-120% of true value; Corrective Action: Lab to recalibrate the instrument and reanalyze any affected samples.

⁹ = Sediment only.

¹⁰ = Instrument Blanks are analyzed after the initial calibration verification (ICV) standard and after each continuing calibration verification standard (CCV).

* = These frequency, control limits, and corrective actions follow method specific guidance.

Table A7-3. Conventional QA/QC Sample Analyses Procedures.*

	Blind Field Duplicates Sediment	Blind Field Replicates Sediment	Field Triplicates Tissue	ICAL	Method Blanks	Duplicates	LCS	MS
Total Organic Carbon	X	X		X ⁶	X ^{1,2}	X ¹	X ^{1,2}	X ^{1,2}
Total Solids	X	X				X ¹		
Particle Size	X	X				X ¹		
Atterberg Limits	X	X						
Specific Gravity	X	X						
Lipids ³			X ⁴	X ⁵		X ¹		

¹ = Frequency of Analysis (FOA) = 5% or one per extraction batch, whichever is more frequent.

² = %recovery limits are listed in Tables A7-7, A7-8 and A7-9, analytes outside of stated limits reanalyses or reextraction may be required, chemistry QA manager must be notified immediately.

³ = See QAPP for guidance

⁴ = Refer to section 3.1.5 Field Triplicates - Tissue Only for explanation.

⁵ = Daily calibration of scale with certified weights.

⁶ = Correlation coefficient ³ 0.995

* = These frequency, control limits, and corrective actions follow method specific guidance.

Table A7-4. Sediment - Project Specific Method Reporting Limits, Analytical Concentration Goals, and Methodology (including SOPs).

Analytes	Extraction / Digestion SOPs	Clean Ups SOPs	Analytical Method	MRL ¹	ACG ²	CAS#	
Conventionals							
Total Solids			EPA-160.3/SM 2540B	0.01 %	NE	NA	
Grain Size			ASTM D-422-63	1%	NE	NA	
Total Organic Carbon			Plumb et al., 1981	0.005 %	NE	NA	
Metals							
				mg/Kg dw (ppm)	mg/Kg dw (ppm)		
Silver - Ag	509S		SW846-7761 GFAA	0.02	*	7782-49-2	
Aluminum - Al	507S		SW846-6010 ICP	5.0	*	7429-90-5	
Arsenic - As	509S		SW846-7060A GFAA	0.10	*	7440-66-6	
Cadmium - Cd	509S		SW846-7131A GFAA	0.02	*	7440-43-9	
Chromium - Cr	507S		SW846-6010 ICP	0.50	*	7440-47-3	
Copper - Cu	507S		SW846-6010 ICP	0.20	*	7440-50-8	
Nickel - Ni	507S		SW846-6010 ICP	1.0	*	7440-50-8	
Lead - Pb	509S		SW846-7421 GFAA	0.10	*	7439-92-1	
Antimony - Sb	507S		SW846-7041 GFAA	0.20	*	7440-36-0	
Selenium - Se	509S		SW846-7040 GFAA	0.20	*	7782-49-2	
Zinc - Zn	507S		SW846-6010 ICP	0.60	*	7440-66-6	
Mercury - Hg	511S		SW846-7471A CVAA	0.05	*	7439-97-6	
Butyltins							
				µg/Kg dw (ppb)	µg/Kg dw (ppb)		
Monobutyltin	315S		Krone et. al	12	*	78763-54-9	
Dibutyltin	315S		Krone et. al	12	*	1002-53-5	
Tributyltin	315S		Krone et. al	6.0	0.08	56573-85-4	
Tetrabutyltin	315S		Krone et. al	NE ^b	*	1461-25-2	
PCBs Aroclors							
				µg/Kg dw (ppb)	µg/Kg dw (ppb)		
Aroclor 1016	359S	345S (Florisil)	335S (Acid)	SW846-8082	5.0	*	12674-11-2
Aroclor 1221	359S	345S (Florisil)	335S (Acid)	SW846-8082	10	*	11104-28-2
Aroclor 1232	359S	345S (Florisil)	335S (Acid)	SW846-8082	5.0	*	11141-16-5
Aroclor 1242	359S	345S (Florisil)	335S (Acid)	SW846-8082	5.0	0.004	53469-21-9
Aroclor 1248	359S	345S (Florisil)	335S (Acid)	SW846-8082	5.0	0.004	12672-29-6
Aroclor 1254	359S	345S (Florisil)	335S (Acid)	SW846-8082	5.0	0.004	11097-69-1
Aroclor 1260	359S	345S (Florisil)	335S (Acid)	SW846-8082	5.0	0.004	11096-82-5
CHLORINATED HERBICIDES							
				µg/Kg dw (ppb)	µg/Kg dw (ppb)		
Dalapon	325S	325S (BackExt)	325S (Water wash)	SW846-8151A	45	*	75-99-0
Dicamba	325S	325S (BackExt)	325S (Water wash)	SW846-8151A	20	*	1918-00-9
MCPA	325S	325S (BackExt)	325S (Water wash)	SW846-8151A	10000	*	94-74-6
Dichlorprop	325S	325S (BackExt)	325S (Water wash)	SW846-8151A	10	*	120-36-5
2,4-D	325S	325S (BackExt)	325S (Water wash)	SW846-8151A	6.6	2.8	94-75-7
2,4,5-TP (Silvex)	325S	325S (BackExt)	325S (Water wash)	SW846-8151A	1.7	2.2	93-72-1
2,4,5-T	325S	325S (BackExt)	325S (Water wash)	SW846-8151A	1.7	2.8	93-76-5
2,4-DB	325S	325S (BackExt)	325S (Water wash)	SW846-8151A	45	2.2	94-82-6
Dinoseb	325S	325S (BackExt)	325S (Water wash)	SW846-8151A	20	*	88-85-7
MCPP	325S	325S (BackExt)	325S (Water wash)	SW846-8151A	10000	*	93-65-2
Pentachlorophenol	325S	325S (BackExt)	325S (Water wash)	SW846-8151A	1.7	0.58	87-86-5
ORGANOCHLORINE PESTICIDES ^a							
				µg/Kg dw (ppb)	µg/Kg dw (ppb)		

Table A7-4. Sediment - Project Specific Method Reporting Limits, Analytical Concentration Goals, and Methodology (including SOPs).

Analytes	Extraction / Digestion SOPs	Clean Ups SOPs	Analytical Method	MRL ¹	ACG ²	CAS#
2,4'-DDD	350S	345S (Florisil)	SW846-8081A	0.4	*	53-19-0
2,4'-DDE	350S	345S (Florisil)	SW846-8081A	0.4	*	3424-82-6
2,4'-DDT	350S	345S (Florisil)	SW846-8081A	0.4	*	789-02-6
4,4'-DDD	350S	345S (Florisil)	SW846-8081A	0.4	0.083	72-54-8
4,4'-DDE	350S	345S (Florisil)	SW846-8081A	0.4	0.0588	72-55-9
4,4'-DDT	350S	345S (Florisil)	SW846-8081A	0.4	0.0588	50-29-3
Total DDT					*	
Aldrin	350S	345S (Florisil)	SW846-8081A	0.2	0.00038	309-00-2
a - BHC	350S	345S (Florisil)	SW846-8081A	0.2	0.001	319-84-6
b - BHC	350S	345S (Florisil)	SW846-8081A	0.2	0.0036	319-85-7
d - BHC	350S	345S (Florisil)	SW846-8081A	0.2	*	319-86-8
g - BHC (Lindane)	350S	345S (Florisil)	SW846-8081A	0.2	0.005	58-89-9
a - Chlordane	350S	345S (Florisil)	SW846-8081A	0.2	*	5103-71-9
g - Chlordane	350S	345S (Florisil)	SW846-8081A	0.2	*	5103-74-2
cis - nonachlor	350S	345S (Florisil)	SW846-8081A	0.4	*	5103-73-1
oxy - chlordane	350S	345S (Florisil)	SW846-8081A	0.4	*	26880-48-8
trans - nonachlor	350S	345S (Florisil)	SW846-8081A	0.4	*	39765-80-5
total Chlordane ^g				1.0	0.057	
Dieldrin	350S	345S (Florisil)	SW846-8081A	0.4	0.0004	60-57-1
Endosulfan I	350S	345S (Florisil)	SW846-8081A	0.2	1.7	959-98-8
Endosulfan II	350S	345S (Florisil)	SW846-8081A	0.4	*	33213-65-9
Endosulfan sulfate	350S	345S (Florisil)	SW846-8081A	0.4	*	1031-07-8
Endrin	350S	345S (Florisil)	SW846-8081A	0.4	0.084	72-20-8
Endrin aldehyde	350S	345S (Florisil)	SW846-8081A	0.4	*	7421-93-4
Endrin ketone	350S	345S (Florisil)	SW846-8081A	0.4	*	53494-70-5
Heptachlor	350S	345S (Florisil)	SW846-8081A	0.2	0.0014	76-44-8
Heptachlor epoxide	350S	345S (Florisil)	SW846-8081A	0.2	0.0007	1024-57-3
Hexachlorobenzene	350S	345S (Florisil)	SW846-8081A	0.2	0.33	118-74-1
Hexachlorobutadiene	350S	345S (Florisil)	SW846-8081A	0.2	0.6	87-68-3
Methoxychlor	350S	345S (Florisil)	SW846-8081A	2.0	1.4	72-43-5
Mirex	350S	345S (Florisil)	SW846-8081A	0.4	0.056	2385-85-5
Toxaphene	350S	345S (Florisil)	SW846-8081A	100	0.0059	8001-35-2
VOLATILE ORGANIC COMPOUNDS				µg/Kg dw (ppb)	µg/Kg dw (ppb)	
1,1,1,2-Tetrachloroethane			SW846-8260B	1.0	*	630-20-6
1,1,1-Trichloroethane			SW846-8260B	1.0	*	71-55-6
1,1,2,2-Tetrachloroethane			SW846-8260B	1.0	*	79-34-5
1,1,2-Trichloroethane			SW846-8260B	1.0	*	79-00-5
1,1-Dichloroethane			SW846-8260B	1.0	*	75-34-3
1,1-Dichloroethene			SW846-8260B SIM	0.1	*	75-35-4
1,2,3-Trichloropropane			SW846-8260B	3.0	*	96-18-4
1,2-Dichloroethane			SW846-8260B	1.0	*	107-06-2
1,2-Dichloropropane			SW846-8260B	1.0	*	78-87-5
2-Butanone			SW846-8260B	5.0	*	78-93-3
2-Chloroethyl Vinyl Ether			SW846-8260B	5.0	*	110-75-8
2-Hexanone			SW846-8260B	5.0	*	591-78-6
4-Methyl-2-Pentanone			SW846-8260B	5.0	*	108-10-1

Table A7-4. Sediment - Project Specific Method Reporting Limits, Analytical Concentration Goals, and Methodology (including SOPs).

Analytes	Extraction / Digestion SOPs	Clean Ups SOPs	Analytical Method	MRL ¹	ACG ²	CAS#
Acetone			SW846-8260B	5.0	*	67-64-1
Acrolein			SW846-8260B	50	*	107-02-8
Acrylonitrile			SW846-8260B	5.0	*	107-13-1
Benzene			SW846-8260B SIM	0.11	*	71-43-2
Bromochloromethane			SW846-8260B	1.0	*	74-97-5
Bromodichloromethane			SW846-8260B	1.0	*	75-27-4
Bromoethane			SW846-8260B	2.0	*	598-31-2
Bromoform			SW846-8260B	1.0	*	75-25-2
Bromomethane			SW846-8260B	1.0	*	74-83-9
Carbon Disulfide			SW846-8260B	1.0	*	75-15-0
Carbon Tetrachloride			SW846-8260B	1.0	*	56-23-5
Chlorobenzene			SW846-8260B	1.0	*	108-90-7
Chlorodibromomethane			SW846-8260B	1.0	*	124-48-1
Chloroethane			SW846-8260B	1.0	*	75-00-3
Chloroform			SW846-8260B	1.0	*	67-66-3
Chloromethane			SW846-8260B	1.0	*	74-87-3
<i>cis</i> - 1,3-Dichloropropene			SW846-8260B	1.0	*	10061-01-5
Dibromomethane			SW846-8260B	1.0	*	74-95-3
Dichlorodifluoromethane			SW846-8260B	1.0	*	75-71-8
Ethyl Benzene			SW846-8260B SIM	0.11	*	100-41-4
Hexachloro-1,3-Butadiene			SW846-8260B	5.0	*	87-68-3
Iodomethane			SW846-8260B	1.0	*	74-88-4
Isopropyl Benzene			SW846-8260B	10	*	98-82-8
m,p-Xylene			SW846-8260B SIM	0.22	*	108-38-3/106-42-3
Methylene Chloride			SW846-8260B	2.0	*	75-09-2
Methyl-t-butyl ether (MTBE)			SW846-8260B SIM	0.11	*	80-62-6
Naphthalene			SW846-8260B	5.0	23	91-20-3
o-Xylene			SW846-8260B SIM	0.11	*	95-47-6
Styrene			SW846-8260B	1.0	*	100-42-5
Tetrachloroethene			SW846-8260B SIM	0.1	*	127-18-4
Toluene			SW846-8260B SIM	0.11	*	108-88-3
<i>trans</i> - 1,2-Dichloroethene			SW846-8260B	0.15	*	156-60-5
<i>trans</i> - 1,3-Dichloropropene			SW846-8260B	0.11	*	10061-02-6
<i>trans</i> - 1,4-Dichloro-2-Butene			SW846-8260B	5.0	*	110-57-6
Trichloroethene			SW846-8260B SIM	0.1	*	79-01-6
Trichlorofluoromethane			SW846-8260B	1.0	*	75-69-4
Vinyl Acetate			SW846-8260B	5.0	*	108-05-4
Vinyl Chloride			SW846-8260B SIM	0.1	*	75-01-4
SEMIVOLATILE ORGANIC COMPOUNDS Full Scan				µg/Kg dw (ppb)	µg/Kg dw (ppb)	
1,2,4-Trichlorobenzene	374S		SW846-8270C	20	*	120-82-1 26
1,2-Dichlorobenzene	374S		SW846-8270C	20	184	95-50-1 12
1,3-Dichlorobenzene	374S		SW846-8270C	20	*	541-73-1 7
1,4-Dichlorobenzene	374S		SW846-8270C	20	2.0	106-46-7 9
2,2'-oxybis(1-chloropropane)	374S		SW846-8270C	20	*	108-60-1 14
2,4-Dinitrotoluene	374S		SW846-8270C	100	*	121-14-2 48

Table A7-4. Sediment - Project Specific Method Reporting Limits, Analytical Concentration Goals, and Methodology (including SOPs).

Analytes	Extraction / Digestion SOPs	Clean Ups SOPs	Analytical Method	MRL ¹	ACG ²	CAS#	
2,6-Dinitrotoluene	374S		SW846-8270C	100	*	606-20-2	41
2-Chloronaphthalene	374S		SW846-8270C	20	*	91-58-7	37
2-Nitroaniline	374S		SW846-8270C	100	*	88-74-4	38
3,3'-Dichlorbenzidine	374S		SW846-8270C	100	*	91-94-1	70
3-Nitroaniline	374S		SW846-8270C	120	*	99-09-2	43
4-bromophenyl-phenyl ether	374S		SW846-8270C	20	*	101-55-3	56
4-Chloroaniline	374S		SW846-8270C	60	*	106-47-8	29
4-Chlorophenyl-phenyl ether	374S		SW846-8270C	20	*	7005-72-3	51
4-Nitroaniline	374S		SW846-8270C	100	*	100-01-6	52
Aniline	374S		SW846-8270C	20	*	62-53-3	91
Benzoic Acid	374S		SW846-8270C	200	*	65-85-0	24
Benzyl Alcohol	374S		SW846-8270C	20	*	100-51-6	11
Bis-(2-chloroethoxy) methane	374S		SW846-8270C	20	*	111-91-1	23
Bis-(2-chloroethyl) ether	374S		SW846-8270C	40	*	111-44-4	4
Hexachlorobenzene	374S		SW846-8270C	100	0.3	118-74-1	57
Hexachlorobutadiene	374S		SW846-8270C	100	0.6	87-68-3	30
Hexachlorocyclopentadiene	374S		SW846-8270C	100	*	77-47-4	33
Isophorone	374S		SW846-8270C	20	*	78-59-1	20
Nitrobenzene	374S		SW846-8270C	20	*	98-95-3	19
n-Nitrosodimethylamine	374S		SW846-8270C	100	0.0073	62-75-9	90
n-Nitroso-di-n-propylamine	374S		SW846-8270C	20	0.053	621-64-7	16
n-Nitrosodiphenylamine	374S		SW846-8270C	20	*	86-30-6	54
Phenol				µg/Kg dw (ppb)	µg/Kg dw (ppb)		
2,4,5-trichlorophenol	374S		SW846-8270C	100	524	95-95-4	34
2,4-Dimethylphenol	374S		SW846-8270C	20	*	105-67-9	22
2,4-Dinitrophenol	374S		SW846-8270C	200	*	51-28-5	45
2-Methylphenol	374S		SW846-8270C	20	*	95-48-7	13
2-Nitrophenol	374S		SW846-8270C	100	*	88-75-5	21
4,6-Dinitro-2-Methylphenol	374S		SW846-8270C	200	*	534-52-1	53
4-Chloro-3-methylphenol	374S		SW846-8270C	40	*	59-50-7	31
4-Nitrophenol	374S		SW846-8270C	100	*	100-02-7	47
Phenol	374S		SW846-8270C	20	3146	108-95-2	3
2-Chlorophenol	374S		SW846-8270C	20	26	95-57-8	6
4-Methylphenol	374S		SW846-8270C	20	26	106-44-5	15
2,4,6-trichlorophenol	374S		SW846-8270C	100	1.8	88-06-2	35
2,4-Dichlorophenol	374S		SW846-8270C	60	16	120-83-2	25
PAH				µg/Kg dw (ppb)	µg/Kg dw (ppb)		
Naphthalene	374S		SW846-8270C	20	24	91-20-3	28
2-Methylnaphthalene	374S		SW846-8270C	20	*	91-57-6	32
Acenaphthylene	374S		SW846-8270C	20	*	208-96-8	40
Acenaphthene	374S		SW846-8270C	20	72	83-32-9	44
Fluorene	374S		SW846-8270C	20	48	86-73-7	49
Phenanthrene	374S		SW846-8270C	20	*	85-01-8	60
Anthracene	374S		SW846-8270C	20	360	120-12-7	61
Fluoranthene	374S		SW846-8270C	20	48	206-44-0	64
Pyrene	374S		SW846-8270C	20	36	129-00-0	65

Table A7-4. Sediment - Project Specific Method Reporting Limits, Analytical Concentration Goals, and Methodology (including SOPs).

Analytes	Extraction / Digestion SOPs	Clean Ups SOPs	Analytical Method	MRL ¹	ACG ²	CAS#	
Phthalate				µg/Kg dw (ppb)	µg/Kg dw (ppb)		
Dimethylphthalate	374S		SW846-8270C	20	20000	131-11-3	39
Diethylphthalate	374S		SW846-8270C	20	*	84-66-2	50
Di-n-butylphthalate	374S		SW846-8270C	20	204	84-74-2	63
Butylbenzylphthalate	374S		SW846-8270C	20	400	85-68-7	67
Di-n-octylphthalate	374S		SW846-8270C	20	40.9	117-84-0	73
bis(2-Ethylhexyl) phthalate	374S		SW846-8270C	20	3.4	117-81-7	72
SEMIVOLATILE ORGANIC COMPOUNDS Full Scan + SIM				µg/Kg dw (ppb)	µg/Kg dw (ppb)		
2,3,4,6-Tetrachlorophenol	374S		SW846-8270C	NE ^b	157	58-90-2	?
Tetrachlorophenol (2,3,4,5 and 2,3,5,6)	374S		SW846-8270C	NE ^b	157	25167-83-3	?
Hexachloroethane	374S		SW846-8270C SIM	NE ^b	2.0	67-72-1	17
Dibenzofuran	374S		SW846-8270C SIM	6.7	8.2	132-64-9	46
Pentachlorophenol	374S		SW846-8270C SIM	34	0.58	87-86-5	58
Carbazole	374S		SW846-8270C SIM	6.7	6.12	86-74-8	62
Benzo(a)Anthracene	374S		SW846-8270C SIM	6.7	0.038	56-55-3	68
Chrysene	374S		SW846-8270C SIM	6.7	3.8	218-01-9	71
Benzo(b)Fluoranthene	374S		SW846-8270C SIM	6.7	0.038	205-99-2	74
Benzo(k)Fluoranthene	374S		SW846-8270C SIM	6.7	0.38	207-08-9	75
Benzo(a)Pyrene	374S		SW846-8270C SIM	6.7	0.0038	50-32-8	76
Indeno(1,2,3-cd)Pyrene	374S		SW846-8270C SIM	6.7	0.038	193-39-5	78
Dibenz(a,h)Anthracene	374S		SW846-8270C SIM	6.7	0.0038	53-70-3	79
Benzo(ghi)Perylene	374S		SW846-8270C SIM	6.7	*	191-24-2	80
1,2-diphenylhydrazine ^f	374S		SW846-8270C SIM	20	0.0025		92
CHLORINATED BIPHENYL CONGENERS^d				pg/g dw (ppt)	pg/g dw (ppt)		
3,3',4,4'-Tetrachlorobiphenyl BZ077	A-010 REV 4 07/03/02		Method 1668A	0.5-1.0	*	32598-13-3	
2,3,3',4,4'-Pentachlorobiphenyl BZ105	A-010 REV 4 07/03/02		Method 1668A	0.5-1.0	*	32598-14-4	
2,3,4,4',5-Pentachlorobiphenyl BZ114	A-010 REV 4 07/03/02		Method 1668A	0.5-1.0	*	74472-37-0	
2,3',4,4',5-Pentachlorobiphenyl BZ118	A-010 REV 4 07/03/02		Method 1668A	0.5-1.0	*	31508-00-6	
2,3',4,4',5'-Pentachlorobiphenyl BZ123	A-010 REV 4 07/03/02		Method 1668A	0.5-1.0	*	65510-44-3	
3,3',4,4',5-Pentachlorobiphenyl BZ126	A-010 REV 4 07/03/02		Method 1668A	0.5-1.0	0.01	57465-28-8	
2,3,3',4,4',5-Hexachlorobiphenyl BZ156	A-010 REV 4 07/03/02		Method 1668A	0.5-1.0	*	38380-08-4	
2,3,3',4,4',5'-Hexachlorobiphenyl BZ157	A-010 REV 4 07/03/02		Method 1668A	0.5-1.0	*	69782-90-7	
2,3',4,4',5,5'-Hexachlorobiphenyl BZ167	A-010 REV 4 07/03/02		Method 1668A	0.5-1.0	*	52663-72-6	
3,3',4,4',5,5'-Hexachlorobiphenyl BZ169	A-010 REV 4 07/03/02		Method 1668A	0.5-1.0	*	32774-16-6	
2,2',3,3',4,4',5-Heptachlorobiphenyl BZ170	A-010 REV 4 07/03/02		Method 1668A	0.5-1.0	*	35065-30-6	
2,2',3,4,4',5,5'-Heptachlorobiphenyl BZ180	A-010 REV 4 07/03/02		Method 1668A	0.5-1.0	*	35065-29-3	
2,3,3',4,4',5,5'-Heptachlorobiphenyl BZ189	A-010 REV 4 07/03/02		Method 1668A	0.5-1.0	*	39635-31-9	
TETRA-OCTA-CHLORINATED DIOXINS AND FURANS^e				pg/g dw (ppt)	pg/g dw (ppt)		
2,3,7,8-TCDD	›X-1613B REV 7 05/07/01		Method 1613B	0.01	0.0001	1746-01-6	
2,3,7,8-TCDF	›X-1613B REV 7 05/07/01		Method 1613B	0.01	0.001	51207-31-9	
1,2,3,7,8-PeCDD	›X-1613B REV 7 05/07/01		Method 1613B	0.01	0.001	40321-76-4	
1,2,3,7,8-PeCDF	›X-1613B REV 7 05/07/01		Method 1613B	0.01	0.001	57117-41-6	
2,3,4,7,8-PeCDF	›X-1613B REV 7 05/07/01		Method 1613B	0.01	0.0002	57117-31-4	
1,2,3,4,7,8-HxCDD	›X-1613B REV 7 05/07/01		Method 1613B	0.01	0.01	39227-28-6	
1,2,3,6,7,8-HxCDD	›X-1613B REV 7 05/07/01		Method 1613B	0.01	0.01	57653-85-7	

Table A7-4. Sediment - Project Specific Method Reporting Limits, Analytical Concentration Goals, and Methodology (including SOPs).

Analytes	Extraction / Digestion SOPs	Clean Ups SOPs	Analytical Method	MRL ¹	ACG ²	CAS#
1,2,3,7,8,9-HxCDD	X-1613B REV 7 05/07/01		Method 1613B	0.01	0.01	19408-74-3
1,2,3,4,7,8-HxCDF	X-1613B REV 7 05/07/01		Method 1613B	0.01	0.01	70648-26-9
1,2,3,6,7,8-HxCDF	X-1613B REV 7 05/07/01		Method 1613B	0.01	0.01	57117-44-9
1,2,3,7,8,9-HxCDF	X-1613B REV 7 05/07/01		Method 1613B	0.01	0.01	72918-21-9
2,3,4,6,7,8-HxCDF	X-1613B REV 7 05/07/01		Method 1613B	0.01	0.01	60851-34-5
1,2,3,4,6,7,8-HpCDD	X-1613B REV 7 05/07/01		Method 1613B	0.03	0.09	35822-46-9
1,2,3,4,6,7,8-HpCDF	X-1613B REV 7 05/07/01		Method 1613B	0.03	0.09	67562-39-4
1,2,3,4,7,8,9-HpCDF	X-1613B REV 7 05/07/01		Method 1613B	0.03	0.09	55673-89-7
OCDD	X-1613B REV 7 05/07/01		Method 1613B	0.05	9.4	3268-87-9
OCDF	X-1613B REV 7 05/07/01		Method 1613 B	0.05	9.4	39001-02-0

^a = Sample amount extracted 25 grams with a final extract volume of 1 ml

^b = R&D would need to be performed.

^c = Estimation based off "clean sediment"

^d = 10 g sample weight. Sample amount, final extract volume and injection size adjustments will be made by the laboratory to get the MRLs closer to the ACGs.

^e = 50g sample weight. Normal sediments have higher results for the Octa then do they for the Tetra congeners. With the 0.003 ug/kg MRL established by EPA, a larger

^f = Diphenylhydrazine cannot be separated from Azobenzene using this method.

^g = Total chlordane will be calculated off of the 5 isomers. The isomers do not have established ACGs.

¹ = MRL are project specific.

² = ACG are the "goals" established by EPA from Ad Hoc meeting with LWG May 10, 2002.

NA = Non-Applicable

NE = Non-Established: An MRL has not been established.

Bold = ACG not met with present laboratory methodology

* = A risk based ACG has not been established.

Table A7-5. Tissue - Project Specific Method Reporting Limits, Analytical Concentration Goals, and Methodology.

Analytes	Extraction SOPs	Clean Ups SOPs	Clean Ups SOPs	Analytical Method	MRL ¹	ACG ²	CAS#
Conventionals							
Lipids	SOC-LIPID			See QAPP for guidance	1%	*	NA
Total Solids				Lyphosation CAS inhouse SOP	0.01 %	*	NA
Metals							
					mg/kg ww (ppm)	mg/kg ww (ppm)	
Silver - Ag	GEN-TISP, MET-TDIG			SW846-6020 ICP-MS (MET-6020)	0.004	0.089	7782-49-2
Aluminum - Al	GEN-TISP, MET-TDIG			SW846-6020 ICP-MS (MET-6020)	0.1	*	7429-90-5
Arsenic -As	GEN-TISP, MET-TDIG			SW846-6020 ICP-MS (MET-6020)	0.1	0.00027	7440-66-6
Cadmium - Cd	GEN-TISP, MET-TDIG			SW846-6020 ICP-MS (MET-6020)	0.01	0.01	7440-43-9
Chromium - Cr	GEN-TISP, MET-TDIG			SW846-6010B ICP(MET-6010)	0.1	0.054	7440-47-3
Copper - Cu	GEN-TISP, MET-TDIG			SW846-6020 ICP-MS (MET-6020)	0.02	0.67	7440-50-8
Manganese - Mn	GEN-TISP, MET-TDIG			SW846-6020 ICP-MS (MET-6020)	0.01	0.431	
Nickel - Ni	GEN-TISP, MET-TDIG			SW846-6020 ICP-MS (MET-6020)	0.04	0.36	7440-50-8
Lead - Pb	GEN-TISP, MET-TDIG			SW846-6020 ICP-MS (MET-6020)	0.004	*	7439-92-1
Antimony - Sb	GEN-TISP, MET-TDIG			SW846-6020 ICP-MS (MET-6020)	0.004	*	7440-36-0
Selenium - Se	GEN-TISP, MET-TDIG			SW846-7740 GFAA (MET-GFAA)	0.2	*	7782-49-2
Thallium - Tl	GEN-TISP, MET-TDIG			SW846-6020 ICP-MS (MET-6020)	0.004	0.001	
Zinc - Zn	GEN-TISP, MET-TDIG			SW846-6020 ICP-MS (MET-6020)	0.1	5.4	7440-66-6
Mercury - Hg	MET-7471A			SW846-7471A CVAA (MET-7471A)	0.004	0.005	7439-97-6
Butyltins							
					µg/kg ww (ppb)	µg/kg ww (ppb)	
Monobutyltin	SOC-OSWT	SOC-3630		SOC-BUTYL	2.0	*	78763-54-9
Dibutyltin	SOC-OSWT	SOC-3630		SOC-BUTYL	2.0	*	1002-53-5
Tributyltin	SOC-OSWT	SOC-3630		SOC-BUTYL	2.0	5.4	56573-85-4
Tetrabutyltin	SOC-OSWT	SOC-3630		SOC-BUTYL	10	*	1461-25-2
PCBs Aroclors							
					µg/kg ww (ppb)	µg/kg ww (ppb)	
Aroclor 1016	EXT-3540	EXT-FLOR	SOC-3665 & SOC-3640A	SOC-8082A	2.0	0.21	12674-11-2
Aroclor 1221	EXT-3540	EXT-FLOR	SOC-3665 & SOC-3640A	SOC-8082A	4.0	0.21	11104-28-2
Aroclor 1232	EXT-3540	EXT-FLOR	SOC-3665 & SOC-3640A	SOC-8082A	2.0	0.21	11141-16-5
Aroclor 1242	EXT-3540	EXT-FLOR	SOC-3665 & SOC-3640A	SOC-8082A	2.0	0.21	53469-21-9
Aroclor 1248	EXT-3540	EXT-FLOR	SOC-3665 & SOC-3640A	SOC-8082A	2.0	0.21	12672-29-6
Aroclor 1254	EXT-3540	EXT-FLOR	SOC-3665 & SOC-3640A	SOC-8082A	2.0	0.21	11097-69-1
Aroclor 1260	EXT-3540	EXT-FLOR	SOC-3665 & SOC-3640A	SOC-8082A	2.0	0.21	11096-82-5
ORGANOCHLORINE PESTICIDES							
					µg/kg ww (ppb)	µg/kg ww (ppb)	
2,4'-DDD	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	*	53-19-0
2,4'-DDE	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	*	3424-82-6
2,4'-DDT	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	*	789-02-6
4,4'-DDD	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	5.4	72-54-8
4,4'-DDE	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	3.8	72-55-9
4,4'-DDT	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	3.8	50-29-3
Total DDT						*	
Aldrin	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	0.025	309-00-2
a - BHC	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	0.067	319-84-6
b - BHC	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	0.233	319-85-7
g - BHC (Lindane)	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	0.322	58-89-9
d - BHC	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	*	319-86-8
g - Chlordane	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	*	5103-74-2
a - Chlordane	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	*	5103-71-9
oxy - chlordane	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	*	26880-48-8

Table A7-5. Tissue - Project Specific Method Reporting Limits, Analytical Concentration Goals, and Methodology.

Analytes	Extraction SOPs	Clean Ups SOPs	Clean Ups SOPs	Analytical Method	MRL ¹	ACG ²	CAS#
<i>cis</i> - nonachlor	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	*	5103-73-1
<i>trans</i> - nonachlor	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	*	39765-80-5
total Chlordane ^c					5.0	3.7	
Dieldrin	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	0.026	60-57-1
Endosulfan I	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	108	959-98-8
Endosulfan II	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	*	33213-65-9
Endosulfan sulfate	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	*	1031-07-8
Endrin	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	5.4	72-20-8
Endrin aldehyde	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	*	7421-93-4
Endrin ketone	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	*	53494-70-5
Heptachlor	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	0.0933	76-44-8
Heptachlor epoxide	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	0.046	1024-57-3
Hexachlorobenzene	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	0.26	118-74-1
Hexachlorobutadiene	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	5.4	87-68-3
Hexachloroethane	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	18	67-72-1
Methoxychlor	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	90	72-43-5
Mirex	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	1.0	3.6	2385-85-5
Toxaphene	EXT-3540	EXT-FLOR	SOC-3640A	SOC-8081	50	0.38	8001-35-2
SEMIVOLATILE ORGANIC COMPOUNDS Full Scan					µg/kg ww (ppb)	µg/kg ww (ppb)	
2,2'-oxybis(1-chloropropane)	340S			SW846-8270C	300	*	108-60-1 14
2-Chloronaphthalene	340S			SW846-8270C	200	*	91-58-7 37
2-Nitroaniline	340S			SW846-8270C	500	*	88-74-4 38
3,3'-Dichlorbenzidine	340S			SW846-8270C	500	*	91-94-1 70
3-Nitroaniline	340S			SW846-8270C	500	*	99-09-2 43
4-Nitroaniline	340S			SW846-8270C	500	*	100-01-6 52
Aniline	340S			SW846-8270C	200	*	62-53-3 91
Benzoic Acid	340S			SW846-8270C	1000	72000	65-85-0 24
Benzyl Alcohol	340S			SW846-8270C	600	5400	100-51-6 11
Bis-(2-chloroethoxy) methane	340S			SW846-8270C	100	*	111-91-1 23
Hexachlorocyclopentadiene	340S			SW846-8270C	500	*	77-47-4 33
Isophorone	340S			SW846-8270C	200	*	78-59-1 20
n-Nitrosodiphenylamine	340S			SW846-8270C	200	*	86-30-6 54
Phenols					µg/kg ww (ppb)	µg/kg ww (ppb)	
2,4,5-trichlorophenol	340S			SW846-8270C	500	1800	95-95-4 34
2,4-Dimethylphenol	340S			SW846-8270C	200	*	105-67-9 22
2,4-Dinitrophenol	340S			SW846-8270C	1000	*	51-28-5 45
2-Methylphenol	340S			SW846-8270C	600	*	95-48-7 13
2-Nitrophenol	340S			SW846-8270C	500	*	88-75-5 21
4,6-Dinitro-2-Methylphenol	340S			SW846-8270C	1000	*	534-52-1 53
4-Chloro-3-methylphenol	340S			SW846-8270C	200	*	59-50-7 31
4-Nitrophenol	340S			SW846-8270C	600	*	100-02-7 47
Phenol	340S			SW846-8270C	300	10800	108-95-2 3
Tetrachlorophenol (2,3,4,5 and 2,3,5,6)	340S			SW846-8270C	NE	540	58-90-2 ?
Phthalate esters					µg/kg ww (ppb)	µg/kg ww (ppb)	
Butylbenzylphthalate	340S			SW846-8270C	200	3600	85-68-7 67
Diethylphthalate	340S			SW846-8270C	200	*	84-66-2 50
Dimethylphthalate	340S			SW846-8270C	200	180,000	131-11-3 39
Di-n-butylphthalate	340S			SW846-8270C	200	1800	84-74-2 63
Di-n-octylphthalate	340S			SW846-8270C	200	360	117-84-0 73

Table A7-5. Tissue - Project Specific Method Reporting Limits, Analytical Concentration Goals, and Methodology.

Analytes	Extraction SOPs	Clean Ups SOPs	Clean Ups SOPs	Analytical Method	MRL ¹	ACG ²	CAS#	
SEMIVOLATILE ORGANIC COMPOUNDS Full scan + SIMs					µg/kg ww (ppb)	µg/kg ww (ppb)		
1,2,4-Trichlorobenzene	340S	371S (Alumina)		SW846-8270C SIM	12.5	*	120-82-1	26
1,2-Dichlorobenzene	340S	371S (Alumina)		SW846-8270C SIM	12.5	1620	95-50-1	12
1,3-Dichlorobenzene	340S	371S (Alumina)		SW846-8270C SIM	12.5	*	541-73-1	7
2,4-Dinitrotoluene	340S	371S (Alumina)		SW846-8270C SIM	12.5	*	121-14-2	48
2,6-Dinitrotoluene	340S	371S (Alumina)		SW846-8270C SIM	12.5	*	606-20-2	41
2-Chloronaphthalene	340S	371S (Alumina)		SW846-8270C SIM	12.5	*	91-58-7	37
4-bromophenyl-phenyl ether	340S	371S (Alumina)		SW846-8270C SIM	12.5	*	101-55-3	56
4-Chlorophenyl-phenyl ether	340S	371S (Alumina)		SW846-8270C SIM	12.5	*	7005-72-3	51
Bis-(2-chloroethyl) ether	340S	371S (Alumina)		SW846-8270C SIM	12.5	*	111-44-4	4
Hexachlorobenzene	340S	371S (Alumina)		SW846-8270C SIM	12.5	0.26	118-74-1	57
Hexachlorobutadiene	340S	371S (Alumina)		SW846-8270C SIM	12.5	5.4	87-68-3	30
Nitrobenzene	340S	371S (Alumina)		SW846-8270C SIM	12.5	*	98-95-3	19
1,2-diphenylhydrazine ^b	340S	371S (Alumina)		SW846-8270C SIM	12.5	0.16		111
1,4-Dichlorobenzene	340S	371S (Alumina)		SW846-8270C SIM	12.5	17	106-46-7	9
Hexachloroethane	340S	371S (Alumina)		SW846-8270C SIM	12.5	18	67-72-1	17
2,4,6-trichlorophenol	340S			SW846-8270C SIM	1000	117	88-06-2	35
2,4-Dichlorophenol	340S			SW846-8270C SIM	600	54	120-83-2	25
4-Chloroaniline	340S			SW846-8270C SIM	600	*	106-47-8	29
4-Methylphenol	340S			SW846-8270C SIM	200	90	106-44-5	15
bis(2-Ethylhexyl) phthalate	340S			SW846-8270C SIM	200	30	117-81-7	72
2-Chlorophenol	340S			SW846-8270C SIM	200	90	95-57-8	6
n-Nitrosodimethylamine	340S			SW846-8270C SIM	400	0.025	62-75-9	90
n-Nitroso-di-n-propylamine	340S			SW846-8270C SIM	1000	0.18	621-64-7	16
Pentachlorophenol	340S			SW846-8270C SIM	1000	3.5	87-86-5	58
PAHs					µg/kg ww (ppb)	µg/kg ww (ppb)		
2-Methylnaphthalene	340S	371S (Alumina)		SW846-8270C SIM	12.5	*	91-57-6	32
Acenaphthene	340S	371S (Alumina)		SW846-8270C SIM	12.5	1080	83-32-9	44
Acenaphthylene	340S	371S (Alumina)		SW846-8270C SIM	12.5	*	208-96-8	40
Anthracene	340S	371S (Alumina)		SW846-8270C SIM	12.5	5400	120-12-7	61
Benzo(a)Anthracene	340S	371S (Alumina)		SW846-8270C SIM	12.5	0.575	56-55-3	68
Benzo(a)Pyrene	340S	371S (Alumina)		SW846-8270C SIM	12.5	0.0575	50-32-8	76
Benzo(b)Fluoranthene	340S	371S (Alumina)		SW846-8270C SIM	12.5	0.575	205-99-2	74
Benzo(ghi)Perylene	340S	371S (Alumina)		SW846-8270C SIM	12.5	*	191-24-2	80
Benzo(k)Fluoranthene	340S	371S (Alumina)		SW846-8270C SIM	12.5	5.75	207-08-9	75
Carbazole	340S	371S (Alumina)		SW846-8270C SIM	12.5	21	86-74-8	62
Chrysene	340S	371S (Alumina)		SW846-8270C SIM	12.5	58	218-01-9	71
Dibenz(a,h)Anthracene	340S	371S (Alumina)		SW846-8270C SIM	12.5	0.0575	53-70-3	79
Dibenzofuran	340S	371S (Alumina)		SW846-8270C SIM	12.5	72	132-64-9	46
Fluoranthene	340S	371S (Alumina)		SW846-8270C SIM	12.5	720	206-44-0	64
Fluorene	340S	371S (Alumina)		SW846-8270C SIM	12.5	720	86-73-7	49
Indeno(1,2,3-cd)Pyrene	340S	371S (Alumina)		SW846-8270C SIM	12.5	0.575	193-39-5	78
Naphthalene	340S	371S (Alumina)		SW846-8270C SIM	12.5	16	91-20-3	28
Phenanthrene	340S	371S (Alumina)		SW846-8270C SIM	12.5	*	85-01-8	60
Pyrene	340S	371S (Alumina)		SW846-8270C SIM	12.5	540	129-00-0	65
CHLORINATED BIPHENYL CONGENERs					pg/g ww (ppt)	pg/g ww (ppt)		
3,3',4,4'-Tetrachlorobiphenyl BZ077	MLA-010 REV 4 07/03/02			Method 1668 A	0.1 ^a	*	32598-13-3	
2,3,3',4,4'-Pentachlorobiphenyl BZ105	MLA-010 REV 4 07/03/02			Method 1668 A	0.1 ^a	*	32598-14-4	
2,3,4,4',5-Pentachlorobiphenyl BZ114	MLA-010 REV 4 07/03/02			Method 1668 A	0.1 ^a	*	74472-37-0	

Table A7-5. Tissue - Project Specific Method Reporting Limits, Analytical Concentration Goals, and Methodology.

Analytes	Extraction SOPs	Clean Ups SOPs	Clean Ups SOPs	Analytical Method	MRL ¹	ACG ²	CAS#
2,3',4,4',5-Pentachlorobiphenyl BZ118	MLA-010 REV 4 07/03/02			Method 1668 A	0.1 ^a	*	31508-00-6
2,3',4,4',5'-Pentachlorobiphenyl BZ123	MLA-010 REV 4 07/03/02			Method 1668 A	0.1 ^a	*	65510-44-3
3,3',4,4',5-Pentachlorobiphenyl BZ126	MLA-010 REV 4 07/03/02			Method 1668 A	0.1 ^a	0.03	57465-28-8
2,3,3',4,4',5-Hexachlorobiphenyl BZ156	MLA-010 REV 4 07/03/02			Method 1668 A	0.1 ^a	*	38380-08-4
2,3,3',4,4',5'-Hexachlorobiphenyl BZ157	MLA-010 REV 4 07/03/02			Method 1668 A	0.1 ^a	*	69782-90-7
2,3',4,4',5'-Hexachlorobiphenyl BZ167	MLA-010 REV 4 07/03/02			Method 1668 A	0.1 ^a	*	52663-72-6
3,3',4,4',5'-Hexachlorobiphenyl BZ169	MLA-010 REV 4 07/03/02			Method 1668 A	0.1 ^a	*	32774-16-6
2,2',3,3',4,4',5-Heptachlorobiphenyl BZ170	MLA-010 REV 4 07/03/02			Method 1668 A	0.1 ^a	*	35065-30-6
2,2',3,4,4',5'-Heptachlorobiphenyl BZ180	MLA-010 REV 4 07/03/02			Method 1668 A	0.1 ^a	*	35065-29-3
2,3,3',4,4',5'-Heptachlorobiphenyl BZ189	MLA-010 REV 4 07/03/02			Method 1668 A	0.1 ^a	*	39635-31-9
TETRA-OCTA-CHLORINATED DIOXINS AND FURANS					pg/g ww (ppt)	pg/g ww (ppt)	
2,3,7,8-TCDD	DOC DX-1613B REV 7 05/07/01			Method 1613 B	0.003 ^a	0.0028	1746-01-6
2,3,7,8-TCDF	DOC DX-1613B REV 7 05/07/01			Method 1613 B	0.003 ^a	0.028	51207-31-9
1,2,3,7,8-PeCDD	DOC DX-1613B REV 7 05/07/01			Method 1613 B	0.003 ^a	0.0028	40321-76-4
1,2,3,7,8-PeCDF	DOC DX-1613B REV 7 05/07/01			Method 1613 B	0.003 ^a	0.056	57117-41-6
2,3,4,7,8-PeCDF	DOC DX-1613B REV 7 05/07/01			Method 1613 B	0.003 ^a	0.0056	57117-31-4
1,2,3,4,7,8-HxCDD	DOC DX-1613B REV 7 05/07/01			Method 1613 B	0.003 ^a	0.028	39227-28-6
1,2,3,6,7,8-HxCDD	DOC DX-1613B REV 7 05/07/01			Method 1613 B	0.003 ^a	0.028	57653-85-7
1,2,3,7,8,9-HxCDD	DOC DX-1613B REV 7 05/07/01			Method 1613 B	0.003 ^a	0.028	19408-74-3
1,2,3,4,7,8-HxCDF	DOC DX-1613B REV 7 05/07/01			Method 1613 B	0.003 ^a	0.028	70648-26-9
1,2,3,6,7,8-HxCDF	DOC DX-1613B REV 7 05/07/01			Method 1613 B	0.003 ^a	0.028	57117-44-9
1,2,3,7,8,9-HxCDF	DOC DX-1613B REV 7 05/07/01			Method 1613 B	0.003 ^a	0.028	72918-21-9
2,3,4,6,7,8-HxCDF	DOC DX-1613B REV 7 05/07/01			Method 1613 B	0.003 ^a	0.028	60851-34-5
1,2,3,4,6,7,8-HpCDD	DOC DX-1613B REV 7 05/07/01			Method 1613 B	0.003 ^a	0.028	35822-46-9
1,2,3,4,6,7,8-HpCDF	DOC DX-1613B REV 7 05/07/01			Method 1613 B	0.003 ^a	0.28	67562-39-4
1,2,3,4,7,8,9-HpCDF	DOC DX-1613B REV 7 05/07/01			Method 1613 B	0.003 ^a	0.28	55673-89-7
OCDD	DOC DX-1613B REV 7 05/07/01			Method 1613 B	0.015 ^a	2.8	3268-87-9
OCDF	DOC DX-1613B REV 7 05/07/01			Method 1613 B	0.015 ^a	2.8	39001-02-0

^a = Based on a 75g sample weight.^b = Diphenylhydrazine cannot be separated from Azobenzene using this method.^c = Total chlordane will be calculated off of the 5 isomers. The isomers do not have established ACGs.¹ = MRL are project specific.² = ACG are the "goals" established by EPA from Ad Hoc meeting with LWG May 10, 2002.

NA = Non-Applicable

NE = Non-Established: An MDL has not been established.

* = A risk based ACG has not been established.

Bold = Not able to meet with present laboratory methodology*Italic = Waiting on MDL study to be finished by ARI. These MRLs are full scan.*

Table A7-6. Sediment - QA/QC Sample Control Limits for the Chemical Analyses.

Analysis ^{a,b}	Analytes	Percent Recovery		RPD	PARCC % Complete	
		Sample Matrix Spike	Method Blank/LCS			
<i>Volatile Organics SW846-8260B^d surrogates</i>	<i>Dibromomethane</i>	50-150	50-150	30	90	
	<i>d4-1,2-Dichloroethane</i>	50-150	50-150			
	<i>d8-Toluene</i>	50-150	50-150			
	<i>4-Bromofluorobenzene</i>	50-150	50-150			
	<i>d4-1,2-Dichlorobenzene</i>	50-150	50-150			
	<i>ms spikes</i>	<i>Vinyl Chloride</i>	50-150			
		<i>1,1-Dichloroethane</i>	50-150			
		<i>Chloroform</i>	50-150			
		<i>1,2-Dichloropropane</i>	50-150			
		<i>Trichloroethene</i>	50-150			
		<i>Benzene</i>	50-150			
		<i>Toluene</i>	50-150			
	<i>lcs spikes</i>	<i>Chlorobenzene</i>	50-150			
		<i>Ethylbenzene</i>	50-150			
		<i>Chloromethane</i>				41-132
<i>Bromomethane</i>			67-142			
<i>Vinyl Chloride</i>			51-145			
<i>Chloroethane</i>			65-131			
<i>Methylene Chloride</i>			23-134			
<i>Acetone</i>			44-126			
<i>Carbon Disulfide</i>			49-151			
<i>1,1-Dichloroethene</i>			66-135			
<i>1,1-Dichloroethane</i>			78-128			
<i>trans-1,2-Dichloroethene</i>			78-133			
<i>cis-1,2-Dichloroethene</i>			85-134			
<i>Chloroform</i>			71-124			
<i>1,2-Dichloroethane</i>			69-136			
<i>2-Butanone</i>			58-137			
<i>1,1,1-Trichloroethane</i>			82-129			
<i>Carbon Tetrachloride</i>			82-137			
<i>Vinyl Acetate</i>		43-110				
<i>Bromodichloromethane</i>		85-126				
<i>1,2-Dichloropropane</i>		84-121				
<i>cis-1,3-dichloropropene</i>		70-133				

Table A7-6. Sediment - QA/QC Sample Control Limits for the Chemical Analyses.

Analysis ^{a,b}	Analytes	Percent Recovery		RPD	PARCC % Complete
		Sample Matrix Spike	Method Blank/LCS		
	<i>Tirchloroethene</i>		66-130		
	<i>Dibromochloromethane</i>		85-136		
	<i>1,1,2-Trichloroethane</i>		75-124		
	<i>Benzene</i>		85-121		
	<i>trans-1-Dichloropropene</i>		80-131		
	<i>2-Chloroethylvinylether</i>		64-146		
	<i>Bromoform</i>		83-134		
	<i>4-Methyl-2-pentanone</i>		65-138		
	<i>2-Hexanone</i>		60-145		
	<i>Tetrachloroethylene</i>		87-132		
	<i>1,1,2,2-tetrachloroethane</i>		78-130		
	<i>Toluene</i>		75-123		
	<i>Chlorobenzene</i>		88-118		
	<i>Ethylbenzene</i>		88-127		
	<i>Styrene</i>		88-122		
	<i>Xylenes, Total</i>		83-123		
	<i>Trichlorofluoromehtane</i>		62-136		
	<i>Trichloroftrifluoroethane</i>		53-135		
	<i>tot 1,3-Dichloropropene</i>		na		
	<i>m,p-Xylene</i>		85-124		
	<i>o-Xylene</i>		84-120		
	<i>1,2-Dichlorobenzene</i>		88-119		
	<i>1,3-Dichlorobenzene</i>		88-123		
	<i>1,4-Dichlorobenzene</i>		87-121		
	<i>Acrolein</i>		52-166		
	<i>Methyl Iodide</i>		42-127		
	<i>Bromoethane</i>		56-124		
	<i>Acrylonitile</i>		55-137		
	<i>1,1-Dichlorpropene</i>		85-130		
	<i>Dibromomethane</i>		76-126		
	<i>1,1,1,2-Tetrachloroethane</i>		84-131		
	<i>Dibromo-3-chloropropane</i>		59-134		
	<i>1,2,3-Trichloroprpane</i>		75-137		

Table A7-6. Sediment - QA/QC Sample Control Limits for the Chemical Analyses.

Analysis ^{a,b}	Analytes	Percent Recovery		RPD	PARCC % Complete
		Sample Matrix Spike	Method Blank/LCS		
	<i>trans</i> - 1,4-Dichloro-2-butene		49-119		
	1,3,5-Trimethylbenzene		89-134		
	1,2,4-Trimethylbenzene		91-135		
	1,2,3-Trimethylbenzene		na		
	Hexachlorobutadiene		73-144		
	Ethylene Dibromide		69-128		
	Bromochloromethane		81-123		
	2,2-Dichloropropane		81-139		
	1,3-Dichloropropane		81-128		
	Isopropylbenzene		90-134		
	<i>n</i> -Propylbenzene		90-137		
	Bromobenzene		92-124		
	2-Chlortoluene		83-133		
	4-Chlortoluene		83-132		
	<i>tert</i> -Butylbenzene		87-130		
	<i>sec</i> -Butylbenzene		90-134		
	4-Isopropyltoluene		91-135		
	<i>n</i> -Butylbenzene		88-136		
	1,2,4-Trichlorobenzene		79-130		
	Naphthalene		68-135		
	1,2,3-Trichlorobenzene		79-128		
<i>Semivolatiles SW846-8270C surrogates</i>	<i>d</i> 4-2-Chlorophenol	20-108	41-114	30	90
	<i>d</i> 4-1,2-Dichlorobenzene	12-108	33-120		
	2,4,6-Tribromophenol	10-124	32-105		
	2-Fluorophenol	17-110	42-118		
	<i>d</i> 5-Phenol	16-116	36-121		
	<i>d</i> 5-Nitrobenzene	11-111	39-113		
	2-Fluorobiphenyl	10-124	37-119		
	<i>d</i> 14- <i>p</i> -Terphenyl	13-125	36-125		
<i>Semivolatiles SW846-8270C spikes</i>	Phenol	25-116	45-123		
	2-Chlorophenol	23-113	49-116		
	1,4-Dichlorobenzene	16-108	30-115		
	<i>N</i> -nitroso- <i>di-n</i> -propylamine	12-121	26-102		
	1,2,4-Trichlorobenzene	23-112	35-111		
	4-Chloro-3-methylphenol	24-121	31-119		

Table A7-6. Sediment - QA/QC Sample Control Limits for the Chemical Analyses.

Analysis ^{a,b}	Analytes	Percent Recovery		RPD	PARCC % Complete
		Sample Matrix Spike	Method Blank/LCS		
	<i>Acenaphthene</i>	15-125	37-102		
	<i>4-Nitrophenol</i>	10-141	28-111		
	<i>2,4-Dinitrotoluene</i>	10-139	33-118		
	<i>Pentachlorophenol</i>	10-141	18-125		
	<i>Pyrene</i>	10-139	29-95		
<i>SIM surrogate</i>	<i>d10-2-Methylnaphthalene</i>	36-122	52-104		
	<i>d14-Dibenzo (a,h) anthracene</i>	18-139	40-123		
<i>SIM spikes</i>	<i>Phenanthrene</i>	49-118	58-114		
	<i>Benzo (k) fluoranthene</i>	38-124	50-15		
	<i>Chrysene</i>	42-117	58-108		
<i>Pesticides SW846-8081A surrogates</i>	<i>Tetrachloro-meta-xylene (TCMX)</i>	24-111	38-114	30	90
	<i>Decachlorobiphenyl</i>	20-119	51-112		
<i>spikes</i>	<i>Lindane</i>	10-130	34-120		
	<i>Heptachlor</i>	10-127	49-108		
	<i>Aldrin</i>	10-112	43-102		
	<i>Dieldrin</i>	10-144	54-116		
	<i>Endrin</i>	10-147	56-123		
	<i>DDT</i>	10-161	48-127		
<i>PCBs SW846-8082A surrogates</i>	<i>Tetrachloro-meta-xylene (TCMX)</i>	18-132	37-139	30	90
	<i>Decachlorobiphenyl</i>	16-140	50-127		
<i>PCBs SW846-8082A spikes</i>	<i>Aroclor 1242</i>	10-146	51-126		
<i>Herbicides SW846-8151 surrogates</i>	<i>2,4-Dichlorophenylacetic acid</i>	22-153	22-151	30	90
<i>spikes</i>	<i>2,4,5-TP</i>	22-162	47-118		
	<i>2,4-D</i>	27-122	46-90		
	<i>Dicamba</i>	17-179	47-118		
<i>TBT (lab specific methods) surrogates</i>	<i>Tripentyl Tin</i>	13-113	25-127	30	90
	<i>Tripropyl Tin</i>	21-125	36-137		
<i>TBT spikes</i>	<i>Tetrabutyl Tin</i>	25-126	24-134		
	<i>Tributyl Tin</i>	10-203	38-188		
	<i>Dibutyl Tin</i>	29-147	23-182		
	<i>Butyl Tin</i>	10.0-98	10-186		
<i>PCB Congeners Method 1668Revision A surrogates</i>	<i>13C12 -3,3',4,4'-Tetrachlorobiphenyl BZ077</i>	25-150	30-140	30	90

Table A7-6. Sediment - QA/QC Sample Control Limits for the Chemical Analyses.

Analysis ^{a,b}	Analytes	Percent Recovery		RPD	PARCC % Complete
		Sample Matrix Spike	Method Blank/LCS		
<i>PCB Congeners Method 1668 Revision A spikes</i>	<i>13C12 -2,3,3',4,4'-Pentachlorobiphenyl BZ105</i>	25-150	30-140		
	<i>13C12 -2,3,4,4',5-Pentachlorobiphenyl BZ114</i>	25-150	30-140		
	<i>13C12 -2,3',4,4',5-Pentachlorobiphenyl BZ118</i>	25-150	30-140		
	<i>13C12 -2,3',4,4',5'-Pentachlorobiphenyl BZ123</i>	25-150	30-140		
	<i>13C12 -3,3',4,4',5-Pentachlorobiphenyl BZ126</i>	25-150	30-140		
	<i>13C12 -2,3,3',4,4',5-Hexachlorobiphenyl BZ156</i>	25-150	30-140		
	<i>13C12 -2,3,3',4,4',5'-Hexachlorobiphenyl BZ157</i>	25-150	30-140		
	<i>13C12 -2,3',4,4',5,5'-Hexachlorobiphenyl BZ167</i>	25-150	30-140		
	<i>13C12 -3,3',4,4',5,5'-Hexachlorobiphenyl BZ169</i>	25-150	30-140		
	<i>13C12 -2,3,3',4,4',5,5'-Heptachlorobiphenyl BZ189</i>	25-150	30-140		
	<i>3,3',4,4'-Tetrachlorobiphenyl BZ077</i>	NA	50-150		
	<i>2,3,3',4,4'-Pentachlorobiphenyl BZ105</i>	NA	50-150		
	<i>2,3,4,4',5-Pentachlorobiphenyl BZ114</i>	NA	50-150		
	<i>2,3',4,4',5-Pentachlorobiphenyl BZ118</i>	NA	50-150		
	<i>2,3',4,4',5'-Pentachlorobiphenyl BZ123</i>	NA	50-150		
	<i>3,3',4,4',5-Pentachlorobiphenyl BZ126</i>	NA	50-150		
	<i>2,3,3',4,4',5-Hexachlorobiphenyl BZ156</i>	NA	50-150		
	<i>2,3,3',4,4',5'-Hexachlorobiphenyl BZ157</i>	NA	50-150		
	<i>2,3',4,4',5,5'-Hexachlorobiphenyl BZ167</i>	NA	50-150		
	<i>3,3',4,4',5,5'-Hexachlorobiphenyl BZ169</i>	NA	50-150		
<i>2,2',3,3',4,4',5-Heptachlorobiphenyl BZ170</i>	NA	50-150			
<i>2,2',3,4,4',5,5'-Heptachlorobiphenyl BZ180</i>	NA	50-150			
<i>2,3,3',4,4',5,5'-Heptachlorobiphenyl BZ189</i>	NA	50-150			
<i>PCDDs/PCDFs EPA 1613 surrogates^a</i>	<i>¹³C₁₂-2,3,7,8-TCDD</i>	25-164	20-175	30	90
	<i>¹³C₁₂-2,3,7,8-TCDF</i>	24-169	22-152		
	<i>¹³C₁₂-1,2,3,7,8-PeCDD</i>	25-181	21-227		
	<i>¹³C₁₂-1,2,3,7,8-PeCDF</i>	24-185	21-192		
	<i>¹³C₁₂-2,3,4,7,8-PeCDF</i>	21-178	13-328		
	<i>¹³C₁₂-1,2,3,4,7,8-HxCDD</i>	32-141	21-193		
	<i>¹³C₁₂-1,2,3,6,7,8-HxCDD</i>	28-130	25-163		
	<i>¹³C₁₂-1,2,3,4,7,8-HxCDF</i>	26-152	19-202		
	<i>¹³C₁₂-1,2,3,6,7,8-HxCDF</i>	26-123	21-159		
	<i>¹³C₁₂-1,2,3,7,8,9-HxCDF</i>	29-147	17-205		

Table A7-6. Sediment - QA/QC Sample Control Limits for the Chemical Analyses.

Analysis ^{a,b}	Analytes	Percent Recovery		RPD	PARCC % Complete
		Sample Matrix Spike	Method Blank/LCS		
	<i>¹³C₁₂-2,3,4,6,7,8,-HxCDF</i>	28-136	22-176		
	<i>¹³C₁₂-1,2,3,4,6,7,8-HpCDD</i>	23-140	26-166		
	<i>¹³C₁₂-1,2,3,4,6,7,8-HpCDF</i>	28-143	21-158		
	<i>¹³C₁₂-1,2,3,4,7,8,9-HpCDF</i>	26-138	20-186		
	<i>¹³C₁₂-OCDD</i>	17-156	13-199		
	<i>³⁷Cl₄-2,3,7,8-TCDD</i>	35-197	31-191		
	<i>spikes (OPR*)^e</i>				
	<i>2,3,7,8-TCDD</i>	NA ^f	67-158		
	<i>2,3,7,8-TCDF</i>	NA ^f	75-158		
	<i>1,2,3,7,8-PeCDD</i>	NA ^f	70-142		
	<i>1,2,3,7,8-PeCDF</i>	NA ^f	80-134		
	<i>2,3,4,7,8-PeCDF</i>	NA ^f	68-160		
	<i>1,2,3,4,7,8-HxCDD</i>	NA ^f	70-164		
	<i>1,2,3,6,7,8-HxCDD</i>	NA ^f	78-134		
	<i>1,2,3,7,8,9-HxCDD</i>	NA ^f	64-162		
	<i>1,2,3,4,7,8-HxCDF</i>	NA ^f	72-134		
	<i>1,2,3,6,7,8-HxCDF</i>	NA ^f	84-130		
	<i>1,2,3,7,8,9-HxCDF</i>	NA ^f	78-130		
	<i>2,3,4,6,7,8-HxCDF</i>	NA ^f	70-156		
	<i>1,2,3,4,6,7,8-HpCDD</i>	NA ^f	70-140		
	<i>1,2,3,4,6,7,8-HpCDF</i>	NA ^f	82-122		
	<i>1,2,3,4,7,8,9-HpCDF</i>	NA ^f	78-138		
	<i>OCDD</i>	NA ^f	78-144		
	<i>OCDF</i>	NA ^f	63-170		
Mercury SW846-7471A	Hg	75-125	75-125	35	90
Metals SW846-6010/6020	Ag, Al, As, Cd, Cr, Cu, Hg, Ni, Pb, Sb, Se, Zn	75-125	75-125	35	90
Radiochemical Studies Appendix D	⁷ Be and ²¹⁰ Pb	na	na	na	na
Total Solids 160.3/SM 2540B	TS	na	na	35	90
Grainsize ASTM D-422-63 Wet Sieve		na	na	35	90
Total Organic Carbon ASTM D4129-82m 415.1	TOC	75-125	80-120	35	90

* = Ongoing Precision and Recovery standard (OPR): a laboratory blank spike with known quantities of analytes. The OPR is analyzed exactly like a sample. Its purpose is to assure that the results produced by the laboratory remain within the limits specified in this method for precision and recovery.

Table A7-6. Sediment - QA/QC Sample Control Limits for the Chemical Analyses.

Analysis ^{a,b}	Percent Recovery			RPD	PARCC % Complete
	Analytes	Sample Matrix Spike	Method Blank/LCS		
Total Dissolved Solids EPA 160.1/SM 2540C	<i>TDS</i>	na	na	30	90
Total Suspended Solids 160.2/SM 2540D	<i>TSS</i>	na	na	30	90

^a = Complete method references are provided in Table B4-1.

^b = Spikes include LCS/LCSD and MS/MSD

^c = Precision and accuracy % recoveries are based off of laboratory generated statistical limits. This QAPP will be updated annually with laboratory generated statistics for each analytical method.

^d = Specifications from Table 7. EPA Method 1613B

^e = Specifications from Table 6. EPA Method 1613B

^f = Method 1613 will be running OPR and laboratory duplicate. The methodology doesn't require a MS/MSD

Table A7-7. Tissue - QA/QC Sample Control Limits for the Chemical Analyses.

Analysis ^{a,b}	Analytes	Percent Recovery		RPD	PARCC % Complete	
		Sample Matrix Spike	Method Blank/LCS			
<i>Semivolatiles SW846-8270C surrogates</i>	<i>d4-2-Chlorophenol</i>	20-108	41-114	30	90	
	<i>d4-1,2-Dichlorobenzene</i>	12-108	33-120			
	<i>2,4,6-Tribromophenol</i>	10-124	32-105			
	<i>2-Fluorophenol</i>	17-110	42-118			
	<i>d5-Phenol</i>	16-116	36-121			
	<i>d5-Nitrobenzene</i>	11-111	39-113			
	<i>2-Fluorobiphenyl</i>	10-124	37-119			
	<i>d14-p-Terphenyl</i>	13-125	36-125			
	<i>Phenol</i>	25-116	45-123			
	<i>2-Chlorophenol</i>	23-113	49-116			
	<i>1,4-Dichlorobenzene</i>	16-108	30-115			
	<i>N-nitroso-di-n-propylamine</i>	12-121	26-102			
	<i>1,2,4-Trichlorobenzene</i>	23-112	35-111			
	<i>4-Chloro-3-methylphenol</i>	24-121	31-119			
	<i>Acenaphthene</i>	15-125	37-102			
	<i>Semivolatiles SW846-8270C spikes</i>	<i>4-Nitrophenol</i>	10-141			28-111
<i>2,4-Dinitrotoluene</i>		10-139	33-118			
<i>Pentachlorophenol</i>		10-141	18-125			
<i>Pyrene</i>		10-139	29-95			
<i>d10-2-Methylnaphthalene</i>		36-122	52-104			
<i>d14-Dibenzo (a,h) anthracene</i>		18-139	40-123			
<i>Phenanthrene</i>		49-118	58-114			
<i>Benzo (k) fluoranthene</i>		38-124	50-15			
<i>Chrysene</i>		42-117	58-108			
<i>SIM surrogate</i>						
<i>SIM spikes</i>						
<i>Pesticides SW846-8081A surrogates</i>		<i>Tetrachloro-meta-xylene (TCMX)</i>	43-106	43-106	40	90
		<i>Decachlorobiphenyl</i>	46-126	46-126		
<i>Pesticides SW846-8081A spikes</i>		<i>4,4'-DDD</i>	58-154	78-143		
	<i>4,4'-DDE</i>	57-152	81-135			
	<i>4,4'-DDT</i>	52-138	75-139			
	<i>Aldrin</i>	50-146	63-137			
	<i>alpha - BHC</i>	53-130	48-129			
	<i>alpha - Chlordane</i>	51-142	75-126			
	<i>beta - BHC</i>	58-130	76-127			
	<i>delta - BHC</i>	43-134	51-124			
	<i>Dieldrin</i>	64-137	80-132			
	<i>Endosulfan I</i>	45-141	61-137			

Table A7-7. Tissue - QA/QC Sample Control Limits for the Chemical Analyses.

Analysis ^{a,b}	Analytes	Percent Recovery		RPD	PARCC % Complete
		Sample Matrix Spike	Method Blank/LCS		
	<i>Endosulfan II</i>	60-140	69-136		
	<i>Endosulfan Sulfate</i>	49-119	67-121		
	<i>Endrin</i>	47-153	75-134		
	<i>Endrin Aldehyde</i>	23-130	45-132		
	<i>Endrin Ketone</i>	66-144	82-140		
	<i>gamma - BHC</i>	56-137	55-146		
	<i>gamma - Chlordane</i>	48-143	72-129		
	<i>Heptachlor</i>	51-132	61-135		
	<i>Heptachlor Epoxide</i>	43-157	67-135		
	<i>Methoxychlor</i>	46-144	69-138		
	<i>Toxaphene</i>	70-130	70-130		
<i>PCBs SW846-8082A surrogates spikes</i>	<i>Decachlorobiphenyl</i>	40-151	40-151	40	90
	<i>Aroclor 1016</i>	31-147	57-134		
	<i>Aroclor 1260</i>	33-154	64-136		
<i>Herbicides SW846-8151 surrogates spikes</i>	<i>2,4-Dichlorophenylacetic acid</i>	14-144	14-144	40	90
	<i>2,4,5-TP</i>	42-124	24-125		
	<i>2,4-D</i>	14-156	32-147		
	<i>2,4,5-T</i>	16-156	23-137		
<i>TBT (lab specific methods) surrogates spikes</i>	<i>Tripentyl Tin</i>	NA	NA	40	90
	<i>Tripropyl Tin</i>	26-169	26-169		
	<i>Tetrabutyl Tin</i>	10-206	17-185		
	<i>Tributyl Tin</i>	10-206	17-185		
	<i>Dibutyl Tin</i>	18-179	17-185		
	<i>Butyl Tin</i>	20-153	17-185		
<i>PCB Congeners Method 1668Revision A surrogates</i>	<i>13C12 -3,3',4,4'-Tetrachlorobiphenyl BZ077</i>	25-150	30-140		
	<i>13C12 -2,3,3',4,4'-Pentachlorobiphenyl BZ105</i>	25-150	30-140		
	<i>13C12 -2,3,4,4',5-Pentachlorobiphenyl BZ114</i>	25-150	30-140		
	<i>13C12 -2,3',4,4',5-Pentachlorobiphenyl BZ118</i>	25-150	30-140		
	<i>13C12 -2,3',4,4',5'-Pentachlorobiphenyl BZ123</i>	25-150	30-140		
	<i>13C12 -3,3',4,4',5-Pentachlorobiphenyl BZ126</i>	25-150	30-140		
	<i>13C12 -2,3,3',4,4',5-Hexachlorobiphenyl BZ156</i>	25-150	30-140		
	<i>13C12 -2,3,3',4,4',5'-Hexachlorobiphenyl BZ157</i>	25-150	30-140		

Table A7-7. Tissue - QA/QC Sample Control Limits for the Chemical Analyses.

Analysis ^{a,b}	Analytes	Percent Recovery		RPD	PARCC % Complete	
		Sample Matrix Spike	Method Blank/LCS			
<i>PCB Congeners Method 1668 Revision A spikes</i>	<i>13C12 -2,3',4,4',5,5'-Hexachlorobiphenyl BZ167</i>	25-150	30-140			
	<i>13C12 -3,3',4,4',5,5'-Hexachlorobiphenyl BZ169</i>	25-150	30-140			
	<i>13C12 -2,3,3',4,4',5,5'-Heptachlorobiphenyl BZ189</i>	25-150	30-140			
	<i>3,3',4,4'-Tetrachlorobiphenyl BZ077</i>	NA	50-150			
	<i>2,3,3',4,4'-Pentachlorobiphenyl BZ105</i>	NA	50-150			
	<i>2,3,4,4',5-Pentachlorobiphenyl BZ114</i>	NA	50-150			
	<i>2,3',4,4',5-Pentachlorobiphenyl BZ118</i>	NA	50-150			
	<i>2,3',4,4',5'-Pentachlorobiphenyl BZ123</i>	NA	50-150			
	<i>3,3',4,4',5-Pentachlorobiphenyl BZ126</i>	NA	50-150			
	<i>2,3,3',4,4',5-Hexachlorobiphenyl BZ156</i>	NA	50-150			
	<i>2,3,3',4,4',5'-Hexachlorobiphenyl BZ157</i>	NA	50-150			
	<i>2,3',4,4',5'-Hexachlorobiphenyl BZ167</i>	NA	50-150			
	<i>3,3',4,4',5'-Hexachlorobiphenyl BZ169</i>	NA	50-150			
	<i>2,2',3,3',4,4',5-Heptachlorobiphenyl BZ170</i>	NA	50-150			
	<i>2,2',3,4,4',5,5'-Heptachlorobiphenyl BZ180</i>	NA	50-150			
	<i>2,3,3',4,4',5,5'-Heptachlorobiphenyl BZ189</i>	NA	50-150			
	<i>PCDDs/PCDFs EPA 1613 surrogates ^c</i>	<i>¹³C₁₂-2,3,7,8-TCDD</i>	25-164	20-175	NA	90
		<i>¹³C₁₂-2,3,7,8-TCDF</i>	24-169	22-152		
<i>¹³C₁₂-1,2,3,7,8-PeCDD</i>		25-181	21-227			
<i>¹³C₁₂-1,2,3,7,8-PeCDF</i>		24-185	21-192			
<i>¹³C₁₂-2,3,4,7,8-PeCDF</i>		21-178	13-328			
<i>¹³C₁₂-1,2,3,4,7,8-HxCDD</i>		32-141	21-193			
<i>¹³C₁₂-1,2,3,6,7,8-HxCDD</i>		28-130	25-163			
<i>¹³C₁₂-1,2,3,4,7,8-HxCDF</i>		26-152	19-202			
<i>¹³C₁₂-1,2,3,6,7,8-HxCDF</i>		26-123	21-159			
<i>¹³C₁₂-1,2,3,7,8,9-HxCDF</i>		29-147	17-205			
<i>¹³C₁₂-2,3,4,6,7,8,-HxCDF</i>		28-136	22-176			
<i>¹³C₁₂-1,2,3,4,6,7,8-HpCDD</i>		23-140	26-166			
<i>¹³C₁₂-1,2,3,4,6,7,8-HpCDF</i>		28-143	21-158			
<i>¹³C₁₂-1,2,3,4,7,8,9-HpCDF</i>		26-138	20-186			
<i>¹³C₁₂-OCDD</i>		17-156	13-199			
<i>PCDDs/PCDFs EPA 1613 spikes (OPR*) ^f</i>	<i>³⁷Cl₄ -2,3,7,8-TCDD</i>	35-197	31-191			
	<i>2,3,7,8-TCDD</i>	Na ^h	67-158			
	<i>2,3,7,8-TCDF</i>	Na ^h	75-158			

* = Ongoing Precision and Recovery standard (OPR): a

Table A7-7. Tissue - QA/QC Sample Control Limits for the Chemical Analyses.

Analysis ^{a,b}	Analytes	Percent Recovery		RPD	PARCC % Complete
		Sample Matrix Spike	Method Blank/LCS		
<i>laboratory blank spike with known quantities of analytes. The OPR is analyzed exactly like a sample. Its purpose is to assure that the results produced by the laboratory remain within the limits specified in this method for precision and recovery.</i>	<i>1,2,3,7,8-PeCDD</i>	Na ^h	70-142		
	<i>1,2,3,7,8-PeCDF</i>	Na ^h	80-134		
	<i>2,3,4,7,8-PeCDF</i>	Na ^h	68-160		
	<i>1,2,3,4,7,8-HxCDD</i>	Na ^h	70-164		
	<i>1,2,3,6,7,8-HxCDD</i>	Na ^h	78-134		
	<i>1,2,3,7,8,9-HxCDD</i>	Na ^h	64-162		
	<i>1,2,3,4,7,8-HxCDF</i>	Na ^h	72-134		
	<i>1,2,3,6,7,8-HxCDF</i>	Na ^h	84-130		
	<i>1,2,3,7,8,9-HxCDF</i>	Na ^h	78-130		
	<i>2,3,4,6,7,8-HxCDF</i>	Na ^h	70-156		
	<i>1,2,3,4,6,7,8-HpCDD</i>	Na ^h	70-140		
	<i>1,2,3,4,6,7,8-HpCDF</i>	Na ^h	82-122		
	<i>1,2,3,4,7,8,9-HpCDF</i>	Na ^h	78-138		
	<i>OCDD</i>	Na ^h	78-144		
	<i>OCDF</i>	Na ^h	63-170		
Mercury SW846-7471A			60-130	60-130	35 90
Metals SW846-6010/6020 & EPA200.8	<i>Ag, Al, As, Cd, Cr^g, Cu, Hg, Ni, Pb, Sb, Se, Zn</i>		70-130	70-130	35 90
Lipids	<i>See QAPP for guidance</i>	na	na	30	90

^a = Complete method references are provided in Table B4-1.

^b = Spikes include LCS/LCSD and MS/MSD

^c = Precision and accuracy % recoveries are based off of laboratory generated statistical limits. This QAPP will be updated annually with laboratory generated statistics for each analytical method.

^d = Specifications from Table 7. EPA Method 1613B

^e = Specifications from Table 6. EPA Method 1613B

^g = %recovery Cr = 75-125

^h = Method 1613 will be running OPR and laboratory duplicate. The methodology doesn't require a MS/MSD

Table B2-1. Sediment and Tissue - Sample Containers, Preservation, Holding Times, and Sample Volume¹.

	Container		Sample Size		Tissue	Preservation	Holding Time ²
	Type	Jar	Sediment	Jar			
Grain size	G/p	2 x 16 oz	100 g	NA	NA	4±2°C	6 months
VOAs	WMG	2 oz/septa	5 g	NA	NA	4±2°C	14 days
Hg		2 oz	50 g - 100 g	2 oz	10 g		6 months ³
Total organic carbon		4 oz	20g	NA	NA		6 months
Metals and Total Solids		4 oz	50 g - 100 g	4 oz	10g		1 year
Tributyltin		4 oz	20-250g	8 oz	20g		6 months
SVOCs				8 oz	20g		6 months
PCBs	WMG	32 oz	20-250g	8 oz	20 - 75g	Deep Frozen (-20±4°C)	1 year
Pesticides				8 oz	20g		1 year
Herbicides				NA	NA		1 year
PCDD/PCDFs		16 oz	50 g	8 oz	75g		1 year
PCB congener		16 oz	10 g	8 oz	75g		1 year
Archival		8 oz	NA	2 x 8 oz	NA		1 year

WMG = Wide Mouth Glass

HDPE = High Density Polyethylene

AG = Amber Glass

G/p = Glass or Plastic

¹ = All samples will need a minimum of 5% QA. Collection of 3x normal amount will be necessary.

² = Samples must either be analyzed or frozen within 24 hrs. Frozen tissue and sediment may be held up to the stated limits before analysis per PSEP Guidelines.

³ = EPA Guidance on Fish Sampling and Analysis

Table B4-1. Analysis Methods.

	Sediment	Tissue	Analysis Method
Volatile Organics (VOC)	X		SW846-8260B Volatile Organic Compounds By Gas Chromatography/Mass Spectrometry (GC/MS)
Semivolatiles (SVOC) Full Scan	X	X	SW846-8270C Semivolatile Organic Compounds By Gas Chromatography/Mass Spectrometry(GC/MS)
Semivolatiles (SVOC-SIMs)	X	X	SW846-8270C-SIMs Semivolatile Organic Compounds By Gas Chromatography/Mass Spectrometry(GC/MS)
PCBs aroclors	X	X	SW846-8082 Polychlorinated Biphenyls (PCBs) By Gas Chromatography
PCBs congner	X	X	EPA-1668A: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/HRMS
Pesticides	X	X	SW846-8081A Organochlorine Pesticides By Gas Chromatography
Herbacides	X	X	SW846-8151A Chlorinated Herbicides By GC Using Methylation Or Pentafluorobenzylation Derivatization
PCDDs/PCDFs	X	X	EPA-1613B Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS
TBT	X	X	Done on bulk sediment. Methodology is laboraroty/matrix specific.
Mercury	X	X	METHOD 7471A Mercury in solid and semisold wate (Manual Cold-Vapor Technique)
Metals	X	X	WS846-6010B & 6020 Inductively Coupled Plasma-Atomic Emission Spectrometry
Total Organic Carbon	X		Plumb, 1981
Total Solids	X		EPA-160.3/SM 2540B
Lipids		X	See QAPP for guidance
Grain Size ^a	X		PSEP

^a = Grain size analysis will be accomplished using Puget Sound Estuary Program (PSEP) 1986 Tt.

Table B5-1. Quality Control Samples Collection Summary.

Sample Type	Frequency
Temperature Blanks	1 per cooler
Blind Field Duplicates	10 percent
Blind Field Replicates	10 percent
Field Equipment Rinsate Blanks	5 percent
Field Trip Blanks (VOC analysis only)	1 per cooler
Field Triplicates Tissue Refer to section 3.1.5 Field Triplicates - Tissue Only for explanation.	1 per station
Matrix Spike	5 percent
Matrix Spikes Duplicates (organic analysis only)	5 percent
Laboratory Control Spike	5 percent
Laboratory Control Spike Duplicates (organic analysis only)	5 percent
Laboratory Duplicate (inorganic)	5 percent

Table C1-1. Parameters Used to Evaluate Data Quality.

Data Quality Indicator	QA Parameter
Precision	Field Duplicate Sample Laboratory Duplicate QA Laboratory Duplicate Matrix Spike Duplicates
Accuracy	Matrix Spike Surrogate Spike Performance Evaluation Sample Initial Calibration Standards and Blanks Continuing Calibration Standards and Blanks Laboratory Control Samples Trip Blank Rinse Blank Field Blank Method Blank
Representativeness	Rinse Blank Field Blank Method Blank Chain of Custody Holding Times and Preservation Status
Comparability	Method Detection Limits Method Reporting Limits Sample Collection Methods Laboratory Analytical Methods
Completeness	Data Qualifiers Laboratory Deliverables Requested/Reported Valid Results

APPENDIX A

Columbia Analytical Services QA Manual

Previously provided on CD-ROM.

APPENDIX B

Analytical Resources Inc. QA Manual

Previously provided on CD-ROM.

APPENDIX C

AXYS Analytical Services Ltd. QA Manual

Previously provided on CD-ROM.

APPENDIX D

**Benthic Community Quality Assurance Project Plan
and
EcoAnalysts, Inc Quality Assurance Plan**

1.0 BENTHIC INVERTEBRATE ANALYSIS

This quality assurance plan describes the laboratory methods and quality assurance procedures for taxonomic identification and analysis of benthic invertebrate samples for Round 1 of the Portland Harbor Remedial Investigation. Approximately 13 intertidal sediment samples will be collected for benthic invertebrate identification and enumeration. These data will support the preliminary ecological risk assessment. Detailed descriptions of the objectives, station locations, methods, and field procedures are contained in the Field Sampling Plan (SEA et al. 2002).

1.1 ORGANIZATION

The overall project organization is described in Section 1.1 of the QAPP.

EcoAnalysts, Inc., Moscow, Idaho, will perform the laboratory services for benthic infauna taxonomy and sorting. The primary focus of EcoAnalysts is in the area of aquatic biology with an emphasis on benthic macroinvertebrates. Since their inception in 1994, EcoAnalysts has completed more than 200 projects from around the country and processed over 15,00 macroinvertebrate samples. Their project experience includes environmental monitoring programs and watershed assessments for EPA, USGS, and the State of Idaho. Gary Lester will be the EcoAnalysts project manager.

Pam Sparks (SEA) will be the quality assurance manager for benthic invertebrate analysis. She will perform laboratory oversight for the benthic laboratory and will direct the review and analysis of the benthic data.

1.2 SAMPLE HANDLING, DOCUMENTATION AND CUSTODY

The sample will be preserved in the field with 95% ethanol (for a final concentration of 70 – 80% ethanol) and transferred to the laboratory in a sealed plastic jar. Each jar will have two external labels and one internal label. The internal label will be written in pencil or indelible ink in waterproof 100 percent rag paper (e.g., Rite in the Rain paper). The external labels will be printed using an indelible ink pen. One label will be attached to the side of the jar and the second to the lid of the jar.

Procedures for sample documentation and custody will be the same as for chemical samples (QAPP Section 2.6).

1.3 LABORATORY PROCEDURES

The methods used by EcoAnalysts are consistent with EPA protocols for analysis of freshwater invertebrates (Babour et al. 1999). The details of their procedures are described below.

1.3.1 Sample Sorting: Grab Samples

Samples will be sorted to remove benthic macroinvertebrates from debris in the sample prior to taxonomic identification. For Round 1, the samples will be sorted in their entirety so there is no fixed target count of invertebrates to be removed. The sorting procedures to be used by the laboratory will be as follows:

1. The sample will be emptied into a 500 micron mesh screen to remove preservative and then washed into a shallow pan of water where larger pieces of debris will be rinsed, inspected for attached invertebrates, and discarded.
2. The sample will be agitated with water to separate organic matter from inorganic sediments, and the lighter organic material will be poured back into the 500 micron mesh sieve.
3. The inorganic portion of the sample remaining in the pan will be repeatedly washed and decanted into the sieve until no organic matter remains.
4. Once all organic material has been removed from the sample, the remaining inorganic sediments will be inspected under a dissecting microscope for any invertebrates too heavy to have floated off (e.g. mollusks, snails, stone-cased Trichoptera, etc.). The inorganic fraction of the sample will be discarded once it is confirmed that all invertebrates have been removed.
5. The organic material that is retained in the sieve will be evenly distributed in a Caton subsampler (i.e. a gridded tray consisting of 30 squares, each square being 6cm per side), a square will be randomly selected and its contents transferred to a petri dish.
6. The material in the petri dish will be sorted under a dissecting microscope (minimum magnification = 6X) and invertebrates placed into three vials - Oligocheata, Chironomidae, and other – and filled with 70% ethanol.
7. The entire sample will be sorted, square by square, until completion.
8. When sorting is complete the appropriate sample tracking number will be entered on a printed label and inserted into the vials.
9. The sorting bench sheet (see attachment) will be completed.

The sorted debris residue will be saved in a separate container. The sorted residue is preserved in 95% ethanol. Length of storage and archival is determined by the laboratory or benthic section supervisor.

1.3.2 Identification of Organisms

Sorted organisms will be identified and enumerated to the lowest taxonomic level possible, generally the species level. Chironomids will be identified to genus or the lowest practical taxonomic level. Annelids (particularly Oligochaetes) will be

identified to family or the lowest practical taxonomic level. Only those taxonomic keys that have been peer-reviewed and are available to other taxonomists will be used.

Each taxonomist will record identifications on pre-printed and coded forms. These forms will allow easier entry of data into computers for analysis. The taxonomist will initial the form on completion of the sample.

1.3.2.1 Non-oligochaete Identification

Identification of non-oligochaete macroinvertebrates will proceed as follows:

1. A taxonomist will select a sample for identification.
2. The vial containing non-Chironomidae and non-Oligochaeta ("other" vial) will be emptied into a petri dish.
3. Under a dissecting microscope the invertebrates will be sorted to the lowest practical level.
4. The number of individuals of each taxon will be counted and entered directly into the laboratory's macroinvertebrate data entry program.
5. At least one specimen of each taxon encountered will be placed into a 1-dram vial containing 70% ethanol and labeled with identity and sample number. These specimens will comprise the project synoptic reference collection. Labels with specific taxa names (and the taxonomist's initials) are added to the vials of reference specimens by the taxonomist. (Note that individual specimens may be extracted from the sample to be included in a reference collection or to be verified by a second taxonomist.)
6. Once the sample is completed the organisms will be placed into the original vial and the computer automatically prints out a taxonomic bench sheet for the sample.
7. The Chironomidae taxonomist will then process the corresponding sample vial containing Chironomidae at his workstation using the same methods as for the "other" vial.
8. The archived and reference specimen vials, (grouped by station and date), will be placed in jars with a small amount of denatured 70% ethanol and tightly capped. The ethanol level in these jars will be examined periodically and replenished as needed, before ethanol loss from the specimen vials takes place. A stick-on label is placed on the outside of the jar indicating sample identifier, date, and preservative (denatured 70% ethanol).

1.3.2.2 Oligochaete Identification

The following steps will be used to identify aquatic Oligochaeta to the lowest possible level that is usually genus or species, with the exception of the family Enchytraeidae.

1. A vial of oligochaetes will be selected for slide mounting.
2. A log sheet will be filled out with pertinent information for each sample including project number, sample number, and slide number.

3. The vial will be emptied into a petri dish.
4. Under a fume hood, a slide will be labeled with the unique sample number and slide number (example: Slide 1 of 7) and a few drops of mounting CMC-10 media will be placed onto the left and right halves of the slide.
5. Using fine-tipped jeweler's forceps individuals will be removed from the petri dish and lined up in a head-down position in the mounting media. The number of organisms under each cover slip will vary according to their size.
6. A cover slip will be gently put over the slide and any air bubbles will be teased out by applying gentle pressure.
7. When both cover slips are in place the slide will be placed on a slide drying tray over low heat to dry.
8. The number of individuals on the slide will be recorded in the log book.
9. Once all slides for the sample are made the numbering sequence on each will be completed (example: Slide 1 of 7).
10. After all samples are mounted and dried (at least 2 days drying time required) the slides will be removed from the drying tray and placed in slide boxes.
11. Slides will be initialed by the identifying taxonomist. A separate label may be added to slides to include the taxon (taxa) name(s) for use in a voucher or reference collection.

1.4 QUALITY CONTROL REQUIREMENTS

The following quality control procedures will be used by the laboratory during Round 1.

1.4.1 Sample sorting

To ensure that every sample meets a standard organism removal rate of 90% the following steps will be followed:

1. A previously sorted sample will be selected by a second laboratory technician (this cannot be the same person who originally sorted the sample).
2. The second laboratory technician will take the sample and redistribute it into the Caton sorting tray.
3. The second technician will remove randomly selected squares and resort them until 20% of the material is checked (6 of 30 squares). The technician then will calculate an estimated percent efficacy by using the following equations:

Estimating the number of organisms missed:

$$E = (A/B)C$$

where:

E = estimated total number of organisms missed by sorter

A = the number of organisms found in the 20% resort

B = the number of grids resorted (usually 6)

C = the total number of grids in the Caton tray (usually 30)

Estimating the actual total count:

$$T = X + E$$

where:

T = the estimated total number of bugs in the sorted portion of the original sample

X = the number of bugs picked by the first sorter

E = the estimated number of bugs missed

Estimating the percent sorting efficiency:

$$S = (X/T)100\%$$

where:

S = the estimated percent sorting efficiency

X = number of bugs picked by the first sorter

T = the estimated total number of bugs

If the estimated percent sorting efficiency is 90% or greater the sample passes the quality assurance check. However, if the estimate is less than 90%, the sample is completely resorted. If this happens, the sample undergoes the quality assurance process again until it passes the 90% efficiency level.

4. All macroinvertebrates found in the QC check and in re-sorts will be added to the sample vials.
5. Once all samples have been checked, a quality assurance report will be generated and provided to the client at the time of data delivery (e.g., a sorting quality assurance form, Attachment BIO-QAPP).

Complete records on sorting and resorting for each sample will be permanently maintained.

1.4.2 Identification of Organisms

The consistency of identifications among taxonomists and sampling programs is crucial to maintaining a good database. Internal consistency within a laboratory will be maintained by the constant informal interaction among taxonomists. Internal quality control will be maintained by checking identifications against a verified voucher collection.

The following steps will be taken to help ensure the accuracy of the taxonomy:

1. First, a second taxonomist (reviewer not responsible for the original identifications) will examine the synoptic reference collection to verify the accuracy of all taxa identified in the project.
2. Second, 10% of all specimen vials will be randomly selected for re-identification by the reviewer. The last name of the person validating the identification should be added to the vial label.
3. Specimens sent out for taxonomic validations will be recorded in a "Taxonomy Validation Notebook" showing the label information and the date sent. Upon return of the specimens, the date received, the finding, and the

name of the person who performed the validation are also recorded in the notebook.

4. A percent similarity will be calculated for both sets of data.
5. Discrepancies will be discussed and/or re-examined by both taxonomists.
6. The final data will be adjusted according to the recommendations of both taxonomists.
7. A bibliography of the basic taxonomic literature used in aiding identification of specimens will be provided to the reviewer upon request.

Complete records on identification of each sample will be permanently maintained.

1.5 DATA VALIDATION

Benthic infauna validation and verification methods will include a review by the benthic infauna QA officer of the following documents:

- Sample sorting quality control report
- Taxonomic quality control report
- Verification report on the specimen voucher collection.

Reports will be reviewed to ensure that all QC requirements have been met and that completeness is acceptable.

1.6 ARCHIVAL PROCEDURE

The following procedures will be followed during Round 1.

Upon completion of sorting, the sorted debris residue will be saved in a separate container. The sorted residue will be preserved in 95% ethanol, placed in jars and tightly sealed.

1.6.1 Sorted Debris

Upon completion of all QC procedures, the sorted debris residue will be saved in a separate container. The sorted residue will be preserved in 95% ethanol, placed in jars and tightly sealed.

1.6.2 Identified Samples

Upon completion of sample identification and QC, the archived vials containing the major taxonomic groups and reference specimen vials, (grouped by station and date), will be placed in jars with a small amount of 70% ethanol and tightly capped. The ethanol level in these jars will be examined periodically and replenished as needed, before ethanol loss from the specimen vials takes place. A label is placed on the outside of the jar indicating sample identifier, date, and preservative.

1.6.3 Maintenance of a Verified Reference Collection

A verified reference collection of the organisms found during the sampling program will be created. The collection will consist of from one to five individuals of each species. A record of each species name, the taxonomist who made the identification, and the name of the taxonomist who verified the identification will be recorded. The record will also show when the specimen was verified, the location of the specimen in the reference collection, the status of the specimen if it was loaned to outside experts, and references to pertinent literature.

1.7 PERFORMANCE AUDITS

Periodic performance audits will be conducted by the Benthic QA Officer to ensure that the QC objectives are being met by the benthic infauna laboratory. These audits will include the resorting and re-identification of specimens. If the audits identify unacceptable laboratory practices, then corrective actions will be implemented.

1.8 CORRECTIVE ACTION FOR UNACCEPTABLE DATA

The Benthic QA Officer will be responsible for developing and initiating corrective action if the performance audits result in identification of unacceptable sample handling procedures or data. Corrective action reports (see Forms) will be used to document non-conformances and subsequent corrective actions. The RI/FS Project Coordinator will be immediately notified if the problem is of significant magnitude to affect program success. Corrective actions may include the following:

- Additional resorting of samples
- Additional re-identification of samples.

In the event that re-sampling is considered, EPA will be consulted prior to initiating re-sampling.

1.9 DATA DELIVERABLES

The following data will be reported by the benthic laboratory:

- Data forms listing the abundance of all taxa by sample
- Sorting quality control data sheets
- Results from the taxonomic quality control
- Electronic data files listing the abundance of all taxa by sample
- Any problems that may have influenced data quality.

Identification Similarity Report

Identification Quality Check
note changes to original data only
(Project)-(Sample)-QC

	Original (Mike)	QC (John)
Acari (=Acarina)	29	28
Amiocentrus aspilus	16	16
Antocha sp.	10	10
Baetis tricaudatus	5	5
Brachycentrus occidentalis	57	58
Brachycentrus sp.	2	2
Cultus sp.	0	1
Drunella grandis	0	2
Drunella sp.	2	0
Epeorus albertae	1	1
Glossosoma sp.	15	15
Glossosomatidae	1	1
Hexatoma sp.	1	1
Hydropsyche sp.	3	3
Hydropsychidae	1	1
Lepidostoma sp.	20	21
Nematoda	3	4
Optioservus sp.	10	10
Perlidae	1	1
Perlinodes aurea	2	2
Perlodidae	1	0
Psychomyia sp.	1	0
Psychomyiidae	0	1
Rhithrogena sp.	7	7
Sweltsa sp.	6	6
Zaitzevia sp.	19	19
TOTAL:	213	215

Percent Similarity: 97.09

EcoAnalysts, Inc.

Attachments: Biological Laboratory Form

Sorting QA/QC Form

Sorting Efficacy/Label Quality, Project:

A	B	C	D	E	F	G	H	I	J	K	L		
Sample	OS by	Original Count	QC by	20% QC Count	QC'd Grids	Total Grids	Est. Total Missed	Est. Total count	Est. % Effective	Remainder count	Remainder count + 20% QC count	Overall Total Count	Actual % Effective
Example		510		10	2	16	$(C/D)*E=80$	$B + F = 590$	$(B/G)*100 = 86$	70	$I + C = 80$	$B + J = 590$	$(B/K)*100 = 86$
		590		1	2	16	$(C/D)*E = 8$	$B + F = 598$	$(B/G)*100 = 99$				
Labels: Good					Reject Taxa Present: 2 worm parts, one terrestrial beetle					Comments:			
Labels:					Reject Taxa Present:					Comments:			
Labels:					Reject Taxa Present:					Comments:			
Labels:					Reject Taxa Present:					Comments:			
Labels:					Reject Taxa Present:					Comments:			
Labels:					Reject Taxa Present:					Comments:			

ECOANALYSTS, INC.

LABORATORY QUALITY ASSURANCE PLAN



May 1, 2001
EcoAnalysts, Inc.
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Introduction

EcoAnalysts is dedicated to providing the most accurate and reliable data as possible. To that end we implement quality assurance procedures at every step of the process.

Sample Receiving and Chain of Custody

Upon delivery to EcoAnalysts all sample containers are inventoried and reconciled with any accompanying Chain of Custody (COC). We evaluate the condition of the samples and if any containers are damaged or otherwise in poor shape, they are dealt with appropriately. We record the condition of each sample container and any corrective measures taken. If we do not receive a COC form from the client, we provide the client with our internal inventory for approval. Once all samples are present and/or accounted for, we assign each sample a unique internal identification number and enter all information associated with that sample into our laboratory information management system (LIMS). The sample is referred to and identified with the internal lab number from that point forward. Each jar is tagged with a permanent label that contains the project name, identification number and an indication of the number of jars associated with that sample (i.e. 1 of 4, 2 of 4, etc.). The sample containers are stored in a secure area until they are processed.

Sorting and Subsampling

Sorting in the context of our laboratory is the removal of macroinvertebrates from the background material (e.g. sand, coarse organic matter, fine sediment) found in a sample. Sorting may involve applying a subsampling (removal of a quantified portion of the whole sample) procedure. Our policy is to ensure the sorting process removes at least 90% of the specimens available in the sample. To that end, every sample is quantitatively checked to assess the sorting efficacy according to the following protocol:

When the original technician finishes sorting, the he or she returns the *unsorted* sample matrix to the original sample jar, and places the *sorted* portion in a separate sample jar. The jars are labeled appropriately and both containers are returned to the location specified. As soon as possible, another technician checks the sample.

The quality control technician (QC tech) checks all aspects of the sorting process. The QC tech recounts the removed invertebrates to ensure enough specimens were removed. Concurrently the technician assures the project rejection protocol was correctly observed. The quality control technician also checks all labels and records for clarity and accuracy. To quantitatively check the sorting efficacy, the QC technician redistributes the *sorted* portion in an appropriately sized sorting receptacle. They then randomly remove and inspect 20% of the material. If any invertebrates are found, the QC tech uses simple formulae to estimate the efficacy of the original sort. If the estimate is 90% or greater the sample passes. If the estimate is less than 90 percent, the sample fails QC and must be completely resorted. If this occurs, the QC tech returns the sample to the original sorter. The QC sorter discusses what types of invertebrates were found and where they were found (e.g. moss, inside trichoptera cases, etc.) with the original sorter. The original technician then resorts the sorted portion of the sample. The sample undergoes the QC process again until it passes. All relevant QC and resort information is recorded.

There is one special case in which further investigation is warranted. If the actual percent efficacy (obtained after a full resort) is more than 10 percentage points away from the estimated efficacy, the QC tech, original sorter and a supervisor need discuss why that occurred and implement systematic corrective action if necessary.

Taxonomic Identifications

EcoAnalysts, Inc. uses two methods to ensure taxonomic accuracy and consistency. As a comprehensive, qualitative QC we retain a synoptic voucher collection consisting of at least one good specimen (preferably 3-5 specimens) of each taxon encountered within a project. If multiple taxonomists are involved, they each maintain their own collections. These collections are reviewed by all project taxonomists and a quality control taxonomist every two weeks or at the end of the project, whichever is shorter. In the event none of our taxonomists can identify a particular specimen, we send the specimen to an outside specialist for verification.

A quantitative QC is obtained by re-identifying 10% of the samples in a project. The individual QC samples are randomly chosen *a priori* but the original taxonomist is unaware which samples will be checked. As soon as possible after a QC sample is initially processed, a second taxonomist re-identifies it. The resulting taxa lists are compared using a similarity index. Typically, at genus/species level, experienced taxonomists using a common standard taxonomic effort can attain uncorrected similarities of about 90%. The two taxonomists discuss any differences and determine how best to reconcile those differences. Errors (if any) in identification, enumeration, and data entry are discussed and corrected. If it is determined misidentifications have been pervasive, the original taxonomist must revisit any sample with the taxon in question and ascertain the accuracy of the determination. If enumeration is determined to be an important source of error, the original taxonomist must revisit the samples and attempt a more accurate count.

Exact similarity (100%) is virtually unattainable due to subtle and complex factors (e.g. destructive methods that must be applied to identify some taxa, removal of type specimens for the reference collecting, etc) and a full discussion would go beyond the scope of this document.

Data Entry and Management

Data entry is integrated into the identification process. The identifying taxonomist enters data at his or her workstation using a specially designed macroinvertebrate data entry program. The program has several safeguards built into it which makes it virtually impossible to make many of the most common data entry mistakes (duplicates, typos etc.) By employing this method, we eliminate a secondary step of data entry and we use the person most qualified to recognize errors, the taxonomist, to enter the data. Data are stored using a relational database that is backed up daily. In addition to the above steps, upon completion of the project, the project taxonomist reviews a synoptic list to ensure no taxa have been accidentally introduced.

Sample Return

Unless instructed otherwise, when all processing is complete and data have been delivered and accepted, all sample material is returned to the client. If a COC was provided by the client, it is properly annotated and returned with the samples.

APPENDIX E

Project DQOs

Effects of Sediment Movement on Nature and Extent of Chemical Concentration and Risk.

<i>DQO Step</i>	Output
1. State the Problem	The spatial and temporal scales of sediment transport are not well understood. Sediment transport may affect chemical distribution and ecological and human health exposure. Sediment transport processes / hydrodynamics may affect selection of remedial alternatives.
2. Identify the Decision to be Made	<ul style="list-style-type: none"> • Is sediment transport sufficiently variable that we cannot predict surface chemistry distributions or risks in the ISA? • Do physical processes expose previously buried contaminated sediment? • Do physical processes result in burial of contaminated sediment?
3. Identify the Inputs to the Decision	<ul style="list-style-type: none"> • Time series bathymetric surveys (high and low flow conditions) <ul style="list-style-type: none"> • Sediment stakes in beach areas where bathymetry cannot be measured. • Time series sediment chemistry • Hydrodynamic/Sediment transport model <ul style="list-style-type: none"> • Inputs may include: <ul style="list-style-type: none"> • Current velocity • Grain size • Suspended solids <ul style="list-style-type: none"> • USGS historical data? • Bathymetry • River stage • Other (TBD based on model selected) • Model must document uncertainties and identify which parameters most strongly affect the outcome of the model.
4. Define the Boundaries	<ul style="list-style-type: none"> • Ross Island to confluence with Columbia River • Calibrate within the ISA • Needs to span both high and low flow conditions • Hydrodynamic portion of model needs to predict 500 year (?) event

Effects of Sediment Movement on Nature and Extent of Chemical Concentration and Risk.

<i>DQO Step</i>	Output
5. Develop a Decision Rule	<ul style="list-style-type: none">• If bed elevation losses over time are greater than the surface sampling depth interval, then evaluate subsurface sediment quality• If bed elevation changes are greater than the surface sampling depth interval, then evaluate surface sediment to determine quality of depositing sediment• If bed elevation gains are greater than the surface sampling interval, and area has unacceptable risks, evaluate area for potential for natural attenuation as a remedial alternative.
6. Specify Tolerable Limits on Decision Error	<ul style="list-style-type: none">• Error rate in measurements can't be greater than the depth over which you need to make a decision<ul style="list-style-type: none">• Need ± 6 inches on bathymetric measurements.• Model must be sufficient to provide a reasonable confidence that the spatial range of possible conditions has been sampled.
7. Optimize the Design	<ul style="list-style-type: none">• Select and set up hydrodynamic model using existing data during Round 1• Collect data to calibrate model in Round 2.• Collect bathymetry in the ISA along the shoreline using multibeam acoustic bathymetry.• Set out and monitor sediment stakes in beach areas• Collect hydrodynamic data during unique flow conditions• Mimic sampling approach/methods of previous sediment chemistry effort for time series data

Nature and Extent of Chemical Concentrations in the lower Willamette River.

DQO Step	Output
1. State the Problem	Historical data show that chemical concentrations are present in sediments in the lower Willamette River. Distribution of sediment chemistry is not well documented in all areas. Surface sediments may act as a source to other areas of the river. Based on known physical transport processes, the stability of the sediment chemical concentration distribution is uncertain. Information on concentrations of chemicals in the water column is limited.
2. Identify the Decision to be Made	<ul style="list-style-type: none"> • Do potential COC concentrations exceed background and/or relevant upgradient concentrations? • What is the nature and extent of chemical concentrations in surface sediment and water? • Do chemical concentrations in surface sediment and water result in unacceptable risk to human or ecological receptors? • Do chemical concentrations representing a risk extend beyond the ISA? • Is the distribution of sediments chemistry spatially and temporally consistent? How representative are existing sediment chemistry data of current conditions?
3. Identify the Inputs to the Decision	<ul style="list-style-type: none"> • Distribution of COCs in surface sediment and the water column • Temporal variability in sediment concentrations at historical (fixed) locations or habitat zones • Risk-based sediment criteria developed from risk assessments
4. Define the Boundaries	<ul style="list-style-type: none"> • Round 1 within the ISA ± 1.5 mile • Surface sediment and the water column • For sediment, bank areas to bottom of channel to coincide with risk assessment exposure areas
5. Develop a Decision Rule	<ul style="list-style-type: none"> • Do concentrations exceed a risk-based threshold? (May vary based on what drives risk for any given reach or area of the river.) • Are times series chemistry data within the same order of magnitude as historical concentrations and do they trigger the same sediment management decision?

Nature and Extent of Chemical Concentrations in the lower Willamette River.

DQO Step	Output
6. Specify Tolerable Limits on Decision Error	<ul style="list-style-type: none">• Sampling density within a habitat type or other strata is sufficient to estimate spatial variability.• Sampling density for time series sediment analysis is sufficient to estimate effects of navigational accuracy, analytical variability, and spatial heterogeneity.
7. Optimize the Design	<p data-bbox="762 553 863 581">Sediment</p> <ul style="list-style-type: none">• Stratify sampling area by SPI benthic zone for sediment sampling• Assume historical Category 1 data have characterized sediment quality adequately• Conduct an evaluation of temporal stability in sediment concentrations by resampling some historical stations, mimic original sediment sampling approach• Sample surface sediment within biologically active zone in areas where there are no historical Category 1 data <p data-bbox="667 776 737 803"><i>Water</i></p> <ul style="list-style-type: none">• Collect water samples throughout water column (integrate over depth)• Collect samples along transects from shore to shore in the ISA• Collect samples at either end of the ISA, and below the falls (i.e., measure inputs and outputs from ISA).

Groundwater.

DQO Step	Output
1. State the problem	The risk to ecological and human receptors from exposure to certain chemicals in groundwater discharging to the river may not be determined through sediment sampling. The chemical class of interest that is not addressed through sediment sampling is volatile organic compounds (VOCs).
2. Identify the decision	Determine whether exposure to hazardous substances in the ISA groundwater pose an unacceptable risk to fish species and invertebrate communities.
3. Identify inputs to the decision	<p>Use existing DEQ upland groundwater information and other existing information to describe hydrogeology/chemistry of ISA (Round 1).</p> <p>Identify groundwater sampling and groundwater plume identification techniques (paper evaluation) in Round 1.</p> <p>Identify sensitive receptors and plume locations (Round 1).</p> <p>Use existing information to determine data gaps (no information to assess potential for plume).</p> <p>Use physical and chemical data to determine if chemical will preferentially partition onto sediment.</p> <p>Sample test locations with “plumes” of chemicals with low partitioning coefficients to demonstrate effectiveness of techniques (Round 1).</p> <p>Sample at point of exposure (Round 2).</p> <p>AWQC and no observed effects concentrations(NOECs) from literature studies will be used to assess potential for risk.</p>
4. Define the boundaries to the study	<p>The ISA will be the geographic boundary to the study area.</p> <p>Fish/invertebrates will be evaluated using a range of risk estimates (NOECs and LOECs).</p> <p>Exposure media may be collected in Round 2 for use in decision-making in the fall, 2003.</p>
5. Develop a decision rule	If the groundwater/surface water seep chemical concentration is greater than the NOEC (potential risk to sensitive species), the area will be referred to DEQ for further evaluation or action.

6. Specify tolerable limits on decision errors

Evaluate groundwater contribution in context to surface water inputs from upstream and storm drain outfalls.

Evaluate ecosystem and receptor characteristics that may modify/impact risk management decision.

Evaluate variability of exposure concentrations relative to sample design.

Evaluate variability of toxicity values relative to decision rule.

7. Optimize the design for obtaining data

Review DEQ files and other site-specific information to determine groundwater “plume” locations.

Evaluate techniques for chemical and plume identification.

Conduct a site-specific test of plume identification techniques in Round 1.

Collect surface water in quiescent areas for Round 1 evaluation of risk.

If source information and / or preliminary risk assessment indicate potential groundwater “plume”, develop groundwater sampling plan for sampling in Round 2.

Ecological Risk-Amphibians and Plants.

DQO Step	Outcome
1. State the problem	<p>Amphibians: If amphibian habitat areas are identified in the Round 1a reconnaissance survey, then amphibians may be at risk from exposure to chemicals that are the result of historical and ongoing releases and / or sources within the ISA.</p> <p>Aquatic Plants: If aquatic plants are identified in the Round 1a reconnaissance survey, then aquatic plants may be at risk from exposure to chemicals that are the result of historical and ongoing releases and / or sources within the ISA.</p>
2. Identify the decision	Are there amphibian and plant communities present and co-located with potentially hazardous substances in the ISA.
3. Identify inputs to the decision	<p>Field reconnaissance in Round 1 to determine presence/absence of amphibian and plant habitat and / or occurrence.</p> <p>Existing amphibian life history information and plant community information will be evaluated to determine potential habitat areas and potential for exposure.</p> <p>Toxicological literature will be evaluated to determine potential toxicity and / or bioavailability .</p> <p>Surface water will be collected in Round 1 in potential exposure areas.</p> <p>Detection limits will be lower (if analytically achievable) than risk-based values for protection of amphibians and plants.</p>
4. Define the boundaries to the study	<p>The ISA will be the geographic boundary to the study area.</p> <p>Risk evaluations may be conducted on localized communities of amphibians and plants – based on the results of the reconnaissance study.</p>
5. Develop a decision rule	<p>If habitat within the ISA is conducive to amphibian or plant communities, a risk assessment on plants and / or amphibians will be conducted.</p> <p>If the COPC concentration using the 95th UCL is greater than the NOEC/LOEC, the COPC will be retained for further evaluation (NOEC used for sensitive species).</p>

Ecological Risk-Amphibians and Plants.

DQO Step	Outcome
6. Specify tolerable limits on decision errors	Compare upstream risk levels with ISA risk levels. Evaluate ecosystem and receptor characteristics that may modify/impact risk management decision. Evaluate variability of exposure concentrations relative to sample design. Evaluate variability of toxicity values relative to decision rule.
7. Optimize the design for obtaining data	Conduct a habitat survey. Collect surface water samples in quiescent areas and within other potential habitat areas.

Ecological Risk-Invertebrate Communities.

DQO Step	Outcome
1. State the problem	<p>Aquatic Invertebrates:</p> <ul style="list-style-type: none"> • Aquatic invertebrates may be at risk from exposure to chemicals that are the result of historical and ongoing releases and / or sources within the ISA. • There is a need for additional characterization of the aquatic invertebrate community to better understand their potential exposure pathways and their role in the food chain within the ISA.
2. Identify the decision	<p>Determine whether exposure to hazardous substances in the ISA pose an unacceptable risk to invertebrate communities in the area.</p> <p>Determine the structural characteristics of the benthic community.</p>
3. Identify inputs to the decision	<p>Existing Category 1 sediment data, bathymetry, toxicity testing, and SPI results will be evaluated to determine potential exposure areas and data gaps.</p> <p>Existing invertebrate community information will be evaluated to determine potential exposure areas.</p> <p>Toxicological literature and existing toxicity tests will be evaluated to determine potential toxicity and / or bioavailability issues.</p> <p>Additional grab samples and multi-plate samples in Round 1 will be used to further characterize the invertebrate community.</p> <p>In Round 1, additional surface sediment and invertebrate tissue data will be collected in areas that are likely depositional (e.g., not subject to significant scour under most hydrologic conditions).</p> <p>If Round 1 groundwater DQO process suggests GW sampling is warranted, groundwater may be collected in Round 2 to evaluate potential localized adverse effects.</p> <p>Detection limits will be lower (if analytically achievable) than risk-based values for protection of benthic/epibenthic organisms.</p>

Ecological Risk-Invertebrate Communities.

DQO Step	Outcome
4. Define the boundaries to the study	<p>The ISA will be the geographic boundary to the study area.</p> <p>Invertebrates will be evaluated at the community level, however a range of risk estimates will be provided using the NOECs and the LOECs. Single chemical data for invertebrate adverse effects may be limited, in which case a threshold value will be developed.</p> <p>Data will be collected in Round 1 for use in decision-making in the fall, 2002.</p> <p>Both historic and Round 1 data will be used in the risk estimate to evaluate temporal scale.</p> <p>Groundwater (porewater) may be collected only in “plume” areas (at point of exposure) in Round 2 (if “plume” found in Round 1).</p> <p>Surface sediment defined as upper 15 cm.</p> <p>Timeframe will be summer/fall 2002.</p>
5. Develop a decision rule	<p>If the COPC concentration using the 95th UCL is greater than the NOEC, the COPC will be retained for further evaluation (to bracket potential risk to sensitive species).</p> <p>If the COPC concentration using the 95th UCL is greater than the LOEC, the COPC will be retained for further evaluation.</p> <p>If groundwater DQO process suggests “plume”, additional GW porewater or seep samples in Round 2 will be compared to NOECs and LOECs for protection of aquatic organisms.</p>
6. Specify tolerable limits on decision errors	<p>Evaluate ecosystem and receptor characteristics that may modify/impact risk management decision.</p> <p>Evaluate variability of toxicity values relative to decision rule.</p> <p>Evaluate variability of exposure concentrations relative to sample design.</p> <p>Evaluate variability of natural invertebrate community.</p> <p>Compare data to reference areas.</p>
7. Optimize the design for obtaining data	<p>Collect surface (0-15 cm grab) sediment samples in each invertebrate exposure area.</p> <p>Collect benthic organisms to qualitatively determine community structure.</p> <p>Collect invertebrate samples from multi-plates to qualitatively determine community structure.</p>

Ecological Risk-Fish.

DQO Step	Outcome
1. State the problem	Fish may be at risk from exposure to chemicals that are the result of historical and ongoing releases and / or sources within the ISA.
2. Identify the decision	Determine whether exposure to hazardous substances in the ISA pose an unacceptable risk to fish populations in the area.
3. Identify inputs to the decision	<p>Existing Category 1 sediment and Category 2 tissue data will be evaluated to determine candidate exposure areas and data gaps.</p> <p>Existing fish life history information will be evaluated to determine potential exposure areas.</p> <p>Toxicological literature will be evaluated to determine potential toxicity and / or bioavailability issues.</p> <p>Tissue residue data from the literature will be used to determine adverse effect levels.</p> <p>Surface sediment, surface water, invertebrate tissue (benthic infauna) and fish tissue (sculpin, juvenile Chinook, Pacific lamprey ammocoete, large scale sucker, small mouth bass) data will be collected in exposure areas in Round 1.</p> <p>Detection limits will be lower (if analytically achievable) than risk-based values for protection of fish populations.</p>
4. Define the boundaries to the study	<p>The ISA will be the geographic boundary to the study area.</p> <p>Fish will be evaluated at the population level, however a range of risk estimates will be provided using the NOECs (to evaluate individual level for T&E species) and the LOECs.</p> <p>Exposure media will be collected in Round 1 for use in decision-making in the fall, 2002.</p> <p>Both historic and new data will be used in the risk estimate to evaluate temporal scale.</p>
5. Develop a decision rule	<p>If the COPC concentration using the 95th UCL is greater than the NOEC, the COPC will be retained for further evaluation (to bracket potential risk to sensitive species).</p> <p>If the COPC concentration using the 95th UCL is greater than the LOEC, the COPC will be retained for further evaluation.</p>

Ecological Risk-Fish.

DQO Step	Outcome
6. Specify tolerable limits on decision errors	Compare upstream risk levels with ISA risk levels. Evaluate ecosystem and receptor characteristics that may modify/impact risk management decision. Evaluate variability of exposure concentrations relative to sample design. Evaluate variability of toxicity values relative to decision rule.
7. Optimize the design for obtaining data	Collect surface water samples for comparison to effects-based criteria. Collect fish tissue to compare to tissue residue effects data. Collect invertebrate tissue and sediment (0-15 cm) grab samples to evaluate dietary pathway (dietary based NOEAL or LOAEL).

Ecological Risk-Birds.

DQO Step	Outcome
1. State the problem	Birds may be at risk from exposure to chemicals that are the result of historical and ongoing releases and / or sources within the ISA.
2. Identify the decision	Does exposure to hazardous substances in the ISA pose an unacceptable risk to avian species that may forage in the area.
3. Identify inputs to the decision	<p>Existing Category 1 sediment and Category 2 tissue data will be evaluated to determine potential exposure areas and data gaps.</p> <p>Existing life history information of representative avian species will be reviewed to select appropriate representative species and exposure parameters.</p> <p>Toxicological literature will be searched to develop no observed adverse effects level (NOAEL) and lowest observed adverse effects level (LOAEL) to avian species to determine relative sensitivities.</p> <p>Additional surface sediment and prey data will be collected in avian exposure areas.</p> <p>Detection limits will be lower (if analytically achievable) than risk-based values for protection of avian species.</p>
4. Define the boundaries to the study	<p>The ISA will be the geographic boundary to the study area.</p> <p>Avian species will be evaluated at the population level, however a range of risk estimates will be provided using the NOAEL and the LOAEL.</p> <p>Exposure media will be collected in Round 1 for use in decision-making in the fall, 2002.</p> <p>Both historic and new data may be used in the risk estimate to evaluate temporal scale.</p> <p>New tissue data may be limited to available invertebrate/fish species (as opposed to specific quantities of one prey species) of sufficient quantity for analysis.</p> <p>Data will be collected in summer/fall 2002 for Round 1.</p>
5. Develop a decision rule	<p>If the dose estimate using the 95th UCL is greater than the NOAEL, the COPC will be retained for further evaluation (potential risk to sensitive species).</p> <p>If the dose estimate using the 95th UCL is greater than the LOAEL, the COPC will be retained for further evaluation.</p>

Ecological Risk-Birds.

DQO Step	Outcome
6. Specify tolerable limits on decision errors	Compare upstream risk levels with ISA risk levels. Evaluate ecosystem and receptor characteristics that may modify/impact risk management decision. Evaluate variability of exposure concentrations relative to sample design. Evaluate variability of toxicity values relative to decision rule.
7. Optimize the design for obtaining data	Collect surface (0-15 cm grab) sediment samples in each avian exposure area. Collect prey tissue (invertebrate and / or fish tissue) from each avian exposure area. Invertebrate tissue may include benthic grab infauna samples, crayfish, and clam tissue. Fish tissue may include large scale sucker, small mouth bass, and sculpin.

Ecological Risk-Mammals.

SQO Step	Outcome
1. State the problem	Mammals may be at risk from exposure to chemicals that are the result of historical and ongoing releases and / or sources within the ISA.
2. Identify the decision	Determine whether exposure to hazardous substances in the ISA pose an unacceptable risk to mammalian species that may forage in the area.
3. Identify inputs to the decision	<p>Existing Category 1 sediment and Category 2 tissue data will be evaluated to determine potential exposure areas and data gaps.</p> <p>Existing life history information of representative mammalian species will be reviewed to select an appropriate representative species and exposure parameters.</p> <p>Toxicological literature will be searched to develop no observed adverse effects level (NOAEL) and lowest observed adverse effects level (LOAEL) to mammalian species to determine relative sensitivities.</p> <p>Additional surface sediment (0-15 cm) and prey data (crayfish, fish) will be collected in mammalian exposure areas.</p> <p>Detection limits will be lower (if analytically achievable) than risk-based values for protection of mammalian species.</p>
4. Define the boundaries to the study	<p>The ISA will be the geographic boundary to the study area.</p> <p>Mammalian species will be evaluated at the population level, however a range of risk estimates will be provided using the NOAEL and the LOAEL.</p> <p>Exposure media will be collected in Round 1 for use in decision-making in the fall, 2002.</p> <p>Both historic and new data will be used in the risk estimate to evaluate temporal scale.</p> <p>New tissue data may be limited to available invertebrate/fish species (as opposed to specific quantities of one prey species) of sufficient quantity for analysis.</p>
5. Develop a decision rule	<p>If the dose estimate using the 95th UCL is greater than the NOAEL, the COPC will be retained for further evaluation (bracket potential risk to sensitive species).</p> <p>If the dose estimate using the 95th UCL is greater than the LOAEL, the COPC will be retained for further evaluation.</p>

Ecological Risk-Mammals.

SQO Step	Outcome
6. Specify tolerable limits on decision errors	Compare upstream risk levels with ISA risk levels. Evaluate ecosystem and receptor characteristics that may modify/impact risk management decision. Evaluate variability of exposure concentrations relative to sample design. Evaluate variability of toxicity values relative to decision rule.
7. Optimize the design for obtaining data	Collect surface (0-15 cm grab) sediment samples in each mammalian exposure area. Collect co-located prey tissue (invertebrate and / or fish tissue) from each mammalian exposure area.

Human Health Risk.

<i>DQO Step</i>	Output
1. State the Problem	Need to estimate potential risks to human health associated with exposure to chemicals that are the result of historical and ongoing releases and / or sources within the ISA.
2. Identify the Decision	Determine whether chemicals in sediment, water, or biota that are the result of historic and ongoing sources in the ISA result in unacceptable risks to human health and warrant consideration of further investigation or possible response action.
3. Identify the Inputs to the Decision	<p>Existing sediment, surface water, groundwater and tissue data will be evaluated to identify chemicals associated with historic and ongoing sources in the ISA. These chemicals will be the focus of Round 1 investigations.</p> <p>Historic fish consumption studies will be evaluated to identify biota in the ISA that are consumed by human receptors. An additional qualitative consumption survey may be conducted following the Round 1 Risk Assessment to gather additional site-specific information regarding biota consumed.</p> <p>EPA exposure factors and historic fish consumption studies will be used to identify consumption rates for biota. An additional quantitative consumption survey may be conducted following the Round 1 Risk Assessment to gather additional site-specific information regarding consumption rates.</p> <p>Zoning maps, aerial photos, and city plans will be used to identify human use areas in the ISA.</p> <p>Round 1 investigations will measure concentrations of chemicals in biota tissue consumed by human receptors.</p> <p>Round 1 investigations will measure concentrations of chemicals in sediment where human exposure may occur.</p> <p>Round 1 investigations will measure concentrations of chemicals in surface water where human exposure may occur.</p> <p>Detection limits will be lower (if analytically achievable) than risk-based values for protection of human health.</p> <p>A Field Reconnaissance Survey will identify locations of groundwater seeps where human exposure may occur.</p> <p>Toxicity databases will be evaluated to select dose response data for COPCs with potential adverse effects to human health.</p>

Human Health Risk.

<i>DQO Step</i>	Output
4. Define the Boundaries	<p>Target populations:</p> <ul style="list-style-type: none">Composite sediment samplesComposite surface water samplesComposite tissue samples <p>Spatial boundaries:</p> <ul style="list-style-type: none">Sediment – Surface sediment within human use areas in the ISASurface water – River water samples within quiescent areas of the ISA used for recreation (e.g., Willamette Cove) and in the main channelTissue – Fish and shellfish collected within ISA <p>Timeframe:</p> <ul style="list-style-type: none">Sediment – During low water when most of bank is exposed and during summer when beach use is most likelySurface water – During summer when swimming would occurTissue – All times with emphasis during April through October <p>Practical constraints:</p> <ul style="list-style-type: none">Tissue – Sufficient quantity of individual species within ISA for composite samples
5. Develop a Decision Rule	<p>If the risk estimated using the 95th percentile upper confidence limit on the average exceeds EPA-acceptable risk levels, then evaluate the need for further investigations to gather additional site-specific data.</p>
6. Specify Tolerable Limits on Decision Error	<p>Conservative assumptions will be used and risks will be estimated using ranges of potential exposure values.</p>
7. Optimize the Design	<ul style="list-style-type: none">Collect surface sediment samples in human use areasCollect fish and shellfish tissue – target fish species for human consumption, whole body and filletsCollect surface water samples in human use areas

Potential for Recontamination

DQO Step	Output
1. State the Problem	Need to understand the potential for recontamination at locations where remedial action is undertaken.
2. Identify the Decision	<p>Determine if there are locations where there is an unacceptable risk of contaminated sediment from else where on the Site, to recontaminate the location (All remedies)</p> <p>Determine if there are locations where there is an unacceptable risk of recontamination by surface water, point source discharges, groundwater, or seeps?</p>
3. Identify the Inputs to the Decision	<p>Identification of locations that are depositional in nature, such that sediment chemistry from other, nearby locations may settle out, based on Hydrodynamic Model.</p> <p>Surface sediment chemistry results from Round 1 sampling.</p> <p>Identify potential sources of point source discharges (permitted or unpermitted), groundwater, or seeps that could reenter a site, during Round 1.</p> <p>An understanding of the state of source control from ODEQ.</p> <p>Sedimentation rates from settling traps and select Be7 and Pb210 cores, during Round 2</p> <p>Concentrations from point source discharges (permitted or unpermitted), groundwater, or seeps identified in Round 1, collected in Round 2.</p>
4. Define the Boundaries	The ISA will be the geographic boundary. Expand to other areas in later rounds if the Risk Assessment dictates.
5. Develop a Decision Rule	For specific areas that are identified as remediation locations and are potential candidates for reentry of chemicals, run a recontamination numeric model to evaluate the potential for and uncertainty of reentry.
6. Specify Tolerable Limits on Decision Error	<p>Evaluate the recontamination model results and a Risk Assessment evaluation based on those model results. Uncertainty and sensitivity analysis of recontamination model and risk assessment will affect the decision.</p> <p>Dependent on the time frame for source control implementation.</p>

Potential for Recontamination

DQO Step	Output
7. Optimize the Design	Initial evaluation of Round 1 data will focus the locations where there is a concern about recontamination. This will allow for limited and specific data collection for the specific sites. If concern for recontamination is great, efforts may initially best be spent on source control (e.g., outfall or seep discharge) or elimination of chemical source (e.g., upstream sediment source), prior to performing remedy at the location in question.

Natural Attenuation Potential.

DQO Step	Output
1. State the Problem	Need to understand specific elements of the physical system sufficient to make an initial determination of candidate natural attenuation areas.
2. Identify the Decision	Determine if natural attenuation is a viable alternative that needs further investigation. If so, where are the areas most likely to be suitable for natural attenuation that require further study?
3. Identify the Inputs to the Decision	Need data sufficient to run a preliminary natural attenuation model. These include: Surface sediment chemistry – from General Round 1 Nature and Extent Sampling Water content/sp. Gravity/grain size – from General Round 1 Nature and Extent Sampling Hydrodynamic model results – preliminary run will be sufficient Uncertainty and sensitivity analysis of the hydrodynamic model Sedimentation rates – select water column samples and select Be 7 and Pb210 cores (Round 2) Chemistry of Incoming Sediments – select water column samples for TSS, dissolved and total chemical analysis (Round 2) Mixed Layer Depth – Select Be7 and Pb210 cores (Round 2)
4. Define the Boundaries	Conduct Round 1 data gathering in the area covered by the nature and extent sampling, with a focus on the area outside of the navigation channel. Expand to other areas in later rounds as Risk Assessment dictates.
5. Develop a Decision Rule	If an area has desirable physical and chemical characteristics that make it suitable for natural attenuation, then collect further additional data to do a more refined evaluation of the area. Use general physical information to determine most likely areas for natural attenuation (e.g., not the “chute” but the depositional area). Conduct select sampling described in 3, in these areas. Use information to define a range of model parameter values likely. Input range of values into model and identify areas with the highest probability of natural attenuation as a viable alternative.

Natural Attenuation Potential.

DQO Step	Output
6. Specify Tolerable Limits on Decision Error	Sampling and hydrodynamic modeling must be sufficient to provide a reasonable confidence that the spatial range of possible conditions has been sampled. Some understanding of the variability over time will be needed as well. Thus, water column sampling must occur over a range of river flow/runoff conditions especially low and high river velocity events, if possible.
7. Optimize the Design	<p>The overall approach to natural attenuation modeling is described in the Natural Attenuation memorandum. Budget constraints relative to other sampling efforts must be understood to determine numbers and locations of samples for natural attenuation sampling.</p> <p>A budget priority decision is needed on whether to proceed with this analysis, prior to doing any detailed Sampling Design.</p> <p>The overall approach to natural attenuation modeling is described in the Natural Attenuation memorandum. Budget constraints relative to other sampling efforts must be understood to determine numbers and locations of samples for natural attenuation sampling.</p> <p>A budget priority decision is needed on whether to proceed with this analysis, prior to doing any detailed Sampling Design.</p>

APPENDIX F

**Manchester Environmental Laboratory
Standard Operation Procedure for the Determination of Percent Lipids in Fish version 2.0
MAN SOP LIPIDS 730009_Lipid.PDF**

MANCHESTER

ENVIRONMENTAL

LABORATORY

Standard Operating Procedure for the Determination of Percent Lipids in Fish

Version 2.0

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Index No. 730009

Standard Operating Procedure for the Determination of Percent Lipids In Fish Tissue

1.0 Scope and Application.

- 1.1 The determination of percent lipids in fish tissue at the Manchester Laboratory is a variation of the established survey methodology found in "National Study of Chemical Residues in Fish".
- 1.2 This method is restricted for use by, or under the supervision of, analysts experienced in extraction techniques employed at the Manchester Laboratory.

2.0 Summary of Method

- 2.1 An aliquot of fish is extracted using a soxhlet extractor. The extract is placed into a tared beaker and dried to constant weight.

3.0 Interferences

- 3.1 Method interferences may arise from excess water and salts carried over from the extraction process. The tissue must be well dried with Na_2SO_4 .
- 3.2 All glassware must be scrupulously washed with hot water and detergent then thoroughly rinsed with acetone and the solvent to be used. After the glassware is cleaned, it is rinsed with DI water and heated in a muffle furnace for 30 minutes at 430° C.

4.0 Sample Collection, Preservation, Storage and Holding Times.

- 4.1 See SOP 73002.

5.0 Apparatus and Materials

- 5.1 Three ball Snyder columns, 300 mm long.
- 5.2 500 mL Kuderna-Danish flask (K-D) with 19/22 receiving joint and 24/40 condenser joint.
- 5.3 10 mL concentrator tubes with 19/22 joint.
- 5.4 Scoopulas
- 5.5 50 mL, 150 mL and 250 mL beakers.

- 5.6 Top loading balance accurate to 0.0001 g.
- 5.7 Drying oven capable of maintaining 110• C.

6.0 Reagents

- 6.1 Hexane and Methylene Chloride - Pesticide grade or equivalent.
- 6.2 Anhydrous Sodium Sulfate - Muffle in a shallow tray at 430• C overnight to remove interfering organic substances.

7.0 Procedure

- 7.1 Have the chemist complete an "Extraction Worksheet."
- 7.2 The % lipids extraction may be combined with the extraction for other analyses. Label and weigh to 0.0005 g one 50 mL beaker for each sample.
- 7.3 Remove frozen fish tissue from the freezer and place them in the walk-in cooler one day prior to extraction. Do not allow them to stand at room temperature overnight!
- 7.4 Weigh out approximately 50 g of Na₂SO₄ into a 250 mL beaker. Retare the balance and weigh the tissue, not to exceed 10 g, onto the Na₂SO₄. Record the weight to the nearest hundredth of a gram and mix thoroughly until uniform. If the mixture is still clumpy or wet add 5 g more Na₂SO₄ and mix again.
- 7.5 Carefully transfer the mixture to a 94 mm x 33 mm extraction thimble. For the less dense fish fillets or those that require extra Na₂SO₄, use 110 mm x 33 mm thimbles.
- 7.6 Transfer the thimble to a soxhlet extractor charged with 50:50 methylene chloride:hexane and rinse the 250 mL beaker with 10 mL of methylene chloride. Pour the rinsate onto the thimble. If the extract is to be used for other analyses add spike mixtures specified in the extraction worksheet. Fit the condenser to the soxhlet. Turn on the chiller. Turn on the hotplate using the switches on the north wall and extract overnight.
- 7.7 After extracting for 18 hours, turn hotplates off and allow the flasks to cool. Rinse the joint between condenser and soxhlet with hexane and drain liquid in the soxhlet into the flask below. Remove and place the thimble in plastic waste bucket under the fume hood. Rinse the bottom 24/40 joint of the soxhlet and allow the rinsate to flow into the flask.
- 7.8 Pour the extract into a 500 mL KD. Rinse the flask three times with hexane pouring the contents into the KD each time. Rinse any solidified fat with hexane and decant the solvent free of the solidified fat into the KD.
- 7.9 Add a boiling chip, fit the KD with a snyder column and condenser. Wet the snyder

column with 1-2 mL hexane and reduce the volume of solvent to 5-10 mL on a steam bath, collecting the distilled solvent for disposal.

- 7.10 Allow the K-D apparatus to cool. Once cool, disassemble, rinsing all glass connections with hexane into the concentrator tube. If splitting off a portion of the extract for gas chromatographic analysis, dilute to 10 mL with hexane, mix and transfer the required fraction to the tared beaker. Transfer the label to the concentrator tube. If the entire extract is for % lipids transfer the entire contents with rinsings to the tared beaker.
- 7.11 Allow the extract to evaporate to dryness in the hood. Place the beakers in the drying oven overnight (16 hours) at 110° C. Remove and allow to cool to room temperature before weighing. Calculate the percent lipids using the formula in section 9.1.

8.0 Calibration and Standardization

- 8.1 No standards are used in this procedure.

9.0 Calculations

$$9.1 \quad \% \text{ Lipids} = \frac{(\text{final weight} - \text{tare weight}) \times 100}{(\text{Weight extracted} \times \text{fraction used for \% lipids})}$$

10.0 Quality Control

- 10.1 A method blank, processed as samples, must accompany each sample batch.
- 10.2 No standard reference material for percent lipids in fish tissue is available at this time. In order to determine accuracy and precision associated with the analysis of a set of samples, analyze a reference fish sample with the sample set and compare the result to the mean recovery obtained using the same tissue during the initial demonstration of capability. Agreement should be within 2 standard deviations (95%) of the mean. If the result falls outside this acceptance limit, the data associated with the data set are suspect and the analyst should repeat the analysis.

11.0 Precision and Accuracy

- 11.1 At least one blank is analyzed with each set. Weight difference (final weight - tare weight) of the blank should be less than or equal to the least weight difference of any sample.

12.0 Safety

- 12.1 The toxicity and carcinogenicity of each reagent has not been precisely defined. Each chemical must be treated as a potential health hazard and exposure must be reduced to the

lowest possible level. Refer to the general safety policy and procedures found in the Manchester Laboratory Safety and Health Manual.

- 12.2 The analysts must be familiar with the location and proper use of the fume hoods, eye washes, safety showers and fire extinguishers. In addition, the analysts must wear protective clothing and eye protection at all times.
- 12.3 Fume hoods must be utilized whenever possible to avoid potential exposure to organic solvents. When concentrating extracts or exchanging solvents on the steam bath, pull the hood sash down BELOW eye level or wear a full-sized face shield.
- 12.4 Work with solvents or chemicals may be performed only when another person is present.
- 12.5 Follow guidelines in the Manchester Environmental Laboratory Chemical Hygiene Plan

13.0 Hazardous Waste Disposal

- 13.1 Solvent wastes, glassware and equipment rinsates, must be discarded in labeled solvent waste bottles stored in cabinets beneath fume hoods in extraction rooms 8 and 10.
- 13.2 Solid wastes (thimbles from extracted samples, disposable Pasteur pipets and drying agents) must be discarded in plastic buckets stored under fume hoods in rooms 8 and 10.
- 13.3 All waste containers must be labeled indicating the type of waste present (ie. halogenated or non-halogenated solvents, recyclable).
- 13.4 Place damaged or broken glassware with the repairable glassware or discarded in the appropriate boxes in rooms 8 and 10.
- 13.5 Follow guidelines found in the Manchester Laboratory Waste Disposal Manual.

14.0 Bibliography

- 14.1 National Study of Chemical Residues in Fish, Volume II, US EPA Office of Science and Technology, EPA 823-R-92-008b, September 1992.
- 14.2 Manchester Laboratory SOP 730001, Pesticides Screening and Compound Independent Elemental Quantitation by Gas Chromatography with Atomic Emission Detection (AED), Method 8085.
- 14.3 Manchester Laboratory SOP 730002, Analysis of Organochlorine Pesticides, PCB Congeners, and Polychlorinated Biphenyls by GC/ECD SW-846 Methods 8000B/8080/8081/8082.

- 14.4 Manchester Laboratory SOP 730072, Extraction of Fish Tissue for Semi-Volatile Analytes, including Pesticides, PCBs and BNAs by GC/AED, GC/ECD and/or GC/MS.
- 14.5 Manchester Laboratory Safety and Health Manual.
- 14.6 Manchester Environmental Laboratory Chemical Hygiene Plan.
- 14.7 Manchester Laboratory Waste Disposal Manual.

