# Lower Duwamish Waterway Group

Port of Seattle / City of Seattle / King County / The Boeing Company

## QUALITY ASSURANCE PROJECT PLAN: FISH AND CRAB TISSUE COLLECTION AND CHEMICAL ANALYSES

FINAL

For submittal to

**The US Environmental Protection Agency Region 10** Seattle, WA

**The Washington State Department of Ecology Northwest Regional Office** Bellevue, WA

August 27, 2004

Prepared by: Wind

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## TITLE AND APPROVAL PAGE FISH AND CRAB TISSUE COLLECTION AND CHEMICAL ANALYSES QUALITY ASSURANCE PROJECT PLAN

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## Acronyms

Acronym	Definition
%RSD	percent relative standard deviation
ACG	analytical concentration goal
Axys	Axys Analytical Services, Ltd.
CAS	Chemical Abstracts Services
COC	chain of custody
Columbia	Columbia Analytical Services, Inc.
DGPS	differential global positioning system
DQO	data quality objective
DQI	data quality indicator
dw	dry weight
Ecology	Washington Department of Ecology
EPA	US Environmental Protection Agency
EPC	exposure point concentration
ERA	ecological risk assessment
FC	field coordinator
Frontier	Frontier Geosciences Inc.
GIS	geographic information system
GPS	global positioning system
HHRA	human health risk assessment
HSP	health and safety plan
LCS	laboratory control standard
LDW	Lower Duwamish Waterway
LDWG	Lower Duwamish Waterway Group
MDL	method detection limit
MLLW	mean lower low water
NMFS	National Marine Fisheries Service
OSHA	Occupational Safety and Health Administration
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl
PM	project manager
PSAMP	Puget Sound Ambient Monitoring Program
PSEP	Puget Sound Estuary Program
QA/QC	quality assurance/quality control
QAPP	Quality Assurance Project Plan
RBG	risk-based goal
RI	Remedial Investigation
RM	river mile

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Acronym	Definition
ROC	receptor of concern
RPD	relative percent difference
SDG	sample delivery group
SRM	standard reference material
SVOC	semivolatile organic compound
ТВТ	tributyltin
TEQ	toxic equivalent
тм	task manager
UCL	upper confidence limit
USFWS	US Fish and Wildlife Service
WAAS	wide area augmentation system
WDFW	Washington Department of Fish and Wildlife
YOY	young-of-the year



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## 1.0 Introduction

This quality assurance project plan (QAPP) describes the quality assurance (QA) objectives, methods, and procedures for collecting fish and crab tissue for chemical analyses from the Lower Duwamish Waterway (LDW) Remedial Investigation (RI) study area. As described in the Phase 2 RI work plan (Windward 2004g), fish and crab tissue data will be used to support the Phase 2 ecological risk assessment (ERA) and human health risk assessment (HHRA) in the RI, and may be used to estimate sediment risk-based goals (RBGs), using a sediment-to-biota food-web model. Section 3.1.6 of the Phase 2 work plan presented a preliminary study design for crab and fish tissue collection, and the associated chemical analyses to provide stakeholders with a common understanding of the objectives, background, and general design of this study. This QAPP presents the design for this study, including details on project organization, field data collection, laboratory analyses, and data management.

US Environmental Protection Agency (EPA) guidance for QAPPs was followed in preparing this project plan (EPA 2002a). The remainder of this plan is organized into the following sections:

- Section 2 Project management
- Section 3 Data generation and acquisition
- Section 4 Assessment and oversight
- Section 5 Data validation and usability
- Section 6 References
- Section 7 Oversize figures (11 x 17)

Appendix A is a health and safety plan (HSP) designed to protect on-site personnel from physical, chemical, and other hazards posed by the field sampling effort. Field collection forms are included as Appendix B. Standard operating procedures for laboratory tissue processing are included as Appendix C. The derivation of risk-based analytical concentration goals is presented in Appendix D. Tissue sample collection from background areas is discussed in Appendix E.

## 2.0 Project Management

This section describes the overall management of the project. Elements addressed include project organization and key personnel, problem definition and background, project description and scheduling, quality objectives and criteria, special training requirements and certification, and documentation and records.

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## 2.1 **PROJECT ORGANIZATION**

The overall project organization and the individuals responsible for the various tasks required for the tissue sample collection and analysis are shown in Figure 2-1. Responsibilities of project team members, as well as laboratory project managers, are described in the following sections.

## 2.1.1 Project management

The Lower Duwamish Waterway Group (LDWG), EPA, and the Washington Department of Ecology (Ecology) will be involved in all aspects of this project, including discussion, review, and approval of the QAPP, and interpretation of the results of the investigation. EPA and Ecology will be represented by their task managers (PMs) for this QAPP, Nancy Harney and Rick Huey, respectively. Allison Hiltner is the EPA PM for the Phase 2 RI.

Kathy Godtfredsen will serve as the Windward PM, responsible for overall project coordination and providing oversight on planning and coordination, work plans, all project deliverables, and performance of the administrative tasks needed to ensure timely and successful completion of the project. She will also be responsible for coordinating with LDWG, EPA, and Ecology on schedule, deliverables, and other administrative details. Dr. Godtfredsen can be reached as follows:

Kathy Godtfredsen Windward Environmental LLC 200 W. Mercer St., Suite 401 Seattle, WA 98119 Telephone: 206.577.1283 Facsimile: 206.217.0089 E-mail: kathyg@windwardenv.com

Matt Luxon will serve as the Windward Task Manager (TM). The TM is responsible for project planning and coordination, production of work plans, production of project deliverables, and performance of the administrative tasks needed to ensure timely and successful completion of the project. The TM is responsible for communicating with the Windward PM on progress of project tasks and any deviations from the QAPP. Significant deviations from the QAPP will be further reported to LDWG, EPA, and Ecology. Mr. Luxon can be reached as follows:

Matt Luxon Windward Environmental LLC 200 W. Mercer St., Suite 401 Seattle, WA 98119 Telephone: 206.577.1293 Facsimile: 206.217.0089 Email: <u>mattl@windwardenv.com</u>



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### Figure 2-1. Project organization and team responsibilities

Note: Allison Hiltner is the EPA project manager for the Phase 2 RI; Nancy Harney is the EPA task manager for the fish and crab tissue QAPP and study.

### 2.1.2 Field coordination

Bob Complita will serve as the Windward Field Coordinator (FC). The FC is responsible for managing the field sampling activities and general field and quality assurance/quality control (QA/QC) oversight. He will ensure that appropriate protocols for sample collection, preservation, and holding times are observed and will oversee delivery of environmental samples to the designated laboratories for chemical analyses. Deviations from this QAPP will be reported to the TM and PM for consultation. Significant deviations from the QAPP will be further reported to representatives of LDWG, EPA, and Ecology. Mr. Complita can be reached as follows:

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Fish and Crab Tissue QAPP August 27, 2004 Page 3 Bob Complita Windward Environmental LLC 200 W. Mercer St., Suite 401 Seattle, WA 98119 Telephone: 206.577.1297 Facsimile: 206.217.0089 Email: <u>bobc@windwardenv.com</u>

## 2.1.3 Trawl boat captain

Charlie Eaton will serve as the trawl boat captain. The trawl boat captain is responsible for operating the trawl boat and for decisions pertinent to the operation of the trawl. The trawl boat captain will work in close coordination with the FC to ensure that samples are collected in keeping with the methods and procedures presented in this QAPP. Mr. Eaton can be reached as follows:

Charles Eaton Bio-Marine Enterprises 2717 3rd Ave N Seattle, WA 98109 Telephone: 206.282.4945 Mobile: 206.714.1055 Email: <u>cmeaton@msn.com</u>

## 2.1.4 Beach seine operation

Taylor Associates will operate the beach seine. Jim Shannon is responsible for overseeing the beach seine crew and is responsible for decisions pertinent to beach seining. Taylor Associates will work in close coordination with the FC to ensure that samples are collected in keeping with the methods and procedures presented in this QAPP. Taylor Associates can be reached as follows:

Jim Shannon Taylor Associates 7104 Greenwood Ave N Seattle, WA 98103 Telephone: 206.267.1409 Mobile: 206.794.0095 Facsimile: 206.267.1401 Email: jim@taylorassoc.net

## 2.1.5 Quality assurance/quality control

Tad Deshler of Windward will oversee QA/QC for the project. As the QA/QC manager, he will oversee coordination of the field sampling and laboratory programs, and supervise data validation and project QA coordination, including coordination with the EPA QA officer, Ginna Grepo-Grove.

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Mr. Deshler can be reached as follows:

Tad Deshler Windward Environmental LLC 200 W. Mercer St., Suite 401 Seattle, WA 98119 Telephone: 206.577.1285 Facsimile: 206.217.0089 Email: <u>tad@windwardenv.com</u>

Ms. Grepo-Grove can be reached as follows:

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

Susan McGroddy will serve as Windward's QA/QC coordinator for chemical analyses. Dr. McGroddy can be reached as follows:

Susan McGroddy Windward Environmental LLC 200 W. Mercer St., Suite 401 Seattle, WA 98119 Telephone: 206.577.1292 Facsimile: 206.217.0089 Email: <u>susanm@windwardenv.com</u>

Independent third-party chemical data review and validation will be provided by Cari Sayler of Sayler Data Solutions, or a suitable alternative. Ms. Sayler can be reached as follows:

Cari Sayler Sayler Data Solutions, Inc. 14257 93<sup>rd</sup> Court NE Bothell, WA 98011 Telephone: 425.820.7504 Email: <u>cari@saylerdata.com</u>

### 2.1.6 Laboratory project management

Susan McGroddy of Windward will serve as the laboratory coordinator for the analytical chemistry laboratory. Columbia Analytical Services (Columbia) will perform all chemical analyses on the tissue samples, except for PCB congeners, dioxins and furans (if required), and inorganic arsenic. Axys Analytical Services Ltd. (Axys) will perform PCB congener analysis and archive samples for potential future dioxin and

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furan analyses, and Frontier Geosciences Inc. (Frontier) will perform inorganic arsenic analyses.

The laboratory PM at Columbia can be reached as follows:

Greg Salata Columbia Analytical Services, Inc. 1317 So. 13th Avenue Kelso, WA 98626 Telephone: 360.577.7222 Facsimile: 360. 636.1068 Email: <u>gsalata@kelso.caslab.com</u>

The laboratory PM at Axys can be reached as follows:

Georgina Brooks Axys Analytical Services Ltd. PO Box 2219 2045 Mills Road Sidney, British Columbia V8L 3S8 Canada Telephone: 250.656.0881 Facsimile: 250.656.4511 Email: <u>gbrooks@axys.com</u>

The laboratory PM at Frontier can be reached as follows:

Frank Colich Frontier Geosciences Inc. 414 Pontius Ave N Seattle, WA 98109 Telephone: 206.622.6960 Facsimile: 206.622.6870 Email: <u>frankc@frontiergeosciences.com</u>

The laboratories will accomplish the following:

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

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 allow EPA and the QA/QC manager, or a representative, to perform laboratory and data audits

### 2.1.7 Data management

Tad Deshler of Windward will oversee data management, and ensure that analytical data are incorporated into the LDWG database with appropriate qualifiers following acceptance of the data validation. QA/QC of the database entries will ensure accuracy for use in Phase 2.

## 2.2 PROBLEM DEFINITION AND SUPPORTING INFORMATION

The Phase 2 RI work plan (Windward 2004g) identified the need for additional fish and crab tissue data to support the human health and ecological risk assessments. This section presents the objectives, rationale, and background information for collecting and analyzing fish and crab tissue samples from the LDW. An overview of this study and its schedule is presented in Section 2.3, and a detailed study design is presented in Section 3.1. The objectives, rationale, and background information for collecting fish and crabs from background areas are presented in Appendix E.

Three fish species (juvenile chinook salmon,<sup>1</sup> English sole, and Pacific staghorn sculpin) and crabs have been identified as receptors of concern (ROCs) for the Phase 2 ERA (Windward 2003a). Risks to these organisms will be assessed, in part, through comparison of chemical concentrations in their tissues to tissue residue benchmarks of toxicity. In addition, humans and some wildlife species consume fish (including English sole, shiner surfperch, and sculpin) and crab<sup>2</sup> from the LDW, and thus concentrations of chemicals in the tissues of fish and crab are also needed to assess risks to humans and piscivorous wildlife. Existing fish and crab tissue data are insufficient to characterize risks to fish, crabs, piscivorous wildlife, or humans who consume fish or crabs from the LDW, and additional data are needed.<sup>3</sup>

Tissue samples are needed to meet the following objectives:

<sup>&</sup>lt;sup>3</sup> Although shrimp have been observed in the northernmost region of the LDW (Windward 2004a, c, d) and may be consumed by humans, they will not be collected for chemical analyses. Risk estimates associated with the consumption of crabs will adequately represent risks associated with shrimp consumption because crabs: 1) are larger, more abundant, and more widespread than shrimp in the LDW; 2) are more closely associated with the sediment than shrimp; and 3) live longer than shrimp. Consequently, chemical concentrations in crab, particularly those of bioaccumulative chemicals, are likely to be higher in crab than in shrimp.



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<sup>&</sup>lt;sup>1</sup> The collection and chemical analyses of juvenile chinook salmon tissue samples, described in the juvenile chinook salmon QAPP (Windward 2003c), was completed in 2003 (see juvenile chinook salmon data report (Windward 2004e).

<sup>&</sup>lt;sup>2</sup> Clam and market basket benthic invertebrate tissue chemistry data will also be collected to supplement the available wildlife prey data as described in the Benthic Invertebrate QAPP (Windward 2004f).

- to supplement existing fish and crab tissue chemistry data so that exposure of fish and crabs to bioaccumulative chemicals can be estimated for the assessment of risks using a critical tissue residue approach
- to supplement existing fish and crab tissue chemistry data to make possible an estimate of human, fish,<sup>4</sup> and wildlife exposure to bioaccumulative chemicals in fish or crab, using a dietary approach
- to provide data on chemical concentrations in fish and crab tissue throughout the LDW to calibrate a food-web model that will be applied to evaluate the relationship between chemicals in sediment and chemicals in tissue
- to provide congener-specific tissue chemistry data regarding polychlorinated biphenyls (PCBs) in critical prey species to supplement existing PCB Aroclor tissue chemistry data for the assessment of PCB risks to wildlife and humans
- to provide data regarding the concentrations of total arsenic (and inorganic arsenic in a subset of samples), in English sole, perch, and crabs collected from the LDW and from background areas<sup>5</sup> for the Phase 2 HHRA

A summary of existing tissue chemistry data for targeted species for this QAPP is presented in Table 2-1 and Figure 2-2 (oversized figure, Section 7.0). Chemical data are available for English sole, perch, and crabs collected from several areas within the LDW. All the chemical data from these studies are acceptable for all uses in Phase 2, as described in the historical data technical memorandum (Windward 2004i). No chemical data are available for Pacific staghorn sculpin. The sufficiency of the data collected under this QAPP and the historical data to meet the above objectives are described for each species in Section 3.1.6.

<sup>&</sup>lt;sup>5</sup> Clams will also be collected in background areas, as discussed in Appendix E. Clam collection in the LDW is discussed in the benthic invertebrate QAPP (Windward 2004f).



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<sup>&</sup>lt;sup>4</sup> Metals (except mercury) and PAHs will be assessed using a dietary approach; all other chemicals will be assessed using a critical tissue residue approach.

# Table 2-1. Tissue chemistry samples for targeted species collected from the LDW since 1990

TITLE	YEAR	SPECIES	N <sup>a</sup>	SAMPLE TYPE	NUMBER OF ANIMALS PER SAMPLE	CHEMICALS	
Waterway Sediment Operable		English sole	3	skinless fillet	5		
Unit Harbor Island Superfund	1008	red rock crab	3	edible meat	5	Ha TRT PCBs	
risks from the consumption of	1990	Dungeness crab	1	edible meat	1	rig, 101, 1003	
seafood (ESG 1999)		striped perch	3	skinless fillet	1-5		
		Dungeness crab	2	edible meat	3		
King County Combined Sewer Overflow Water Quality		Durigeness crab	1	hepatopancreas	3		
Assessment for the Duwamish River and Elliott Bay (King County 1999b) <sup>b</sup>	1996- 1997	English sole	3	skinless fillet	20	metals, TBT, SVOCs, PCBs	
			3	whole body <sup>c</sup>	20		
		shiner surfperch	3	whole body	10		
	1992	English sole	3	skinless fillet	10-20	SVOCs, organochlorine pesticides, PCBs, As, Cu, Pb, Hg	
Puget Sound Ambient Monitoring Program – annual sampling (West et al. 2001)	1995	English sole	3	skinless fillet	10-20	organochlorine pesticides, PCBs, As, Cu, Pb, Hg	
	1997	English sole	3	skinless fillet	10-20	Hg, organo-chlorine pesticides	
Elliott Bay/Duwamish River Fish Tissue Investigation (Battelle 1996; EVS unpublished; Frontier Geosciences 1996)	1995	English sole	3	skinless fillet	6	PCBs, Hg, MeHg, TBT	

MeHg – methylmercury

PCB – polychlorinated biphenyl

SVOC - semivolatile organic compound

TBT – tributyltin

- <sup>a</sup> Number of individual or composite samples
- <sup>b</sup> Data from crab and English sole samples that were cooked were collected during the King County Water Quality Assessment, but were not used in the Phase 1 RI or in the quantitative sections of the Phase 1 risk assessment. These data were used by King County (1999a) in their HHRA.
- <sup>c</sup> Samples are remnants following the subsampling of fillet tissue. In addition, livers were removed from some fish in the composite samples. Therefore, these samples will not be used to calculate exposure point concentrations (EPCs) in the Phase 2 risk assessment.

As discussed in the Phase 2 work plan (Windward 2004g), existing data on fish and crab tissue chemistry are not sufficient to meet data needs identified for the Phase 2 RI. Data needs for targeted tissue samples are summarized in Table 2-2.



TISSUE TYPE	HHRA DATA NEED	ERA DATA NEEDS
English sole, whole body	Site-wide chemical data needed for ingestion dose estimate, including total and inorganic arsenic; risks associated with specific LDW areas will be discussed in the uncertainty analysis; background site data needed for both total and inorganic arsenic	Site-wide chemical data needed for critical tissue residue approach for fish; and ingestion dose estimate for sculpin and wildlife
English sole, fillet	Site-wide chemical data needed for ingestion dose estimate, including total and inorganic arsenic; risks associated with specific LDW areas will be discussed in the uncertainty analysis; background site data needed for both total and inorganic arsenic	Not used
Shiner surfperch, whole body <sup>a</sup>	Site-wide chemical data needed for ingestion dose estimate, including total and inorganic arsenic; risks associated with specific LDW areas will be discussed in the uncertainty analysis; background site data needed for both total and inorganic arsenic	Site-wide chemical data needed for ingestion dose estimate for sculpin and wildlife
Sculpin, whole body	Not used	Site-wide chemical data needed for critical tissue residue approach for fish; and ingestion dose estimate for wildlife
Crab, edible meat	Site-wide chemical data needed for ingestion dose estimate, including total and inorganic arsenic; risks associated with specific LDW areas will be discussed in the uncertainty analysis; background site data needed for both total and inorganic arsenic	Site-wide chemical data needed for critical tissue residue approach for crabs; and ingestion dose estimate for fish and wildlife
Crab, hepatopancreas	Site-wide chemical data needed for ingestion dose estimate, including total and inorganic arsenic; risks associated with specific LDW areas will be discussed in the uncertainty analysis; background site data needed for both total and inorganic arsenic	Site-wide chemical data needed for critical tissue residue approach for crabs (hepatopancreas-based TRVs); and ingestion dose estimate for fish and wildlife <sup>b</sup>

Table 2-2. Summary of data needs for fish and crab tissue samples

The collection of juvenile chinook salmon tissue samples for chemical analyses is described in the juvenile chinook salmon QAPP (Windward 2003c). Tissue sample collection from background areas is discussed in Appendix E.

<sup>a</sup> If a sufficient number of adult striped or pile perch are caught while sampling for other fish species, fillets from these fish will be composited separately, in consultation with EPA and Ecology, and chemically analyzed.

<sup>b</sup> Crab hepatopancreas data will be combined with edible crab meat data to estimate whole-body crab concentrations for use in sculpin and wildlife dietary dose estimates.

## 2.3 PROJECT DESCRIPTION AND SCHEDULE

To meet the objectives presented in Section 2.2, the crab and fish tissues identified in Table 2-2 will be collected from specific areas throughout the LDW and from Puget Sound background areas, as described in Section 3.1 and Appendix E, respectively. Samples will be collected from multiple areas in the LDW; these areas were selected based on the distribution of PCB concentrations in sediment.

Shiner surfperch sampling will be conducted from August 2 to 6, 2004, prior to the anticipated final approval date of this QAPP on August 27, 2004, because shiner surfperch abundance has typically been low in the LDW in September (see shiner

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surfperch, Section 3.2.2.3). A technical memorandum documenting collecting, processing, and archiving procedures to be following for the August 2 to 6 sampling event was approved by EPA on July 29, 2004 (Windward 2004j). All other fish<sup>6</sup> and crab tissue sampling in the LDW will take place August 30 to September 10, 2004. Trawl and beach seine sampling will occur simultaneously in early August, and trawl and trap sampling will occur simultaneously in late August/early September. Chemical analyses of the samples should be completed by October 2004 (or 30 days after compositing is completed). Background sampling will take place in September after the LDW sampling is completed (see Appendix E for additional details on the background sampling). A draft data report summarizing the survey and tissue chemistry results will be submitted to EPA and Ecology by January 7, 2005.

## 2.4 QUALITY OBJECTIVES AND CRITERIA FOR CHEMICAL MEASUREMENT DATA

The overall DQO for the collection of fish and crab tissue is to develop and implement procedures that will ensure the collection of representative data of known, acceptable, and defensible quality. Parameters used to assess data quality are precision, accuracy, representativeness, comparability, completeness, and sensitivity. These parameters are discussed, and specific data quality indicators (DQIs) for tissue laboratory analyses are presented in Section 3.4.3.

## 2.5 SPECIAL TRAINING/CERTIFICATION

The Superfund Amendments and Reauthorization Act of 1986 required the Secretary of Labor to issue regulations through the Occupational Safety and Health Administration (OSHA) providing health and safety standards and guidelines for workers engaged in hazardous waste operations. Federal regulation 29CFR1910.120 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-hour HAZWOPER training course and 8-hour refresher courses, as necessary, to meet the OSHA regulations.

Other relevant regulations involve collection permits. Three fish sampling permits are needed for the sampling described in this QAPP (Table 2-3). Permits are required by the Washington Department of Fish and Wildlife (WDFW) for any scientific collection of organisms and by the service agencies (National Marine Fisheries Service [NMFS] and US Fish and Wildlife Service [USFWS]) for incidental take of threatened fish species (i.e., chinook salmon and bull trout). The FC and the leader of each sampling team (i.e., trawl sampling, trap sampling, and beach seine sampling) will be in possession of a copy of each permit, as required by the permits. Copies of permits are available upon request.

<sup>&</sup>lt;sup>6</sup> Pacific staghorn sculpin (>10 cm), English sole (>20 cm), starry flounder (>20 cm), rockfish (> 20 cm), pile perch (> 8 cm), and striped perch (>8 cm) collected as by-catch during shiner surfperch sampling will be archived. Fish will be held frozen at Axys until target numbers for each species have been obtained.



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### Table 2-3. Permits required for sampling

Регміт	CONTACT PERSON /PERMIT HOLDER	Permit NUMBER
USFWS incidental take permit for threatened and endangered species (bull trout); required even though this species is not targeted for collection, because they may be caught incidentally in the sampling gear	Matthew Luxon, Windward Environmental	Threatened Species Permit TE088853-0
NMFS incidental take permit for threatened and endangered species (chinook salmon); required even though this species is not targeted for collection, because they may be caught incidentally in the sampling gear	George Blomberg, Environmental Management, Port of Seattle	Scientific Research Permit 1314
WDFW scientific collection permit	Matthew Luxon, Windward Environmental	Scientific Collection Permit 04-273a

NMFS – National Marine Fisheries Service

USFWS - US Fish and Wildlife Service

WDFW – Washington Department of Fish and Wildlife

## 2.6 DOCUMENTATION AND RECORDS

This section describes documentation and records needed for field activities and laboratory analysis. In addition, the data reduction process and contents of the data report are described. The documentation and record-keeping described below applies to both LDW and background area sampling.

#### 2.6.1 Field observations

All field activities will be recorded in a field logbook maintained by the FC. The field logbook will provide a description of all sampling activities, conferences associated with field sampling activities, sampling personnel, and weather conditions, plus a record of all modifications to the procedures and plans identified in this QAPP and the HSP (Appendix A). The field logbook will consist of bound, numbered pages. All entries will be made in indelible ink. The field logbook is intended to provide sufficient data and observations to enable participants to reconstruct events that occurred during the sampling period.

The following field data collection sheets, included as Appendix B, will also be used to record pertinent information after sample collection:

- target fish and crab species collection form
- protocol modification form
- corrective action form
- non-target species tally form
- composite formation form



### 2.6.2 Laboratory records

Laboratories will be responsible for internal checks on sample handling and analytical data reporting, and will correct errors identified during the QA review. The laboratory data package will be submitted electronically and will include the following:

- **Project narrative**: This summary, in the form of a cover letter, will present any problems encountered during any aspect of sample analyses. The summary will include, but not be limited to, discussion of quality control, sample shipment, sample storage, and analytical difficulties. Any problems encountered by the laboratory, and their resolutions, will be documented in the project narrative. In addition, operating conditions for instruments used for the analysis of each suite of analytes and definitions of laboratory qualifiers will be provided.
- **Records**: Legible copies of the chain-of-custody (COC) forms will be provided as part of the data package. This documentation will include the time of receipt and the condition of each sample received by the laboratory. Additional internal tracking of sample custody by the laboratory will also be documented.
- **Sample results**: The data package will summarize the results for each sample analyzed. The summary will include the following information, when applicable:
  - field sample identification code and the corresponding laboratory identification code
  - sample matrix
  - date of sample extraction/digestion
  - date and time of analysis
  - weight and/or volume used for analysis, including final dilution volumes or concentration factor for the sample
  - percent moisture in the samples
  - identification of the instruments used for analysis
  - method detection and reporting limits
  - all data qualifiers and their definitions
- QA/QC summaries: These summaries will contain the results of all QA/QC procedures. Each QA/QC sample analysis will be documented with the same information required for the sample results (see above). The laboratory will make no recovery or blank corrections. The required summaries are listed below.
  - The calibration data summary will contain the concentrations of the initial calibration and daily calibration standards and the date and time of analysis. The response factor, percent relative standard deviation (%RSD), relative

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Fish and Crab Tissue QAPP August 27, 2004 Page 13 percent differences (RPD), and the retention time for each analyte will be listed, as appropriate. Results for standards to indicate instrument sensitivity will be reported.

- The internal standard area summary will report the internal standard areas, as appropriate.
- The method blank analysis summary will report the method blank analysis associated with each sample and the concentrations of all compounds of interest identified in these blanks.
- The surrogate spike recovery summary will report all surrogate spike recovery data for organic analyses. The names and concentrations of all compounds added, percent recoveries, and QC limits will be listed.
- The matrix spike recovery summary will report the matrix spike or matrix spike/matrix spike duplicate recovery data for analyses, as appropriate. The names and concentrations of all compounds added, percent recoveries, and QC limits will be included in the data package. The RPD for all matrix spike and matrix spike duplicate analyses will be reported.
- The matrix duplicate summary will report the RPD for all matrix duplicate analyses. The QC limits for each compound or analyte will be listed.
- The standard reference material (SRM) analysis summary will report the results of the SRM analyses and compare these results to published concentration ranges for the SRMs.
- The laboratory control analysis summary will report the results of the analyses of laboratory control samples. The QC limits for each compound or analyte will be included in the data package.
- The relative retention time summary will report the relative retention times for the primary and confirmational columns of each analyte detected in the samples, as appropriate.
- **Original data:** Legible copies of the original data generated by the laboratory will be provided, including the following:
  - sample refrigerator temperature logs
  - sample extraction/digestion, preparation, and cleanup logs
  - instrument specifications and analysis logs for all instruments used on days of calibration and analysis

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• reconstructed ion chromatograms for all samples, standards, blanks, calibrations, spikes, replicates, laboratory control samples, and standard reference materials



- raw and enhanced spectra of detected compounds with associated best-match spectra for each sample
- printouts and quantitation reports for each instrument used, including reports for all samples, standards, blanks, calibrations, spikes, replicates, and laboratory control samples, and SRMs
- original data quantification reports for each sample

The contract laboratories for this project will submit data electronically, in Microsoft Excel® or delimited-text format. Guidelines for electronic data deliverables for chemical data are as follows:

- Each row of data will contain only one analyte for a given sample. Therefore, one complete sample will require multiple rows.
- Each row should contain the following information at a minimum: Windward sample identifier, sample matrix, laboratory sample identifier (if used), date of sampling, date of laboratory analysis, laboratory method, analyte name, measured result, laboratory qualifiers, units, and measurement basis.
- If using a spreadsheet file to produce the electronic deliverable, the value representing the measured concentration or detection limit will be rounded to show the correct number of significant figures and will not contain any trailing digits that are hidden in the formatting.
- If using a database program to produce the electronic deliverable, the value representing the measured concentration or detection limit will be stored in a character field, or a field in addition to the numeric result field will be provided to define the correct number of significant figures.
- If a result for an analyte is below the detection limit, the laboratory qualifier will be U, and the value in the result column will be the sample-specific detection limit.
- Analytical results of laboratory samples for QA/QC will be included and clearly identified in the file with unique laboratory sample identifiers. Additional columns may be used to distinguish the sample type (e.g., matrix spike, matrix spike duplicate).
- If replicate analyses are conducted on a submitted field sample, the laboratory sample identifier must distinguish among the replicates.
- Wherever possible, all analytes and replicates for a given sample will be grouped together.

An example of the acceptable organization of the electronic deliverable for analytical chemistry data is provided in Table 2-4.



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Table 2-4. Example of acceptable organization of electronic deliverable for	or
analytical chemistry data	

FIELD NAME	REQUIRED OR OPTIONAL
Event name	required
Chain of custody ID	required
Laboratory sample ID	required
Matrix	required
Sample collection date/time	required
Requested analysis	required
Analyte	required
Chemical Abstracts Services (CAS) registry number	required
Date/time analyzed	required
Detection limit	required
Reporting limit	required
Reporting limit type	required
Sample result	required
Units	required
Number of significant figures	required
Laboratory qualifier	optional <sup>a</sup>
Analysis batch	required
True value/spiked amount	optional
Percent recovery	optional <sup>a</sup>
Upper limit	optional
Lower limit	optional
Analyst	required
Dilution	required
Extraction batch	required
Extraction date/time	required
Extraction method	required
Laboratory notes	optional
Laboratory	required

<sup>a</sup> Required when available. Not all samples are qualified. Blanks and laboratory control standards (LCSs) have no percent moisture. Field samples have no percent recovery.

### 2.6.3 Data reduction

Data reduction is the process by which original data (analytical measurements) are converted or reduced to a specified format or unit to facilitate analysis of the data. Data reduction requires that all aspects of sample preparation that could affect the test result, such as sample volume analyzed or dilutions required, be taken into account in the final result. It is the laboratory analyst's responsibility to reduce the data, which are subjected to further review by the laboratory PM, the Windward PM, the Project QA/QC Coordinator, and independent reviewers. The data will be generated in a form amenable to review and evaluation. Data reduction may be performed manually or electronically. If performed electronically, all software used must be demonstrated to be true and free from unacceptable error.

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### 2.6.4 Data report

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

- summary of all field activities, including descriptions of any deviations from the approved QAPP
- fish and crab sampling locations reported in latitude and longitude to the nearest one-tenth of a second and in northing and easting to the nearest foot
- plan view of the study area showing the actual sampling locations
- catch data for all target and non-target species
- summary of the QA/QC review of the chemical data
- copies of field logs (appendix)
- copies of COC forms (appendix)
- data validation report (appendix)
- results from the analyses of field samples, both as summary tables in the main body of the report and appendices with data forms submitted by the laboratories and as cross-tab tables produced from Windward's database

Chemical data will be validated within four weeks of receiving data packages from the respective laboratories. A draft data report will be submitted to EPA and Ecology eight weeks after receipt of the validated analytical results. A final data report will be submitted to EPA and Ecology three weeks after receiving comments on the draft report. Once the data report has been approved by EPA and Ecology, a database export will be created from Windward's database. The data will be exported in SEDQUAL format as well as the format used to export the historical chemistry data in Phase 1, which consists of separate tables for events, locations, samples, and results.

## 3.0 Data Generation and Acquisition

This section describes the methods that will be used to collect, process, and analyze fish and crab tissue samples collected from the LDW. Elements include sampling design, fish and crab sampling methods, sample handling and custody requirements, analytical chemistry methods, QA/QC, instrument/equipment testing, inspection and maintenance, instrument calibration, supply inspection/acceptance, non-direct measurements, and data management. The sampling design and methods for collecting tissue and co-located sediment samples from background areas is described separately in Appendix E. All other elements of Section 3.0 are applicable to both background and LDW sampling and analyses.



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## 3.1 SAMPLING DESIGN

In this section, sampling designs are described for fish and crab collection in the LDW, including the selection of sampling areas, targeted species, the number of composite samples per area, the compositing scheme, and target sizes for collected fish and crabs. Section 3.2 describes the selected sampling methods for each targeted species.

As discussed in Sections 2.2 and 3.1.6, these chemical data will support the Phase 2 HHRA and ERA. Thus, tissue data must represent the exposure area for each receptor evaluated in the risk assessments and provide a sufficient total number of samples to derive an exposure point concentration (EPC). Another use of these tissue data is to calibrate a sediment-to-biota food-web model. This model will be used to estimate concentrations of chemicals in tissue for the residual risk assessments and to develop sediment RBGs for bioaccumulative chemicals that may be risk drivers within the LDW (see Windward 2004e). The data needed for model calibration was a consideration for the study design presented below.

## 3.1.1 Sampling areas

EPA, Ecology, and LDWG agreed on the number, size, and location of four sampling areas to derive river-wide and area-specific chemical concentrations in tissues to support risk assessment and sediment-to-biota food-web modeling based on a series of technical meetings and analyses (see Section 3.1.6.2 of the Phase 2 work plan, Windward 2004e). This section presents a summary of these decisions as well as a brief rationale.

Four sampling areas have been selected for collection of fish and crabs (Figure 3-1; oversized figure, Section 7.0). The locations and number of areas were based primarily on the longitudinal distribution of PCB concentrations in the sediment as defined through the use of a rolling average PCB sediment concentration (Figure 3-2). Using inverse distance weighting analysis in the project geographic information system (GIS), an interpolated grid of total PCB concentrations was calculated using Phase 1 surface sediment chemistry data. From this grid, average total PCB concentrations were calculated for 1.0-mi river segments at 0.1-mi intervals. River segments were set at 1.0 mi because trawls over an area of this size may be needed to collect a sufficient number of targeted fish species for chemical analyses. Area-weighted average sediment PCB concentrations were used to select tissue sampling areas, rather than PCB concentrations in individual sediment samples, because fish species integrate exposure over their home ranges.



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# Figure 3-2. One-mile rolling average total PCB concentrations in LDW surface sediment

The maximum rolling average PCB concentrations (> 1,200  $\mu$ g/kg dw) occur at approximately river mile (RM)<sup>7</sup> 3.1-3.4. The rolling average PCB concentration in this area was calculated using data collected between RM 2.6 and RM 3.7, where higher concentrations of PCBs have been measured (this portion of the LDW includes three early action areas). Downstream of this area, the rolling average PCB concentration is much lower (< 400  $\mu$ g/kg dw). Rolling average PCB concentrations of intermediate magnitude (800  $\mu$ g/kg dw) were calculated at the upstream end of the study area. Based on this analysis, four discrete sampling areas were established, centered on approximately RM 0.6, 2.0, 3.3, and 4.6,8 respectively (Figure 3-1; oversized figure, Section 7.0). Each sampling area is approximately 0.8 mi in length based on the feasibility of collecting sufficient numbers of each target species within each sampling area and input from EPA.<sup>9</sup> Feasibility considerations included both the minimum and maximum trawl lengths suitable for the LDW as well as a review of spatial fish abundance data. Thus, Area 1 extends from RM 0.2 to RM 1.0, Area 2 extends from RM 1.6 to RM 2.4, Area 3 extends from RM 2.9 to RM 3.7, and Area 4 extends from RM 4.2 to RM 5.0 (Figure 3-1, oversized figure, Section 7.0).

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<sup>&</sup>lt;sup>7</sup> River miles within the LDW are measured relative to the southern end of Harbor Island.

<sup>&</sup>lt;sup>8</sup> RM 4.6 was selected as the centroid rather than RM 4.8 because there are numerous obstacles in the section of the LDW upstream of RM 4.8, including a footbridge that crosses the LDW at RM 4.8 and underwater rocks, root masses, and other debris, that make trawling infeasible upstream of the bridge (Eaton 2004). Note, however, that an attempt was made to seine in this area as part of the August 2 to 6, 2004 sampling, as described in Section 3.2.3.3. Attempts will be made to catch sculpin and crabs in the area between RM 4.8 and RM 5.2 using shrimp and crab traps as described in Section 3.2.3.2.
<sup>9</sup> Based on discussions at the March 11, 2004 meeting on the tissue sampling design.

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Each of the four sampling areas has been further divided into six subareas, three on the east and three on the west side of the dredged channel centerline, except for Area 4, which was divided into five subareas because of its shape and the difficulty in sampling upstream of RM 4.8 (see Section 3.2.3.1). Dividing the sampling areas into smaller subareas will help account for the influence of varying tissue chemical concentrations within each area in the EPC calculation for the risk assessments, by increasing the variance of the concentration data. High variance in tissue chemical concentrations within an area could result from differences in prey or habitat if fish preferentially forage or have a home range similar to or smaller than each sampling area. Sampling by subarea will also provide additional data points for analyzing the relationship between sediment and tissue chemical concentrations in the food-web model, by creating 24 independent composite tissue samples for each target species. It is important to note that this study design does not allow statistical comparisons in fish and crab tissue chemical concentrations among subareas within a given area.

The tissue sampling areas selected do not provide complete coverage of the LDW, nor are they spaced at exactly equal distances. However, fish and crab are mobile species, and the tissue sampling areas represent the full range of PCB sediment concentrations present in the LDW (see Figure 3-2); these areas should thus provide a suitable dataset to evaluate risks in the LDW as well as to support the sediment-to-biota food-web model. Because the home ranges of the species to be collected are largely unknown in the LDW, the size and number of sampling areas could not be set based on home range expectations.<sup>10</sup>

## 3.1.2 Targeted species

As presented in Section 3.1.6 of the Phase 2 work plan (Windward 2004e), representative species targeted for collection in this QAPP are English sole, Pacific staghorn sculpin, shiner surfperch, and crabs.<sup>11</sup> Rockfish were mentioned in the Phase 2 work plan as another possible fish species to target for chemical analyses, depending on their site use. Based on past surveys and professional judgment of local fish experts, rockfish are not targeted for collection because adult rockfish abundance is expected to be very low,<sup>12</sup> as described in the rockfish technical memorandum (Windward 2004h).

English sole, a benthivorous fish, is targeted because it is an abundant flatfish that is present throughout the LDW and can be exposed to sediment-associated chemicals through its diet and direct contact with sediment. If a sufficient number of English sole of sufficient size cannot be collected (see Section 3.2.2.1), starry flounder will be collected as a surrogate species. Starry flounder is a flatfish species whose trophic

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<sup>&</sup>lt;sup>10</sup> The food-web model will be run considering a range of home range assumptions.

<sup>&</sup>lt;sup>11</sup> The benthic invertebrate QAPP describes the collection of benthic invertebrates, including composite samples of smaller benthic invertebrates, clams, and gastropods.

<sup>&</sup>lt;sup>12</sup> If adult rockfish are incidentally caught during this study (either August or September), they will be archived and may be chemically analyzed following consultation with EPA and Ecology.

status is similar to or higher than that of English sole (Fresh et al. 1979). Starry flounder also has greater freshwater tolerance (Eschmeyer et al. 1983).

Adult Pacific staghorn sculpin, a demersal fish, are targeted for collection because they are a higher-trophic-level fish that consumes other fish as part of their diet (Fresh et al. 1979). Sculpin are exposed to sediment-associated chemicals through their diet and through direct contact with sediment.

Several perch species are found in the LDW, including striped perch, pile perch, and shiner surfperch. Shiner surfperch are the targeted perch species because they are highly abundant, and thus are likely to be consumed by wildlife. Shiner surfperch are also consumed by humans (ATSDR 2003; EPA 1999a; Toy et al. 1996). Perch are exposed to sediment-associated chemicals primarily through their diet. Striped perch, especially adults, are not targeted for sampling because their abundance in the LDW is low and inconsistent (ESG 1999). Pile perch are not targeted because their abundance in the LDW is inconsistent, and they are believed to have less of a benthic component to their diet than shiner perch because encrusting organisms such as mussels and barnacles, which have much less exposure to sediment-associated chemicals, constitute a large portion of their diets (Fritzsche and Hassler 1989). However, because all three species can be consumed by humans, if a sufficient number of adult striped or pile perch are caught while sampling for other fish species, fillets from these fish will be collected and composited separately. Whether these composite samples would be chemically analyzed would be determined in consultation with EPA and Ecology.

As noted in Section 2.3, shiner surfperch will be collected August 2 to 6, 2004, prior to the main tissue collection effort in September 2004. Shiner surfperch move in and out of the LDW on a seasonal basis. Data from Weitkamp and Campbell (1980) suggest that adults may not be present in the LDW in September. Weitkamp and Campbell's description of shiner surfperch dynamics in the LDW is consistent with local expert opinion on fish use of the LDW.<sup>13</sup> Specifically, shiner surfperch enter the system in spring and bear their young in early summer. Adults leave the shallow nearshore waters in summer, and the young remain in shallow water through early fall (Weitkamp and Campbell 1980).

Several crab species are found in the LDW, including Dungeness, red rock, and slender crabs (also known as graceful crabs). People may capture crabs wherever they are abundant enough or large enough to warrant the harvesting effort, even if the crabs are below the legal size limits. Fish and wildlife, including Pacific staghorn sculpin, great blue heron, and river otter, may prey on crabs of appropriate size throughout the LDW wherever they are present. All four quarterly crab and shrimp surveys are complete (Windward 2004b). These surveys are being used to assess the distribution, abundance,

<sup>&</sup>lt;sup>13</sup> Based on opinions expressed at a March 31, 2004 meeting of local fish experts and individuals involved with the LDW RI. The purpose of this March meeting was to gather additional information for targeted species to select the most appropriate sampling approach and compositing scheme for this fish and crab tissue collection effort.



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and size of crabs that inhabit the LDW. As part of each quarterly survey, crab traps were deployed at 38 stations throughout the LDW and allowed to soak for four hours before retrieval. Results from the quarterly surveys are summarized in Tables 3-1 and 3-2. Based on these data, the majority of crabs in the LDW are slender crabs. Abundance of all species is greatest in the downstream, more saline portion of the LDW with total abundance decreasing with distance upstream. The results of the 2003-2004 surveys (Table 3-2) suggest that obtaining sufficient numbers of crabs to meet data needs in the upstream portions of the LDW may not be possible regardless of the season.

Table 5-1. 2005-2004 qualities crab survey results for the LDW										
	SLEN (Cano	NDER CRAB cer gracilis)		Red (Cano	ROCK CRAB	s)	Dung (Can	ENESS CRAI	B 1)	
<b>S</b> URVEY PERIOD	# INDIVIDUALS COLLECTED	LENGTH RANGE (mm)	MEAN LENGTH (mm)	# INDIVIDUALS COLLECTED	LENGTH RANGE (mm)	MEAN LENGTH (mm)	# INDIVIDUALS COLLECTED	Length Range (mm)		
Sep 8-11	294	60-115	92	24	113-184	140	56	87-185	Γ	
Nov 20-23	308	29-134	80	14	120-180	155	39	102-189	Γ	
Feb 16-19	311	55-133	88	2	147-152	150	66	93-172	Γ	

#### Table 3-1. 2003-2004 quarterly crab survey results for the LDW

90

## Table 3-2. 2003-2004 crab survey results relative to fish and crab tissue sampling areas

128-168

150

65

15

SAMPLING AREA	NUMBER OF STATIONS <sup>a</sup>	SLENDER CRAB	RED ROCK CRAB	DUNGENESS CRAB
Downstream of Area 1	3	121	29	43
1	11	460	19	91
Between 1 and 2	4	222	3	15
2	7	252	4	3
Between 2 and 3	4	60	0	11
3	3	53	0	49
Between 3 and 4	1	2	0	11
4	4	1	0	3
Upstream of Area 4	1	0	0	0

10-110

May 24-27

240

<sup>a</sup> Each station consists of one shrimp trap and one crab trap deployed during four quarterly surveys (September and November 2003, February and May 2004). All three crab species were collected in crab traps; some slender crabs were also collected in shrimp traps.

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MEAN LENGTH (mm) 149

> 136 142

152

120-190

Dungeness crabs and red rock crabs may be commonly consumed by people, as evidenced by their presence on the fish advisory recently posted at several LDW fishing locations by the Washington State Department of Health. Slender crabs are less likely to be consumed by people (they were not included on the fish advisory poster), although some Asian anglers may target this species (Frame 2001). River otters also consume crabs, although fish constitute a much larger proportion of their diet, according to a study conducted in southeastern Alaska (Larsen 1984). Of the crab species consumed, Dungeness and helmet crabs were the most frequently consumed in the Larsen study (1984). Heron and sculpin may also consume smaller crabs (Terres 1987). Thus, Dungeness crabs will be targeted for collection. If a sufficient number of Dungeness crabs cannot be collected in a sampling area, the first and second alternative species will be slender and red rock crabs, respectively, based primarily on their abundance in the LDW (Table 3-2).

## 3.1.3 Composite samples per area

As presented in the Phase 2 work plan (Windward 2004e), the goal of the collection effort is to collect and analyze six composite tissue samples (one from each subarea<sup>14</sup>) of each species from each of the four sampling areas. This section discusses the rationale for this number. Composite samples of more than one organism will be analyzed rather than individual organisms because the objective of the fish and crab sampling is to characterize the concentrations of chemicals in representative subpopulations of these species in the LDW. Analyzing a greater number of fish and crabs will then result in a better estimate of the chemical concentrations in each population. These data will then be used to estimate exposure of humans, fish, and wildlife over time through the consumption of these species throughout the LDW.

Alternatively, data from individual organisms provide a better measurement of maximum chemical concentrations. Data on maximum chemical concentrations in individual fish or crabs might be useful for estimating risk from chemicals with acute health effects, but the chemicals associated with the highest risk estimates for the Phase 1 HHRA are all carcinogens, for which average concentrations over a long period of time are more appropriate. Data from individual organisms may also provide a better estimate of the intra-species variability for a particular chemical of concern, which could be useful, but not necessary, if probabilistic risk assessments are performed. Various statistical techniques and assumptions can also provide the needed information on variability using the dataset as a whole.

Theoretically, the requisite number of composite tissue samples per area could be estimated based on the statistical power needed to determine significant differences in chemical concentrations between areas and the minimum number of samples needed to

<sup>&</sup>lt;sup>14</sup> More than one composite sample of perch and English sole each may be collected from some of the subareas in Area 4 because trawling is not possible upstream of the footbridge at RM 4.8, and seining in subarea 4E in August did not yield any perch or English sole for chemical analyses.



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calculate a 95% UCL.<sup>15</sup> However, a robust power analysis was not possible given the relatively small number of existing data and the difficulty in establishing a meaningful target for the minimum detectable difference between areas. Consequently, the number of composite samples per area was set at six for fish whole-body and crab edible meat samples (Table 3-3) to match the site-wide study design assumptions for calculating a 95% UCL for risk assessment purposes. Two composite crab hepatopancreas samples and two composite skin-off English sole fillet samples per sampling area will also be collected. Two hepatopancreas samples are sufficient because this sample type plays only a minor role in the risk assessments. Fewer English sole fillet samples are needed because historical fillet data are available (see Table 3-3), and they are not needed for the sediment-to-biota food-web model, which will be conducted with whole-body fish. Fillets of Pacific staghorn sculpin will not be analyzed because sculpin are not known to be consumed by humans. Fillets of shiner surfperch will not be collected because shiner surfperch are too small to be filleted by anglers.

<sup>&</sup>lt;sup>15</sup> Calculating the 95% upper confidence limit (UCL) on the mean is most effectively done with at least six composite samples, depending on the among-sample variability in concentrations. The threshold of six samples was empirically derived during the Phase 1 HHRA. All 15 EPCs with at least six detected concentrations were based on the 95% UCL. For sample numbers of five and below, the EPCs were based on the maximum concentration because the among-sample variability for several chemicals was too high to calculate a 95% UCL that was lower than the maximum. With a limited number of samples, the 95% UCL on the mean may be higher than the maximum concentration, in which case the maximum is used (EPA 1992).



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		TARGET	TARGET NUMBER OF FISH OR	TARGET NUMBER OF FISH OR CRAB	Total	NUMBER OF COMPOSITE SAMPLES, BY AREA				
SPECIES	SAMPLE Type	SIZE <sup>a</sup> (cm)	CRAB PER COMPOSITE <sup>b</sup>		NUMBER OF		2	3	4	Notes
English sole	whole body	≥ 20	5	30	120	6	6	6	6	Starry flounder (>20 cm) will be collected during both the August and September sampling events as a potential surrogate to be analyzed for areas where a sufficient number of English sole cannot be caught
	fillet	≥ 20	5	10	40	2	2	2	2	Two composite samples per area for fillets are sufficient to supplement existing data (15 existing skin-off fillet samples; 12 from Area 1 and one each from Areas 2-4) and because fillet data will not be used in the food-web model; all samples to be skin-on fillets
Sculpin	whole body	≥ 12	5	30	120	6	6	6	6	Although sculpin larger than 12 cm are targeted, any sculpin larger than 10 cm collected will be archived. Sculpin caught during both August and September sampling events will be archived.
Shiner surfperch <sup>c</sup>	whole body	≥ 8	5	30	120	6	6	6	6	If a sufficient number of adult striped or pile perch are caught while sampling for other fish species, fillets from these fish will be composited in consultation with EPA and Ecology, and may be chemically analyzed. Perch caught during both the August and September sampling events will be archived.
Crab	edible meat	≥ 9	5 <sup>c,d</sup>	30	120	6	6	6	6	Crabs may not be abundant in Area 4
	hepato- pancreas	≥ 9	5 <sup>c,d</sup>	10	40	2	2	2	2	Two samples per area are sufficient because this sample type plays only a minor role in the risk assessments; crabs may be difficult to collect in Area 4

#### Table 3-3. LDW tissue sampling design for fish and crabs

Area 1 centered at RM 0.6, Area 2 centered at RM 2.0, Area 3 centered at RM 3.3, Area 4 centered at RM 4.6

If caught, adult rockfish will be archived for potential chemical analyses as discussed in the rockfish technical memorandum (Windward 2004g). All target and alternate fish collected will be archived until the composite samples are created in consultation with EPA and Ecology. At that time, any remaining homogenates from individual fish or crabs or from composite tissue samples will be archived; whole fish and crabs not homogenized will be archived for this project only until the data validation is approved by EPA. At that time, whole fish and crabs that had been archived frozen will be discarded. Homogenized fish and crab tissue samples will be archived for one year from collection.

<sup>a</sup> Total length

<sup>b</sup> Actual numbers of fish per composite sample and numbers of fish per sampling area will depend on the number of fish caught, as described in Section 3.2.2 for each species

<sup>c</sup> Fillets of shiner surfperch will not be analyzed because fish of this small size are not likely to be filleted prior to human consumption

<sup>d</sup> When possible, the same individuals will be used for edible meat and hepatopancreas samples

<sup>e</sup> Assuming 15 g hepatopancreas tissue and 48 g edible meat per crab

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## 3.1.4 Compositing scheme

The final number of fish or crabs per composite sample will be determined in consultation with EPA and Ecology after all fish and crabs have been collected. Target numbers of fish or crabs per composite sample are established in this QAPP to determine the level of effort necessary for sampling.

Five organisms per composite sample will be targeted for all tissues, except crab hepatopancreas (Table 3-3). The target for crab hepatopancreas composite sample collection is to obtain sufficient mass to meet analytical goals as described below (estimated at approximately five crabs). These targets are based on the expected feasibility of collecting each target species in each area using a reasonable level of effort.

If possible without expanding the level of effort described in Section 3.2.2, greater numbers of fish and crabs than the target numbers will be collected for possible inclusion in each composite tissue sample, up to a maximum of 20 individuals. For a given tissue type, the same number of fish or crabs will be included, if possible, in all composite samples from all sampling areas to provide the most appropriate statistical calculation of the 95% UCL using data from all sampling areas. Therefore, the number of organisms captured in areas of lower abundance may set the number of organisms composite at other sampling locations. The final number of fish per composite will depend on the overall catch and will be determined in consultation with EPA and Ecology.

If a sufficient number of English sole cannot be collected, starry flounder will be collected as a surrogate. If collected, starry flounder and English sole will be counted and composited separately. If sufficient numbers of English sole are subsequently collected, starry flounder will be disposed of.

For crab hepatopancreas samples, the number of crabs per composite sample will be based on collecting sufficient numbers to obtain the 73 g of tissue necessary for analytical tissue mass requirements (Section 3.4.3.6). Edible meat and hepatopancreas tissue mass of a 16.6 cm<sup>16</sup> Dungeness crabs dissected at Windward<sup>17</sup> were 158 g and 49 g, respectively. Based on width/mass relationship data for crabs presented in Atar and Secer (2003), edible meat mass for a 9-cm crab should be about 50 g and hepatopancreas mass should be about 15 g, assuming a constant relationship among the weights of edible meat, hepatopancreas, and whole body. Based on these data, five crabs per composite sample (with 15 g of hepatopancreas tissue per crab) will be necessary to obtain the 73 g of hepatopancreas tissue necessary for analytical goals. The actual number of crabs per composite sample will be based on measured edible meat and hepatopancreas weights from crabs collected in this sampling effort. Extra crabs

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<sup>&</sup>lt;sup>16</sup> Maximum width of the shell from tip of spine to tip of spine.

<sup>&</sup>lt;sup>17</sup> A live Dungeness crab was purchased and dissected at Windward to determine the relative weights of edible meat and hepatopancreas.

will be retained if collected (see Section 3.2.2.4) in case edible meat or hepatopancreas weights per crab are less than expected.

To determine which fish or crabs of a given species from a given sampling area will be composited together, consideration will be given to length, mass, and gender, and the subarea within the sampling area where they were captured. Using this approach, the concentration variance for a given chemical among samples from a given sampling area should be minimized, and will result in a reasonable estimate of the true population mean concentration for each sampling area and the LDW as a whole. As feasible for each target organism, a single composite sample will be created from within each of the six subareas within each sampling area. Decisions on how to account for varying organism sizes or gender ratios within a given area will be made in consultation with EPA and Ecology.

## 3.1.5 Targeted organism size

This section presents the size of organisms targeted to meet the data needs outlined in Section 2.2. Target sizes were selected to represent the preferred prey size ranges of piscivorous wildlife ROCs (Table 3-4), and reasonable size ranges of seafood consumed by humans. Although smaller fish are generally not targeted by wildlife ROCs or humans, they may be targeted by piscivorous fish. Target fish smaller than the targeted size ranges are likely to spend more time in shallow water than the targeted sizes (Eschmeyer et al. 1983; Jones 1962; Palsson 2004), and thus may have somewhat different exposure to sediment-associated chemicals than the targeted fish. Thus, the selected size ranges may result in some uncertainty in tissue concentrations in smaller fish below the target range. However, for many chemicals, concentrations in tissue increase with size or age of fish (e.g., PCBs [Huestis et al. 1997]; mercury [Kohler et al. 1990]), suggesting that the targeted size ranges will provide sufficient data for healthprotective estimates of risk from consumption of all sizes of fish.

PISCIVOROUS WILDLIFE ROCS	PREY SIZE RANGE (CM)	Reference
Great blue heron	8-33	Alexander (1977);Hoffman (1978); Kirkpatrick (1940)
Osprey	11-30	(Van Daele and Van Daele 1982)
River otter	8-41	EPA (1993); Gilbert and Nancekivell (1982); Greer (1955)
Harbor seal	4-28	(Brown and Mate 1983)

Table 3-4. Fiscivolous whunte prey size langes	Table 3	-4. Pisc	vorous	wildlife	prey	size	ranges
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The target size range for English sole is 20 cm (total length) or larger because fish of this size are likely to be targeted for human consumption. In addition, more than 70% of English sole captured in LDW Puget Sound Ambient Monitoring Program (PSAMP) trawls were 20 cm or larger (West 2001), and fish of this size are also generally within the targeted size range of piscivorous wildlife (Table 3-4). The target size for starry flounder, a potential surrogate for English sole, is also 20 cm total length or larger.

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Pacific staghorn sculpin are the representative higher-trophic-level fish that consume fish as well as invertebrates; however, few data are available to indicate length or age at which Pacific staghorn sculpin are capable of piscivory. The targeted size range for Pacific staghorn sculpin is 12 cm total length or larger,<sup>18</sup> as discussed below. Pacific staghorn sculpin mature at one year of age, with length at maturity approximately 12 cm (Emmett et al. 1991). Weitkamp and Campbell (1980) classified LDW sculpin with lengths above 15 cm as "large," and it appears that they include sculpin of this size as potential predators of juvenile salmon. For the purposes of this QAPP, it is assumed that sculpin 12 cm or larger are high-trophic-status fish that may prey on other fish.

The targeted size range for shiner surfperch is 8 cm total length or larger. Weitkamp and Campbell (1980) identified 8 to 14-cm shiner surfperch captured in the LDW as adult fish. Fish of this size are more likely to be targeted for consumption by piscivorous wildlife (Table 3-4) and humans because of their larger size.

The targeted size range for crabs is 9-cm total length or larger. Collecting crabs of this size range maximizes the likelihood of collecting sufficient numbers of crabs for chemical analyses. All but one<sup>19</sup> Dungeness and red rock crab captured in the September 2003 sampling event were approximately 9 cm or larger. In contrast, 40 percent of the slender crabs (the most abundant crabs in the LDW) captured were less than 9 cm. Thus, targeting crabs larger than 9 cm will balance the desire for crabs large enough to be consumed by humans yet of sufficient number to meet the mass requirements for chemical analyses. Additionally, crabs in this size range are mostly adults that have likely been exposed to LDW sediments for a longer period of time compared to younger crabs. Lengths of Dungeness crabs and red rock crabs at maturity are 10 to 12 cm and 4 to 5 cm, respectively [Butler (1961), as cited in Pauley (1988); O'Clair and O'Clair (1998)]. The length of slender crabs at maturity is unknown, but slender crabs are generally smaller than the other two species, so length at maturity is also likely to be less.

### 3.1.6 Data sufficiency

This section describes how data collected according to this QAPP, along with historical data, meet the QAPP objectives presented in Section 2.2.

The first two QAPP objectives are to collect relevant data to estimate risks based on the critical tissue residue approach and the dietary approach. To meet these data needs, fish and crab samples were collected to provide:

- spatial representation within the LDW
- representation of the target species' life history in the LDW (i.e., size of organisms and habitats from which they were collected)

<sup>&</sup>lt;sup>18</sup> Sculpin equal to or larger than 10 cm will also be retained as a contingency.<sup>19</sup> One Dungeness crab was 87 mm.



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• availability of a sufficient number of samples of each tissue type to calculate an EPC for each chemical of interest

Spatial representation of the LDW is addressed through the collection of samples from four sampling areas distributed across the LDW. These areas were selected based on a rolling average concentration of PCBs in sediment (see Section 3.1.1) to ensure that samples were collected within areas representative of low, medium, and higher PCB concentrations in sediment.

Life history of the target species may affect chemical exposure through the dietary items consumed, the locations foraged, and the duration of exposure. Fish and crab will be collected using a variety of sampling techniques from locations covering a range of habitats within the LDW. Also, the size range of the target organisms was selected to represent preferred prey sizes of fish and crab consumers.

To calculate EPCs, this data set will provide 24 independent composite samples for each whole body tissue type, and 8 independent composite samples for fish fillet and crab hepatopancreas tissue types, both of which exceed the six samples desirable to calculate a 95% UCL concentration for each chemical and tissue type for the site (see Section 3.1.3). The use of historical data will further increase the number of available samples to calculate EPCs.

The third QAPP objective is to collect sufficient fish and crab tissue data to calibrate the food-web model. The sampling design specifies the collection of 24 samples for each target species from 23 spatially distinct sampling areas<sup>20</sup> distributed across the LDW. Sampling locations were selected to represent the range of sediment PCB concentrations throughout the LDW and to provide good coverage of the LDW as a whole; thus, a large amount of spatially specific crab and fish tissue data will be available for use in the food-web model. These data will allow for analysis of variation in chemical body burdens for each species within each sampling area and across the LDW as a whole. A technical memorandum will be submitted in March 2005 with additional details of the food-web modeling to be conducted, including a discussion of data sufficiency for each input parameter.

The fourth QAPP objective is to collect PCB congener-specific tissue chemistry data for assessment of PCB risks to wildlife and humans. A tiered approach is specified in this QAPP to collect these data. In the initial round of tissue analyses, one third of all tissue samples will be analyzed for PCB congeners, resulting in a total of eight samples of each sample type (2 samples per sampling area), except crab hepatopancreas (four samples total will be analyzed initially). These sample numbers exceed the six samples desirable to calculate a 95<sup>th</sup> UCL for each chemical for the site (see Section 3.1.3). Four hepatopancreas samples are sufficient because the hepatopancreas is a minor diet component in the overall market basket. Consequently, an EPC based on the maximum concentration rather than the 95<sup>th</sup> UCL should be sufficient for the risk assessment. The

<sup>&</sup>lt;sup>20</sup> Each sampling area has six distinct subareas, except Area 4 which has five.



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samples targeted for PCB congener analysis will be selected to cover the range of total PCBs (an Aroclor sum) and to provide spatial coverage. The relationships among total PCBs (congener sum), dioxin-like PCB congeners (as toxic equivalents [TEQs]), and total PCBs (Aroclor sum) will be assessed to determine the ability of the Aroclor sum to estimate the total PCB concentration in tissue. LDWG will meet with EPA and Ecology to discuss these results. If the Aroclor sum underestimates the total or the relationship between Aroclor and total congener sums is not consistent enough to be useful, and the data suggest that an increased sample size will improve the fit, all of the tissue samples will be analyzed for all 209 PCB congeners, as discussed in Section 3.4.2.

The fifth QAPP objective is to collect fish and crab tissue samples for the analysis of total and inorganic arsenic in specific LDW and background tissue for the Phase 2 HHRA. These data will be used primarily to assess site-related risks from arsenic to human consumers of seafood. Two composite samples of each sample type will be analyzed for inorganic arsenic from each sampling area, resulting in a total of eight samples from the LDW of each sample type. Six to eight samples are regarded as adequate for calculating a 95<sup>th</sup> UCL for the site. Four hepatopancreas samples will also be collected. This sample number is considered sufficient because the hepatopancreas is a minor diet component in the overall market basket. Consequently, an EPC based on the maximum concentration rather than the 95<sup>th</sup> UCL should be sufficient for the risk assessment. Six composite samples of English sole (fillet and whole body), shiner surfperch (whole body), crabs (edible meat), and clams will be collected from two different background locations and analyzed for both total and inorganic arsenic, as described in Appendix E. These data will be used to assess the influence of natural and anthropogenic regional sources of arsenic on tissue concentrations in the LDW.

# 3.2 SAMPLING METHODS

Fish and crabs will be collected from the LDW using a high-rise otter trawl, shrimp and crab traps, and beach seine. Selected methods for each target organism, specific information on each sampling method, sample numbering, field processing methods, sample packaging, and decontamination procedures are discussed in this section.

There may be contingencies during field activities that require modification of the general procedures outlined below. Modification of procedures will be at the discretion of the FC after consultation with the Windward TM and PM, the boat captain, and the EPA or Ecology representative in the field, if applicable. LDWG, EPA, and Ecology will be consulted if significant deviations from the sampling design are required. All modifications will be recorded in the protocol modification form.

# 3.2.1 Sample identification

Unique alphanumeric identification (ID) numbers will be assigned to each individually wrapped fish or crab specimen in the field and recorded on the target fish and crab species form. Organisms other than the targeted fish species will be recorded on the non-target species collection form, but no specimen ID will be assigned. The first three

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characters will be LDW<sup>21</sup> to identify the project area. The next two characters will identify the specific tissue sampling area: T1, T2, T3, or T4. The next character will identify the specific sampling subarea: A, B, C, D, E, F. The next five characters will identify the collection method and effort number: TR, SN, CT, ST, representing trawl, beach seine, crab trap, and shrimp trap, respectively followed by a three-digit number representing the effort number (numbered sequentially over all areas) (e.g., the 15th trawl after the start of sampling would be TR015). The next two characters will identify the individual species type: PS, ES, SS, SP, PP, RF, DC, SC, RC, representing Pacific staghorn sculpin, English sole, shiner surfperch, striped perch, pile perch, rockfish, Dungeness crab, slender crab, and red rock crab, respectively. The alternative target species, starry flounder, will be identified with SF. The final identifier will be numeric and indicate the sequential number of the specimen captured for a given tissue sampling area. As an example, the 11th sculpin captured in Area 1, subarea C, in the 15<sup>th</sup> shrimp trap retrieved would be identified as LDW-T1-C-ST015-PS-11. All relevant information for each individually wrapped and labeled target specimen, including specimen ID, length, weight, gender (if it can be determined without dissection), external abnormalities, sample date, time, and location number will be recorded on the target fish and crab species collection form (Appendix B) and included as an appendix in the final data report. Therefore, all pertinent data associated with each individual fish or crab specimen can be tracked.

Composite samples will be identified using a similar convention, with the following changes. No indication of collection method or effort number will be indicated because specimens from multiple methods and efforts may be included in each composite. Tissue type will be indicated as WB, FL, EM, or HP for whole body, skin-on fillet, edible meat, or hepatopancreas samples, respectively; each sample for a given species and sampling area combination will be numbered sequentially following the letters "comp." For example, the second whole body composite sculpin sample in Area 1, subarea C, would be identified as LDW-T1-C-PS-WB-comp2. If specimens from multiple subareas are included in the composite sample, the subarea designation would be replaced with an M. For example, the previous composite number would become LDW-T1-M-PS-WB-comp2. Information would be compiled regarding the specific number of fish from each subarea that were composited in each sample.

#### 3.2.2 Sampling methods for each target species

This section provides information on methods for the collection of each target species identified in this QAPP. Preferred and alternative collection methods are presented based on past LDW collection data and general life history information, and are summarized in Table 3-3. The maximum level of effort for sampling all target species (including sampling for surrogate species) is 5 days of sampling targeting shiner perch (August 2-7), plus 10 days of sampling for crabs and 7 days of sampling for fish targeting all species not collected in August (August 30–September 10), plus

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<sup>&</sup>lt;sup>21</sup> The sample identification scheme for background sampling is described in Appendix E.

background sampling in the latter part of September as described in Appendix E. If target numbers of fish and crab for each sampling area are not obtained by this level of effort, numbers of fish and crabs per composite or number of composite samples per area will be adjusted based on consultation with EPA and Ecology.

#### 3.2.2.1 English sole

English sole will be collected using a high-rise otter trawl, as described in Section 3.2.3.1. English sole were common and abundant throughout the LDW in trawl surveys (Malins et al. 1980; Meyer et al. 1981; Miller et al. 1975, 1977; West 2001), but were rarely captured using beach seine or other methods (Matsuda 1981; Warner and Fritz 1995; Weitkamp and Campbell 1980). Consequently, no secondary collection method is proposed for English sole.

Trawl data from Miller et al. (1977) show that English sole tend to be more prevalent in the southern portion of the LDW during late summer and fall. Malins et al. (1982) used trawls at four stations up to RM 4.2. Abundance of English sole appeared to follow the leading edge of the saltwater wedge upriver during low flow; abundance was higher in the lower estuary during high runoff. Similar results were observed by Eaton (2004). Consensus at the March 31, 2004 fish experts meeting was that sufficient English sole to meet data objectives should be obtainable throughout the LDW using trawl sampling if sampling is completed before the fall rains cause a large inflow of fresh water.

English sole will be collected and archived during August sampling for perch (see Section 3.2.2.3). Additional trawling for English sole will be conducted in the September sampling event (August 30-September 10) in areas with less than the target number of English sole. Because English sole are believed to have a home range that is larger than the subareas, priority will be placed on collecting a sufficient total number of English sole in each sampling area. Once that is accomplished, focus will be placed on collecting samples in subareas C and D, and then on collecting a sufficient number in each of the remaining sampling subareas.

Trawling will be discontinued in a given area when the target number (40 fish)<sup>22</sup> of English sole has been collected. If the target number of English sole is obtained in all sampling areas in less than 7 days of sampling in September, the remaining effort, up to 7 days total, will focus on areas where relatively fewer English sole were captured, in an attempt to maintain a balanced number of fish among all sampling areas. All English sole of target size will be retained, with the goal of achieving the requisite number of fish per composite sample (up to 20 fish per composite or 160 fish per area) while keeping the number per composite sample for a single tissue type equal among all sampling areas.

<sup>&</sup>lt;sup>22</sup> Six composite samples of at least five fish each in each area for whole body analyses (ideally one per subarea) and two composite samples of at least five fish each in each area for fillet analyses. Fillet composite samples will be made from subareas where fish are more abundant. If starry flounder are needed because a sufficient number of English sole are not caught, these fish will be composited separately.



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Starry flounder (> 20 cm) collected in the August and September 2004 sampling efforts (as described in Section 3.2.3.3) will be retained and archived as a surrogate species for possible use in the event that an insufficient number of English sole are collected. LDW beach seine data indicate that adult starry flounder are uniformly distributed in the shallow, nearshore environment of the lower LDW (Shannon 2004; Weitkamp and Campbell 1980), suggesting this species is more widely distributed in shallow water compared to the deeper waters in which trawling was typically conducted. However, Miller et al. (1977) reported that large numbers of starry flounder were rarely caught at stations downstream (north) of RM 2.5, and were generally most abundant upstream of RM 2.5.

If a sufficient number of English sole and starry flounder have not been caught in a given sampling area after three trawling days in Areas 1 and 2, two days in Area 3, and one day in Area 4 in September, the channel will be trawled as well as the bench areas. If sufficient numbers are not collected after six days of trawling in September, trawling outside the designated areas will be conducted on the last day of trawling by extending the area both upstream and downstream half way to the adjacent sampling area, after consultation with EPA. Additional trawls will be conducted in these areas according to the process described in Section 3.2.3.1. Target fish collected outside of the original sampling area will be noted as such. Any other target fish species besides English sole (e.g., sculpin) collected outside of the original sampling area will be returned to the LDW if the target numbers for these other species have been met within the designated sampling areas.

#### 3.2.2.2 Pacific staghorn sculpin

The primary collection method for Pacific staghorn sculpin is shrimp traps, although sculpin captured using other methods (trawling or seining) will also be retained and archived. Pacific staghorn sculpin have been frequently caught in the LDW in both beach seines and trawls, but sizes of fish caught in these studies are generally not reported (Miller et al. 1975; Warner and Fritz 1995; Weitkamp and Campbell 1980; West 2001). Consensus among fish experts at the March 31, 2004 fish experts meeting was that trawling is an inconsistent method for capturing piscivorous sculpin<sup>23</sup> (>10 to 12 cm) and few sculpin of this size are captured in beach seines,<sup>24</sup> thus alternative methods are likely to be needed.<sup>25</sup>

Windward incidentally collected sculpin in shrimp traps at 28 of 38 LDW sampling locations as part of the first quarterly LDW crab and shrimp sampling efforts in September 2003 (Windward 2004a). The size ranges of the sculpin collected in the traps

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<sup>&</sup>lt;sup>23</sup> The fish experts at the March 31 meeting agreed that sculpin become piscivorous at 10-12 cm in length.

<sup>&</sup>lt;sup>24</sup> The maximum size of sculpin captured by Taylor Associates in LDW beach seines was 7 cm (Shannon 2004)

<sup>&</sup>lt;sup>25</sup> Any sculpin caught in fish trawls or beach seines in August or September will be archived to supplement fish caught in shrimp traps. Also any sculpin larger than 10 cm will be retained in case a sufficient number of sculpin larger than 12 cm (the target size) are not caught.

are presented in Table 3-5. Trap deployment depths at all stations ranged from -9 to -39 ft mean lower low water (MLLW). Sculpin larger than 12 cm were captured over the full range of depths sampled at locations throughout the LDW. These results suggest that shrimp traps are a suitable alternative sampling method for sculpin.

	LENG	LENGTH OF SCULPIN (cm)						
TRAP DEPTH (ft MLLW)	10 - 15	15 - 20	20+	OF SCULPIN > 10 cm				
-9	2	3	1	6				
-10 to -19	2	21	17	40				
-20 to -29	5	12	2	19				
-30 to -39	6	14	6	26				
unknown depth		5	1	6				
Total	15	55	27	97				

 Table 3-5. Number of sculpin collected in shrimp traps in September 2003

Sculpin (> 10 cm) caught in the August trawling and seining events will be archived. In September, the four sampling areas will be sampled in subareas where less than five sculpin were collected in August until 30 sculpin are collected from each area, ideally five fish from each of the six subareas, up to a maximum of 7 days total for all areas. If the target number of sculpin is obtained in all sampling areas in less than 7 days, the remaining effort, up to 7 days total, will focus on any subareas where relatively few Pacific staghorn sculpin were collected. The intent of this sampling design is to maximize the number of fish per composite (up to 20 fish per composite or 120 fish per area) while keeping the number of fish per composite equal across sampling areas.

No more than 20 sculpin will be collected in any subarea, thus no shrimp traps will be placed in subareas with 20 archived sculpin (>12 cm). Priority will be placed on collecting a sufficient total number of Pacific staghorn sculpin (>12 cm) in each sampling area. Once that is accomplished, focus will be placed on collecting samples in subareas C and D, and then on collecting a sufficient number in each of the remaining sampling subareas.

If a sufficient number of Pacific staghorn sculpin have not been caught in a given sampling area after six total days of sampling, shrimp traps will be placed outside the designated areas on the last day of sampling. If needed, the sampling area will be extended both upstream and downstream half way to the adjacent sampling area, after consultation with EPA. Target fish collected outside of the original sampling area will be noted as such. Target fish species other than Pacific staghorn sculpin collected outside the original sampling area will be returned to the LDW if not needed to meet their respective target numbers, or will be processed if needed. No surrogate species have been selected for sculpin because it is unlikely that target numbers of an alternative higher-trophic-level fish species that is piscivorous (e.g., sand sole) would be caught in sufficient numbers in the LDW.



#### 3.2.2.3 Shiner surfperch

Shiner surfperch will be collected using both high-rise otter trawling and beach seining. Trawling is an effective collection method because adult shiner surfperch prefer sandy bottoms that can be efficiently sampling using a trawl. Historical data show that shiner surfperch are commonly caught in LDW trawl surveys, although season-specific data were not available. Miller et al. (1975) reported that shiner surfperch were caught throughout the LDW based on trawl sampling. Miller et al. (1977) reported that 209 shiner surfperch were caught throughout the LDW and they occurred in 30 to 70% of trawl samples. PSAMP data from the vicinity of Kellogg Island show that shiner surfperch were found on all six dates that trawling took place, but not during all trawls (West 2001). When caught, catch numbers ranged from 2-28 fish per trawl.

Beach seining will also be conducted in August as a means of collecting shiner perch from shallow water where trawling is infeasible. Monthly beach seining data from Taylor Associates show that adult shiner surfperch are generally present in shallow water in the LDW from May through August, but generally are absent in September. Taylor and Associates monthly catch data for 2003 show that adult shiner surfperch were abundant in June and July, present on August 21<sup>st</sup> at both Kellogg Island and Turning Basin 3 stations, but were not present at any location on the September 26<sup>th</sup> sampling date (Shannon 2004). Weitkamp and Campbell (1980) reported that in the vicinity of Kellogg Island, mature adults (8-14 cm) were present in May and June, and young-of-the-year (YOY) fish (3.5-4.5 cm) were present in late July and August, but were absent from all seines in September.

Shiner surfperch will be collected by high-rise otter trawl and beach seine in August, as described in Sections 3.2.3.1 and 3.2.3.3, respectively. Beach seining will take place on August 2 and 3, 2004. Three stations in each sampling area will be seined at least one time each over the two days of sampling. After all stations have been visited once, if there is available time on the second day, sampling areas where relatively fewer fish were obtained will be revisited for an additional sample collection. On the subsequent visit, the same sites or different sites within the sampling area may be visited based on availability of sites and best professional judgment of the FC, in consultation with Taylor Associates personnel.

Trawling for shiner surfperch will take place from August 2 to 6. In this sampling event, priority will be placed on collecting a sufficient total number of shiner surfperch in subareas (5 fish per subarea), especially subareas C and D, then on collecting a sufficient number in each sampling area (30 fish per sampling area). On the first day, Areas 1 and 2 will be trawled (approximately 10 trawls per area with at least one trawl per sampling subarea). On the second day, Areas 3 and 4 will be trawled. If a sufficient number of shiner surfperch are not caught in each area on these first two days (i.e., 30 fish per area), these areas will be trawled first on days 3, 4, and 5. Additional sampling will then focus on subareas of sampling areas where relatively fewer shiner surfperch have been captured in an attempt to keep the number of fish even among all sampling



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subareas. For any given subarea, the maximum daily effort will be five trawls. If the target number of shiner surfperch (30 fish per area) is obtained in all sampling areas in less than 5 days, trawling will continue up to 5 days total to increase the number of fish per composite sample (up to 20 fish per composite or 120 fish per area). Note that in Area 4, only four subareas can be sampled by trawl and five areas can be sampled by beach seine. Thus, one or more of the subareas in Area 4 will likely have more than one composite sample. The compositing scheme for Area 4 will depend on the catch of perch in the area and will be determined in consultation with EPA and Ecology.

Shiner surfperch collected in August will be archived (up to 20 fish per subarea). Shiner surfperch collected in September as part of trawling for English sole will also be archived for potential inclusion in composite samples if they are collected in subareas with fewer than 20 shiner surfperch from the August sampling event.

# 3.2.2.4 Crabs

The primary collection method for crabs is crab traps. However, crabs are also frequently collected in trawl samples (Eaton 2004; West 2001). Any crabs caught during trawl sampling in September will be archived for potential use in composite samples.

Sampling areas will be sampled until 30 crabs<sup>26</sup> are collected from each area, ideally five crabs from each subarea, up to a maximum of 10 days total. Priority will be placed on collecting the target total number of crabs per area. Once that is accomplished, focus will be placed on collecting samples in subareas C and D, and then on collecting a sufficient number in each of the remaining sampling subareas. If the target numbers of crabs are obtained in all sampling areas in less than 10 days, the remaining effort, up to 10 days total, will focus on any subareas where relatively fewer crabs were captured, in an attempt to maintain a balanced number of crabs among all sampling subareas. All crabs of sufficient size will be retained up to 20 crabs per subarea.

It is possible that insufficient numbers of Dungeness crab to meet the sample design may be obtained, based on the low Dungeness crab abundance noted during quarterly sampling (Windward 2004a, c, d). Therefore, slender and red rock crabs will also be retained and archived when caught in traps. Based on the relative abundance of the crab species during the quarterly crab surveys (see Table 3-2), and the preferred substrates of slender crab (sandy/muddy) and red rock crab (rocky), slender crabs will be the primary alternative species and red rock crab will be a secondary alternative species.

If a sufficient number of crabs have not been caught in a given sampling area after seven total days of sampling, sampling outside the designated areas will be conducted the last two days by extending the sampling area both upstream and downstream half way to the adjacent sampling area, after consultation with EPA. Additional traps will be randomly placed outside the original sampling area (see Section 3.2.3.2 for random placement methods). Target crabs collected outside of the original sampling area will

<sup>&</sup>lt;sup>26</sup> Both edible meat and hepatopancreas tissues will be analyzed from the same crabs.



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be noted as such. Any other target fish species besides crab collected outside of the original sampling area will be returned to the LDW if the target numbers for these other species has been met within the designated sampling areas.

#### 3.2.3 Collection methods

Fish and crabs will be captured in the field using three different collection methods: a high-rise otter trawl, shrimp and crab traps, and beach seining. This section describes these methods in greater detail.

# 3.2.3.1 High-rise otter trawl

Trawling methods, described in this section, are based on systematic sampling of the four sampling areas and their subareas. The expected maximum daily effort is approximately 20 trawls depending on site conditions and number of fish processed (Eaton 2004). Trawling will be conducted using the vessel *R/V Kittiwake*, captained by Charlie Eaton of Bio-Marine Enterprises.

The high-rise otter trawl consists of a 25-ft (7.6-m) headrope and 29-ft (8.8-m) footline, side panels with 1.5 in. mesh which open to 5 ft at the wing tips, and 24-in. x 36-in. V-shaped galvanized steel trawl doors. The footline consists of 0.5-in. combination poly/wire with 5.33-oz seine leads interspersed with 2-in. rubber discs, and the headrope has eight 5-in. plastic floats. The 1.25-in. mesh codend also has a knotless nylon codend liner with 0.25-in. mesh.

Areas 1, 2, and 3 (Figure 3-1; oversized figure, Section 7.0) were divided into six subareas (A-F), and sampling area 4 was divided into five subareas (A-E), as shown in Figure 3-1 (oversized figure, Section 7.0). Area 4 was divided into only five subareas because of its shape and the difficulty in sampling upstream of RM 4.8 because of a low footbridge at RM 4.8, and because underwater rocks, root masses, and other debris occur throughout the area upstream of the bridge make trawling infeasible in this area (Eaton 2004). Beach seining is also difficult in this area because of fast water flow through this area and woody debris in the channel (Shannon 2004), but will be attempted in subarea 4E along the eastern shoreline around RM 4.9 as described in Section 5.2.2.2. Fyke nets are not proposed because they would likely capture returning Endangered Species Act-listed adult chinook salmon, and Rob Clapp of the endangered species program at National Marine Fisheries Service advised against proposing the use of these nets (Clapp 2004). Subarea 4E (the area upstream of the footbridge) will be sampled in September for Pacific staghorn sculpin and crabs using traps.

At least one trawl will be conducted within each subarea, as described below. Each trawl line will be conducted within the bounding coordinates of the sampling subareas. Within each subarea, an attempt will be made to conduct all trawls outside of the navigation channel in order to capture fish using shallower habitats, although trawling the channel may be necessary if a sufficient number of fish (e.g., English sole) are not caught in certain sampling areas (e.g., Area 4) (see Section 3.2.2.1). Trawling will not be conducted in waters shallower than 6 ft deep (at the time of trawling), because the

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high-rise otter trawl is impractical in shallower areas (Eaton 2004). If one or more slips are present in a given subarea, at least one trawl will be conducted in each slip if feasible, based on barge locations, etc. The specific trawl line and order in which the subareas will be sampled will be determined by the boat captain based on logistical considerations and priorities discussed Section 3.2.2. Subsequent trawls in each subarea may follow the first trawl line or a different trawl line at the discretion of the boat captain in consultation with the FC.

The trawl will be deployed to the bottom using a winch. When the trawl reaches the bottom, the "dog" of the winch will be set (stopping the release of cable from the winch) and the vessel will begin the trawl. The trawl will progress upstream. The trawl speed will remain constant at 2.5 knots. The spread of the trawl will be approximately 4.7 m, with a rise of approximately 1.5 m. When the vessel reaches the end of each trawl line, the dog of the winch will be released and the trawl will be hauled aboard, allowing the captured species to be processed. The date, time, and location of the trawl will be recorded on the fish and crab tissue collection form (Appendix B) after each trawl is hauled out of the water.

Trawl start and end points will be recorded using a Trimble NT300D differential global positioning system (DGPS) with 1-2 m accuracy. When the trawl is deployed on the bottom, GPS and clock readings will be taken to mark the starting point of the trawl. Final GPS and clock readings will be made when net retrieval begins.

Trawling will be conducted from aboard the *R/V Kittiwake* using a live sampling technique, which will minimize the number of non-target species mortalities through species sorting and processing prioritization. Upon completion of an individual trawl, the catch will be hauled aboard and immediately emptied into a large plastic tub filled with running seawater. Field technicians will sort the catch by species and size into numerous smaller tubs, also containing running seawater. Target species will be separated from non-target species and processed as described in Section 3.2.4. Non-target species will be identified to the lowest practical taxon and their numbers estimated. For target species, any prey in the fish's mouth will be assumed to have been consumed in the trawl and will be removed from the fish's mouth before processing.

The order that sampling areas and subareas will be trawled over the course of the project and within a given day will be determined by both the FC and the trawl boat captain following the priorities presented in this section and in Section 3.2.2. Leaving this decision to the discretion of the field personnel maximizes their ability to respond to field conditions and exercise their professional judgment on fishing conditions. The trawl results will be reported each day to the Windward TM and PM, who will provide input on priorities for the subsequent day's sampling effort.

#### 3.2.3.2 Traps

Twelve shrimp traps (deployed to catch sculpin), and twelve crab traps (deployed to catch crabs) will be deployed side by side at maximally dispersed locations outside of

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the navigation channel within a sampling area, two per subarea, for a given day.<sup>27</sup> Traps will be placed in locations where they will not interfere with vessel navigation and will remain covered by water during the entire time they are in the water.<sup>28</sup> Twelve traps per day is a feasible daily effort, based on Windward's experience from quarterly crab and shrimp sampling. Fewer or more traps may be set in a given sampling area depending on success in the first few days of sampling. Only one sampling area will be sampled each day.

Traps will be deployed in a different sampling area on each day until target numbers of each target species are obtained or the maximum level of effort for the project is reached (see Section 3.2.2). The specific area to be sampled will be based primarily on sampling logistics related to the trawl sampling being conducted simultaneously. If target numbers are met in a given sampling area, sampling will be focused on the remaining areas until targets are met for all areas or the maximum level of effort is met.

Shrimp and crab trap sample locations will be recorded using a Magellan SporTrak GPS unit, upgraded to include the latest Wide Area Augmentation System (WAAS) technology, providing accuracy within 3 m. Coordinates will be taken at the deployment location for each trap type. The FC will ensure that specimens are collected within the specified tissue sampling areas (Figure 3-1; oversized figure, Section 7.0). Washington State Plane coordinates North (NAD 83) will be used for the horizontal datum.

Sculpin will be collected using Ladner 30-in. nestable shrimp traps with 0.5-in. mesh. Crabs will be collected using Ladner 30-in. stainless-steel rubber-wrapped crab traps. One trap of each type will be deployed on separate floats at the chosen sampling location (see above). Traps used to capture sculpin will be baited with a mixture of slow- and fast-smolting (dissolving) shrimp pellet bait, or another bait mutually agreed upon by LDWG, EPA, and Ecology. The bait will be placed in 1-quart plastic Scotty brand bait jars with approximately forty 8-mm holes, thus allowing the scent of the bait to spread without allowing access to the bait itself. Crab traps will be baited with a mixture of fish scraps and squid. Crab bait will be placed in mesh bait bags and tied to the inside of the trap so the bag cannot be opened and its contents consumed. All traps will soak for approximately two hours<sup>29</sup> before retrieval. Traps for sculpin will be hauled in first and at a constant rate to prevent the possible escape of any captured fish. All traps will be retrieved in the same order as they were deployed. The field crew will monitor the traps, to the extent possible, when fishing in areas of high vessel traffic. Any trap(s) determined by the FC to be a hazard to navigation will be moved to a new

<sup>&</sup>lt;sup>29</sup> The quarterly crab/shrimp surveys used a 4-hr soak time as a standard for assessing relative abundance at different locations, but a 2-hr soak time should be sufficient to capture the target specimens and still provide the field crew with enough flexibility for multiple deployments of the same trap during a single day



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<sup>&</sup>lt;sup>27</sup> No shrimp traps will be placed in subareas where 20 sculpin were collected in August.

<sup>&</sup>lt;sup>28</sup> Traps will generally be placed in locations deeper than -2 ft MLLW; however, traps may be placed in shallower water if high tides coincide with sampling.

location within the same sampling subarea away from impending vessel traffic. Any traps lost during sampling will be replaced, and all traps will be outfitted with a degradable latch to ensure that escape holes will open if the trap is lost. The degradable latch will ensure that lost traps will not continue to fish indefinitely, thereby harming local crab, shrimp, or fish. The date, time, and location of the trap will be recorded during both trap deployment and retrieval.

During the retrieval phase, captured organisms will be sorted by species into decontaminated bins filled with LDW water. All non-target species will be identified to the lowest practical taxon and their number estimated. For target species, any prey in the fish's mouth will be assumed to have been consumed in the trap and will be removed from the fish's mouth before processing. More sensitive species and life stages (i.e., juvenile salmonids, Pacific herring, smelt, juvenile tomcod) will be handled minimally and returned to the water as quickly as possible.

#### 3.2.3.3 Beach seine

Beach seining will be one of the collection methods used in August for shiner surfperch. Jim Shannon of Taylor Associates will be responsible for beach seining. Beach seines will be deployed from shallow low gradient beaches within each sampling area. Potential beach seining locations identified by NOAA are shown in Figure 3-1 (Field 2004). The particular sites sampled will be at the discretion of the FC in coordination with Taylor Associates personnel.

Three sites will be sampled in each sampling area; sites will be dispersed throughout the sampling areas based on suitable locations, except in Area 4. Based on an initial assessment of available seining sites, the following subareas will be targeted: 1B, 1D, and 1E; 3 sets in 2B; and 3A, 3D, and 3F. In Area 4, priority for seining will be given to subarea 4E. If seining is feasible and fish are caught, the remaining seining will be conducted in subarea 4E (because trawling is not possible in this subarea). If no fish are collected or the net is getting caught in debris, the remaining seining in Area 4 will be conducted in subareas 4A, 4B, and 4D.

Beach seine sample locations will be recorded using a Magellan SporTrak GPS unit, upgraded to include the latest Wide Area Augmentation System (WAAS) technology, providing accuracy within 3 m. Coordinates will be taken at the starting location of each beach seine deployment. Locations of beach seining activities will also be identified by reference to landmarks. Field technicians will note place names or approximate distances to nearby landmarks and photo-document the seining locations. The FC will ensure that specimens are collected within the specified tissue sampling areas (Figure 3-1; oversized figure, Section 7.0). Washington State Plane coordinates North (NAD 83) will be used for the horizontal datum.

The standard beach seine will measure 37 m long and 3 m deep, with 6-mm mesh in the wings and 5-mm mesh in the center bag. The seine will be equipped with floats to minimize snagging of the lead line on submerged pilings, riprap, and other debris, and

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30-m ropes to haul the net to shore. The beach seine will be cleaned of all debris before being deployed. The net will be deployed 30 m from shore and parallel to the beach using an outboard-powered boat and three or four field technicians. One or two technicians will stand on shore holding the 30-m rope attached to one end of the net until the reversing boat pulls the rope taut. Once the rope is taut, another technician will feed the net from the bow of the boat into the water as the skipper slowly motors in reverse to lay out all the net parallel to shore. The rope on the opposite end of the net will then be motored to shore, and the person who was in the bow of the boat deploying the net will jump ashore with the rope end to assist with retrieving the net. Teams of one or two technicians will then stand at each end of the net, approximately 40 m apart, to pull the net toward shore at a steady rate. When the net is approximately 10 m from shore, the two teams will move together until they are about 10 m apart for the final hauling of the net up onto the shore.

Prior to each beach seine deployment, the location, time of day, and weather conditions will be recorded. Upon beach seine retrieval, target species will be sorted from non-target species and retained in decontaminated bins with LDW water. In addition to shiner surfperch, any suitable English sole, starry flounder, or Pacific staghorn sculpin collected in beach seines will be processed and archived. For target species, any prey in the fish's mouth will be assumed to have been consumed in the seine and will be removed from the fish's mouth before processing.

#### 3.2.4 Field sample processing

All species captured using the methods outlined above will be placed in decontaminated bins filled with LDW water. Target fish and crabs of similar size will be preferentially selected and sorted. Specimens of target species that do not meet size requirements will be counted, measured to the nearest 1 mm, and returned to the LDW. Specimens of non-target species will be identified to the lowest practical taxon and their numbers estimated. Special care will be taken to ensure that non-target organisms are returned to the LDW quickly, with minimal handling.

# 3.2.4.1 Fish

Individual fish of the selected target species will be rinsed in LDW water to remove any foreign material from the external surface. Large target fish will be killed using methods outlined in EPA (2000), by a sharp blow to the base of the skull with a wooden club or metal rod. This club or rod will be used solely for the purpose of killing fish, and care will be taken to keep it reasonably clean to prevent contamination of the samples. Small fish will be killed by placing them on ice, as recommended by EPA (2000). Individual specimens of the target species will be grouped by species and general size class, and placed in clean holding trays to prevent contamination. All fish will be inspected carefully to ensure that their skin has not been damaged by the sampling equipment. The FC will discard specimens with broken skin. Each fish within the selected target species will be measured to determine total length (nearest mm) and



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If fish weights are to be measured in the field, fish will be weighed using a handheld scale suited for the weight of the species (Pesola® 100 g x 1 g, Pesola® 300 g x 2 g, and Pesola® 1000 g x 10 g). To be consistent with the convention used by most fisheries biologists in the United States, total length will be measured as the length from the anterior-most part of the fish to the tip of the longest caudal fin ray (when the lobes of the caudal fin are compressed dorsoventrally) (Anderson and Gutreuter 1983). Additional observations of fish collected for chemical analysis will include the determination of gender when distinct visual differences are discernable between sexes (e.g., gravid females), as well as general observations of individual specimen health, such as any visible signs of morphological abnormalities, external lesions, parasites, or fin erosion. If time allows, photographs of external abnormalities will also be taken. If sampling conditions do not allow adequate time for sample processing in the field, individual specimens of the same species from a particular sampling area and gear deployment (i.e., a single trawl, seine, or trap) will be kept together in one large resealable plastic bag with the date, time, effort number, species, and collection method recorded on the outside in indelible ink. All other pertinent information will be traceable through the field notebook and collection forms (Appendix B). The bagged and iced fish will be transported in coolers to Windward for final processing. Fillets will be prepared in the laboratory, not in the field.

# 3.2.4.2 Crabs

Crabs will be inspected to ensure that their exoskeletons have not been cracked or damaged during the sampling process; damaged crabs will be discarded (EPA 2000). After crab traps have been retrieved, captured crabs will be rinsed with LDW water, and individual specimens will be grouped by target species and placed in clean holding trays to prevent contamination. Target crab specimens will be identified to species, measured to the nearest 1 mm, and weighed to nearest 0.5 g. Crabs may be weighed and measured in the field or in the Windward laboratory at the discretion of the FC. Prior to processing, crabs will be placed on dry ice. Dry ice will be used rather than water ice because it is a more humane way of killing the crabs. Crab carapace width measurements will be obtained using stainless-steel calipers and a measuring board, respectively. Crabs will be weighed using a handheld scale suited for the weight of the species (Pesola® 100 g x 1 g, Pesola® 300 g x 2 g, and Pesola® 1000 g x 10 g). In keeping with EPA guidance, crab carapace width measurements will be made laterally across the carapace from tip of spine to tip of spine (EPA 2000). If sampling conditions do not allow adequate time for sample processing in the field, individual specimens of the same species from a particular sampling area and gear deployment (i.e., a single trap) will be kept together in one large resealable plastic bag with the date, time, effort number, species, and collection method recorded on the outside in indelible ink. All other pertinent information will be traceable through the field notebook and collection forms (Appendix B). The bagged and iced crabs will be transported in coolers to

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Windward for final processing. The edible meat and hepatopancreas will be removed from the crabs in the laboratory, not in the field.

#### 3.2.5 Field equipment

The items needed in the field for each sampling method are identified in Table 3-6. The FC will check that all equipment is included and in working order each day before sampling personnel go in the field. A rugged laptop computer complete with navigation software will accompany the FC at all times.

	TISSUE COLLECTION METHOD				
NECESSARY FIELD EQUIPMENT	High-Rise Otter Trawl	CRAB/SHRIMP TRAPS	BEACH SEINE		
QAPP	Х	X	Х		
Key personnel contact information list	Х	X	Х		
Field sample collection forms	X	Х	Х		
Field notebooks (Rite in the Rain <sup>®</sup> )	X	Х	Х		
Chain-of-custody forms	Х	Х	Х		
Pens, pencils, Sharpies	Х	Х	Х		
Tide tables	Х	Х	Х		
Study area maps	X	Х	Х		
Fish identification guides	Х	Х	Х		
GPS (w/ extra batteries)	Х	Х	Х		
Digital camera	Х	Х	Х		
Cellular phone	Х	Х	Х		
Marine radio	Х	Х	Х		
Alconox <sup>®</sup> detergent	Х	Х	Х		
Distilled water	Х	Х	Х		
Garden sprayer (for distilled water)	X	X	Х		
Scrub brushes	X	Х	Х		
Paper towels	Х	Х	Х		
Garbage bags	X	Х	Х		
Buckets (5 and 2 gallon)	X	X	Х		
Coolers	X	X	Х		
Ice (wet and/or dry)	Х	Х	Х		
Heavy duty aluminum foil	X	X	Х		
Ziploc <sup>®</sup> freezer bags (quart and gallon size for individual fish/crabs)	X	X	Х		
Ziploc <sup>®</sup> freezer bags (larger size)	X	X			
Ziploc <sup>®</sup> sandwich bags (for individual sample labels)	X	X	Х		
Plastic bins for specimen sorting	X	X	Х		
Dip nets	X		Х		
Calipers	X	Х	Х		
Measuring boards	Х	X	Х		
Scales	Х	X	Х		
Crab/shrimp traps (complete with floats, line, bait bags/jars, and weights)		Х			

Table 3-6. Fish and crab tissue collection field equipment

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	TISSUE COLLECTION METHOD				
NECESSARY FIELD EQUIPMENT	High-Rise Otter Trawl	CRAB/SHRIMP TRAPS	BEACH SEINE		
Bait for crab/fish traps		Х			
Pike pole (for dislodging nets hung on underwater debris and trap retrieval)	X	Х	Х		
High-rise otter trawl	X				
Beach seine		Х	Х		
Cutting board		Х			
Knife		Х			
Powder-free nitrile exam gloves	X	Х	Х		
Rubber work gloves	X	Х	Х		
Rubber boots	X	Х	Х		
Raingear	X	Х	Х		
Waders		Х	Х		
Personal flotation devices	X	Х	Х		
Hard hats	X				
Anchor			Х		
Head lamps	X	Х	Х		
First aid kit	X	X	Х		
Duct tape	X	Х	Х		
Cable ties	Х	Х	Х		

# 3.3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

This section describes how individual samples will be processed, labeled, tracked, stored, and transported to the laboratory for analysis. In addition, this section describes decontamination procedures, disposal of field generated waste, sample custody procedures, and shipping requirements. Sample custody is a critical aspect of environmental investigations. Sample possession and handling must be traceable from the time of sample collection, through laboratory and data analyses, to delivery of the sample results to the recipient.

#### 3.3.1 Sample handling procedures

Fish and crab processing will be conducted either in the field or at Windward. Field processing is described in Sections 3.2.4.1 and 3.2.4.2. Fish or crabs from each sampling effort (i.e., a single trawl, a single shrimp or crab trap from a given sampling subarea, or a single beach seine set) will be kept separate from one another and processed one at a time to ensure that individual specimens are tracked properly. Each target species will be individually wrapped in heavy duty aluminum foil (shiny side out), enclosed in individual resealable plastic bags with an identification label (also enclosed in a resealable bag) (Appendix B, Form 4), and immediately stored in coolers with wet ice. Crabs and fish (e.g., sculpin) that have spines will be double-wrapped in heavy duty aluminum foil to minimize punctures in the aluminum foil or plastic bag. Prior to bagging, fish spines will be sheared as required to minimize punctures in the aluminum foil packaging (EPA 2000). If processing occurs at Windward, specimens

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transported to Windward will be unpacked from coolers, measured as described in Section 3.2.4.1 and 3.2.4.2, and weighed using an analytical scale accurate to 0.5 g.

The FC will be responsible for reviewing count, length, weight, gender, and external abnormality information of all species recorded in the field on the fish and crab tissue collection form (Appendix B, Form 1), and will correct any improperly recorded information. Within 24 hr of capture whether processed in the field or at Windward, all fish and crabs, will be packed into coolers as described in Section 3.3.5 and sent to Axys to be held frozen until they are composited and homogenized. Specimen labels, as described in Section 3.2.1, will be included with each shipment. Axys will ship subsamples to the other laboratories for analysis, as described in Section 3.4. Homogenization will not occur until EPA, Ecology, and LDWG have agreed on the final compositing scheme, which may depend on the specimens collected.

Sample labels will contain the project number, sampling personnel, date, time, specimen ID, and comments (Appendix B, Form B-4). All of the pertinent information about the specimen, including the specific trawl, trap, or beach seine the specimen came from, and the specific sampling subarea are traceable through the sample label. A complete sample label will be affixed to each sample as described above.

The specimens included in each composite sample will be tracked using a composite specimen tracking form (Appendix B, Form 5). This form will include the project number, the composite sample ID, the sample ID of each specimen included in the composite, and the length and weight of each specimen.

At each laboratory, a unique sample identifier will be assigned to each sample (using either project ID or laboratory ID). The laboratory will ensure that a sample tracking record follows each sample through all stages of laboratory processing. The sample tracking record must contain, at a minimum, the name/initials of responsible individuals performing the analyses, dates of sample extraction/preparation and analysis, and the type of analysis being performed.

#### 3.3.2 **Decontamination procedures**

Sources of extraneous tissue contamination include contamination from sampling gear, grease from ship winches or cables, spilled engine fuel (gasoline or diesel), engine exhaust, dust, ice chests, and ice used for cooling. All potential sources of contamination in the field will be identified by the FC and appropriate steps will be taken to minimize or eliminate contamination. For example, during retrieval of sampling gear, the boat will be positioned when feasible so that engine exhaust does not fall on the deck. Ice chests will be scrubbed clean with detergent and rinsed with distilled water after use in each sampling area to prevent potential crosscontamination. To avoid contamination from melting ice, samples will be placed in waterproof plastic bags (EPA 2000), and the crushed wet ice will be placed in separate plastic bags. Sampling equipment that has obviously been contaminated by oils, grease, diesel fuel, or gasoline will not be used, unless it can be thoroughly decontaminated

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using detergent and distilled water. All utensils or equipment that will be used directly in handling fish or shellfish (e.g., fish measuring board or calipers) will be cleaned in the Windward laboratory prior to each sampling trip, and stored in aluminum foil until use (EPA 2000). Between sampling areas, the field collection team will clean each measurement device with Alconox<sup>®</sup> detergent, rinse it with ambient water, and wrap it in aluminum foil to prevent contamination. The high-rise otter trawl and beach seine will be manually cleaned of all visible debris and washed in LDW water during deployment, because these nets cannot be practically decontaminated using the same protocol as other sampling equipment due to their large size. However, all fish caught by trawl or beach seine will be placed for a few minutes in a decontaminated container with LDW water to rinse them before processing.

In summary, the following practices will be followed to minimize sample contamination:

- Caught fish or crabs will only be placed on clean surfaces, such as aluminum foil (dull side touching the fish)
- Ice chests will be scrubbed with Alconox<sup>®</sup> detergent and rinsed with deionized water prior to any sampling activities
- Samples will be placed in resealable, waterproof plastic bags to avoid contamination from melting ice
- Sampling equipment will be kept free from contaminants such as oils, grease and fuels

#### 3.3.3 Field-generated waste disposal

Excess fish or crabs, generated equipment rinsates, and decontamination water will be returned to each sampling location after sampling is completed for that location. All disposable sampling materials and personal protective equipment used in sample processing, such as disposable coveralls, gloves, and paper towels, will be placed in heavyweight garbage bags or other appropriate containers. Disposable supplies will be removed from the site by sampling personnel and placed in a normal refuse container for disposal as solid waste.

# 3.3.4 Sample custody procedures

Samples are considered to be in custody if they are: 1) in the custodian's possession or view; 2) in a secured place (under lock) with restricted access; or 3) in a container and secured with an official seal(s) such that the sample cannot be reached without breaking the seal(s). Custody procedures will be used for all samples throughout the collection, transport, and analytical process, and for all data and data documentation whether in hard copy or electronic format. Custody procedures will be initiated during sample collection. A COC form will accompany samples to the analytical laboratory. Each person who has custody of the samples will sign the COC form and ensure that

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the samples are not left unattended unless properly secured. Minimum documentation of sample handling and custody will include:

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

The FC will be responsible for all sample tracking and custody procedures for samples in the field. The FC will be responsible for final sample inventory and will maintain sample custody documentation. The FC will also complete COC forms prior to removing samples from the sampling area. At the end of each day, and prior to transfer, COC entries will be made for all samples. Information on the labels will be checked against sample log entries, and sample tracking forms and samples will be recounted. COC forms will accompany all samples. The COC forms will be signed at each point of transfer. Copies of all COC forms will be retained and included as appendices to QA/QC reports and data reports. Tissue samples will be shipped in sealed coolers to Axys.

The laboratories will ensure that COC forms are properly signed upon receipt of the samples and will note questions or observations concerning sample integrity on the COC forms. The laboratories will contact the FC and Project QA/QC Coordinator immediately if discrepancies are discovered between the COC forms and the sample shipment upon receipt.

The laboratory will ensure that a sample-tracking record follows each sample through all stages of laboratory processing. The sample-tracking record must contain, at a minimum, the name/initials of individuals responsible for performing the analyses, dates of sample extraction/preparation and analyses, and the types of analyses being performed.

#### 3.3.5 Shipping requirements

Samples will be shipped in coolers from Windward to Axys. Prior to shipping, sample containers will be wrapped in bubble wrap and securely packed inside a cooler with ice packs. The original signed COC forms will be placed in a sealable plastic bag, sealed, and taped to the inside lid of the cooler. Fiber tape will be wrapped completely around the cooler. 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, and the cooler will be sealed with a custody seal in two locations.

The temperature inside the cooler(s) containing tissue samples will be checked upon receipt of the samples. The laboratories will specifically note any coolers that do not

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contain ice packs or that are not sufficiently cold  $(4^\circ \pm 2^\circ C)$  upon receipt. All samples will be handled so as to prevent contamination or loss of any sample. Samples will be assigned a specific storage area within the laboratory, and individual specimens will be kept frozen there until compositing instructions are received. After composite samples are created, all remaining tissue samples will be disposed of upon receipt of written notification by the Windward PM. Once samples are homogenized, subsamples will be shipped by Axys to the other analytical laboratories.

# 3.4 LABORATORY METHODS

This section discusses laboratory sample handling requirements, standard and alternative analytical methods, and data quality indicators for laboratory analyses.

# 3.4.1 Laboratory sample handling

Samples will be initially shipped to Axys and held frozen at -20°C until all fish or crabs have been collected. Tissues will then be composited at Axys with Windward personnel present. The individual fish or crabs included in each composite sample will be determined based on the compositing scheme described in Section 3.1.3 and any required modifications, determined in consultation with EPA and Ecology.

Tissue homogenization will be conducted by Axys. The laboratory SOP for tissue homogenization is presented as Appendix C. Thawed or partially thawed whole fish will be homogenized as composite samples or individual fish, depending on the size of the fish. Large fish may need to be homogenized individually and then the individual homogenates combined to form the composite sample. Attempts will be made to composite equal weights of homogenates per fish. Smaller fish may be composited prior to homogenization. For fillet samples, partially-thawed whole fish will be filleted (skin-on) and the fillets will then be homogenized. Homogenates may be frozen; however, frozen homogenates from each individual fish comprising a composite sample must be re-homogenized before compositing for analysis. Recommended container materials, storage temperatures, and holding times are given in Section 3.4.3. Any remaining homogenates (either of individual fish included in composite samples or of the composited samples themselves) will be archived.<sup>30</sup> Whole fish and crabs not homogenized will be archived frozen for this project only until the fish and crab tissue chemistry data validation is approved by EPA. At that time, whole fish and crab that had been archived will be discarded. Homogenized fish and crabs will be archived frozen for one year from collection.

<sup>&</sup>lt;sup>30</sup> Based on August sampling, the average tissue masses for individual English sole, Pacific staghorn sculpin, shiner surfperch, pile perch, starry flounder, striped perch, and rockfish are 161, 43, 16, 75, 274, 199, and 501 g, respectively. The analytical tissue mass requirement is 81 g for fish (see Section 3.4.3.6). Based on these data, for all target species except shiner surfperch, composite samples of 5 fish will provide tissue mass well in excess of the analytical requirements. The minimum number of shiner surfperch collected from any subarea in August was 7; thus, approximately 112 g of tissue can be expected from each composite sample of shiner surfperch. Based on these data, excess tissue homogenates are likely to be archived for all fish species.



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#### 3.4.2 Analytical methods

Chemical analyses of the tissue samples will be conducted at three different laboratories. Analyses to be conducted at each laboratory are presented in Table 3-7.

Axys	COLUMBIA	FRONTIER
Homogenization	PCB Aroclors	Inorganic arsenic
PCB congeners	Organochlorine pesticides	
Dioxins and furans	SVOCs	
Storage	Metals including Hg	
	ТВТ	
	Lipids	
	Moisture	

Table 3-7. Procedures to be conducted at each analytical laboratory

All English sole, Pacific staghorn sculpin, shiner surfperch, and crab tissue samples<sup>31</sup> collected from the LDW will be analyzed for tributyltin (TBT), semivolatile organic compounds (SVOCs), including polycyclic aromatic hydrocarbons (PAHs), metals, PCBs as Aroclors, mercury, and organochlorine pesticides. Tissue samples collected from background areas will only be analyzed for total and inorganic arsenic, lipids, and moisture (see Appendix E).

All 209 PCB congeners will be analyzed in a subset of tissue samples of each tissue type using a tiered approach. In this approach, a portion of the tissue mass from each homogenized sample will be archived frozen for potential PCB congener analysis. All tissue samples will first be analyzed for total PCBs (as an Aroclor sum). Based on the Aroclor results, one third of the samples from each species from each area will be selected for PCB congener analysis to cover the range of total PCB concentrations (Aroclor sum) and to provide spatial coverage within the LDW. Thus, for each whole body fish tissue type (English sole, Pacific staghorn sculpin, and shiner surfperch) English sole fillet, and crab edible meat, two of the six samples from each sampling area will be analyzed for PCB congeners; one crab hepatopancreas sample from each sampling area will also be analyzed for PCB congeners (Table 3-8). The relationships among total PCBs (congener sum), dioxin-like PCB congeners (as toxic equivalents [TEQs]), and total PCBs (Aroclor sum) will be assessed to determine the ability of the Aroclor sum to estimate the total PCB concentration in tissue. LDWG will meet with EPA and Ecology to discuss these results. If the Aroclor sum underestimates the total or the relationship between Aroclor and total congener sums is not consistent enough to be useful, and the data suggest that an increased sample size will improve the fit, all of the tissue samples will be analyzed for all 209 PCB congeners.

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<sup>&</sup>lt;sup>31</sup> Analysis of, as well as specific analytes for, rockfish and striped or pile perch fillets will be determined in consultation with EPA and Ecology, if these species are collected.

	ENGLISH SOLE		SCULPIN	SHINER SURFPERCH	Скав	
ANALYTE	WB	FILLET	WB	WB	EDIBLE MEAT	HEPATOPANCREAS
PCB congeners	2	2	2	2	2	1
Dioxins/furans <sup>a</sup>	6	2	6	6	6	2
PCB Aroclors and organochlorine pesticides	6	2	6	6	6	2
SVOCs	6	2	6	6	6	2
PAHs	6	2	6	6	6	2
Mercury	6	2	6	6	6	2
Other metals, including total arsenic	6	2	6	6	6	2
Inorganic arsenic	2	2	0	2	2	1
TributyItin	6	2	6	6	6	2

# Table 3-8. Numbers of composite samples per LDW sampling area to be analyzedfor each analyte group

WB = whole body

Tissue samples will be archived for potential dioxin/furan analyses. The need for these analyses will be determined after reviewing the dioxin/furan sediment chemistry data to be collected by LDWG in early 2005.

From each sampling area, inorganic arsenic will be analyzed in two randomly selected samples of each of the following types: English sole whole body and fillet, shiner surfperch whole body, and crab edible meat (Table 3-8). Only one crab hepatopancreas sample will be analyzed from each area because this sample type is a minor component of the HHRA and four samples (one from each of four areas) should be sufficient for deriving a site-wide estimate of inorganic arsenic.<sup>32</sup>

The portion of each tissue sample homogenate that is archived for potential PCB congener analysis may also be used for potential dioxin/furan analysis. If the sample is extracted for PCB congener analysis, a portion of the extract will be heat-sealed and frozen. Dioxin/furan analysis in tissue will be conducted if the results of the urban background analysis in sediments described in the Phase 2 work plan indicate that quantitative risk characterization is needed. LDWG, EPA, and Ecology will discuss the background analysis and the dioxin/furan results from the Phase 2 sediment sampling before deciding whether analysis of dioxins/furans in tissues is needed.

Analytical methods and laboratory sample handling requirements for all measurement parameters are presented in Table 3-9.

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<sup>&</sup>lt;sup>32</sup> Based on the results from quarterly crab/shrimp monitoring, it may not be possible to collect sufficient numbers of crabs from Area 4 to provide sufficient hepatopancreas tissue to analyze for inorganic arsenic.

#### Table 3-9. Analytical methods and sample handling requirements

			LABORATORY			
PARAMETER	METHOD	REFERENCE		SAMPLE HOLDING TIME	CONTAINER	PRESERVATIVE
PCBs as Aroclors	GC/ECD	EPA 8082A	Columbia	1 year to extract, 40 days to analyze	aluminum foil (whole fish) glass jar (homogenate)	freeze/-20°C
PCB congeners	HRGC/HRMS	EPA 1668	Axys	1 year to extract, 40 days to analyze	aluminum foil (whole fish) glass jar (homogenate)	freeze/-20°C
Dioxins and furans	HRGC/HRMS	EPA 1613B	Axys	1 year to extract, 40 days to analyze	aluminum foil (whole fish) glass jar (homogenate)	freeze/-20°C
Organochlorine pesticides <sup>a</sup>	GC/ECD	EPA 8081A	Columbia	1 year to extract, 40 days to analyze	aluminum foil (whole fish) glass jar (homogenate)	freeze/-20°C
PAHs <sup>b</sup>	GC/MS	EPA 8270- SIM	Columbia	1 year to extract, 40 days to analyze	aluminum foil (whole fish) glass jar (homogenate)	freeze/-20°C
SVOC <sup>c</sup>	GC/MS	EPA 8270- SIM	Columbia	1 year to extract, 40 days to analyze	aluminum foil (whole fish) glass jar (homogenate)	freeze/-20°C
Arsenic (inorganic) <sup>d</sup>	HG-AFS	EPA 1632	Frontier	6 months	aluminum foil (whole fish) glass jar (homogenate)	freeze/-20°C
Chromium <sup>e</sup>	ICP-AES	EPA 6010	Columbia	6 months	aluminum foil (whole fish) glass jar (homogenate)	freeze/-20°C
Mercury	CVAA	EPA 7471	Columbia	60 days	aluminum foil (whole fish) glass jar (homogenate)	freeze/-20°C
Selenium <sup>e</sup>	BHR-AA	EPA 7742	Columbia	6 months	aluminum foil (whole fish) glass jar (homogenate)	freeze/-20°C
Other metals <sup>f</sup>	ICP-MS	EPA 6020	Columbia	6 months	aluminum foil (whole fish) glass jar (homogenate)	freeze/-20°C
Tributyltin, dibutyltin, monobutyltin (as ions)	GC/FPD	Stallard et al. (1988)	Columbia	1 year to extract, 40 days to analyze	aluminum foil (whole fish) glass jar (homogenate)	freeze/-20°C
Lipids	DCM extraction gravimetric	NOAA (1993)	Columbia	1 year	aluminum foil (whole fish) glass jar (homogenate)	freeze/-20°C
Moisture	freeze-dried	PSEP (1997)	Columbia	6 months	aluminum foil (whole fish) glass jar (homogenate)	freeze/-20°C

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- <sup>a</sup> Target pesticides include: 4,4'-DDT, 4,4'-DDE, 4,4'-DDD, 2,4'-DDT, 2,4'-DDE, 2,4'-DDD, aldrin, alpha-BHC, beta-BHC, gamma-BHC, chlordane, dieldrin, endosulfan, endosulfan sulfate, endrin, heptachlor, heptachlor epoxide, hexachlorobenzene, methoxychlor, mirex, and toxaphene
- <sup>b</sup> Ultra-low extraction method; target PAHs include: anthracene, pyrene, dibenzofuran, dibenzothiophene, benzo(g,h,i)perylene, benzo(e)pyrene, indeno(1,2,3-cd)pyrene, perylene, benzo(b)fluoranthene, fluoranthene, benzo(k)fluoranthene, acenaphthylene, chrysene, benzo(a)pyrene, dibenz(a,h)anthracene, benzo(a)anthracene, acenaphthene, phenanthrene, fluorene, 1-methylnaphthalene, naphthalene, 2-methylnaphthalene, and biphenyl
- <sup>c</sup> All SVOCs, including PAHs
- <sup>d</sup> Inorganic arsenic will be quantified in English sole fillet and whole body, shiner surfperch whole body, crab edible meat and hepatopancreas.
- <sup>e</sup> Chromium and selenium cannot be analyzed in tissue using EPA Method 6020 because of matrix interferences
- f Arsenic, antimony, cadmium, cobalt, copper, lead, molybdenum, nickel, silver, thallium, vanadium, and zinc.

BHR-AA -borohydride reduction atomic absorption

CVAA - cold vapor atomic absorption

DCM - dichloromethane

- GC/ECD gas chromatography/electron capture detector
- GC/FPD gas chromatography/flame photometric detection
- GC/MS gas chromatography/mass spectrometry
- HRGC/HRMS high resolution gas chromatography/high resolution mass spectrometry
- HG-AFS hydride generation-atomic fluorescence spectrometry
- ICP-AES inductively couple-plasma atomic emission spectrometry
- ICP-MS inductively coupled-plasma mass spectrometry
- SIM select ion monitoring



#### 3.4.3 Data quality indicators

The parameters used to assess data quality are precision, accuracy, representativeness, comparability, completeness, and sensitivity. Table 3-10 lists specific DQIs for tissue analyses. Interferences in individual samples may result in an increase in the reported detection limits. To achieve the required low detection limits, some modifications to the methods may be necessary.

PARAMETER	UNITS	PRECISION	ACCURACY	Completeness	Sensitivity (Method Detection Limit) <sup>a</sup>
PCBs as Aroclors	µg/kg ww	±50%	38-150%	95%	0.76-4.7
PCB congeners	ng/kg ww	±50%	50-150%	95%	0.27-3.45
Dioxins and furans	ng/kg ww	±50%	50-150%	95%	0.04
Organochlorine pesticides <sup>b</sup>	µg/kg ww	±50%	30-150%	95%	0.099-5.8
PAHs <sup>c</sup>	µg/kg ww	±50%	20-130%	95%	0.045-0.26 <sup>d</sup>
SVOCs <sup>e</sup>	µg/kg ww	±50%	20-130%	95%	1.3-5,000
Arsenic (inorganic) <sup>f</sup>	mg/kg ww	±25%	75-125%	95%	0.05
Other metals <sup>g</sup>	mg/kg ww	±30%	60-130%	95%	0.002-1.0
Tributyltin, dibutyltin, monobutyltin (as ions)	µg/kg ww	±50%	20-130%	95%	0.33-0.38
Lipids	% ww	±30%	na	95%	0.1
Moisture	% ww	±20%	na	95%	0.1

Table 3-10. Data quality indicators for tissue analyses

<sup>a</sup> Method detection limits for individual chemicals are presented in Appendix D

<sup>b</sup> Target pesticides include: 4,4'-DDT, 4,4'-DDE, 4,4'-DDD, 2,4'-DDT, 2,4'-DDE, 2,4'-DDD, aldrin, alpha-BHC, beta-BHC, gamma-BHC, chlordane, dieldrin, endosulfan, endosulfan sulfate, endrin, heptachlor, heptachlor epoxide, hexachlorobenzene, methoxychlor, mirex, and toxaphene

- <sup>c</sup> Target PAHs include: anthracene, pyrene, dibenzofuran, dibenzothiophene, benzo(g,h,i)perylene, benzo(e)pyrene, indeno(1,2,3-cd)pyrene, perylene, benzo(b)fluoranthene, fluoranthene, benzo(k)fluoranthene, acenaphthylene, chrysene, benzo(a)pyrene, dibenz(a,h)anthracene, benz(a)anthracene, acenaphthene, phenanthrene, fluorene, 1-methylnaphthalene, naphthalene, 2-methylnaphthalene, and biphenyl
- <sup>d</sup> MDLs for low-level PAH analyses to be used for all samples except English sole (whole-body) and sculpin because these samples are either not required for the HHRA or are of low importance.
- e All SVOCs, including PAHs
- <sup>f</sup> Inorganic arsenic will be quantified in English sole fillet and whole body, shiner surfperch whole body, crab edible meat and hepatopancreas
- <sup>g</sup> Arsenic, antimony, cadmium, chromium, cobalt, copper, lead, mercury, molybdenum, nickel, selenium, silver, thallium, vanadium, and zinc

ww-wet weight

#### 3.4.3.1 Precision

Precision is the measure of the reproducibility among individual measurements of the same property, usually under similar conditions, such as multiple measurements of the same sample. Precision is assessed by performing multiple analyses on a sample and is expressed as a relative percent difference (RPD) when duplicate analyses are

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performed and as a percent relative standard deviation (% RSD) when more than two analyses are performed on the same sample (e.g., triplicates). Precision is assessed by laboratory duplicate analyses (duplicate samples, MS duplicates, LCS duplicates) for all parameters. 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 DQI for precision varies depending on the analyte (Table 3-10). The equations used to express precision are as follows:

 $RPD = \frac{(measured \ conc-measured \ duplicate \ conc)}{(measured \ conc+measured \ duplicate \ conc) \div 2} \times 100$ 

%RSD=(SD/D<sub>ave</sub>) × 100

where:

$$SD = \sqrt{\left(\frac{\sum (D_n - D_{ave})^2}{(n-1)}\right)}$$

D	=	sample concentration
Dave	=	average sample concentration
n	=	number of samples
SD	=	standard deviation

#### 3.4.3.2 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 recovery for matrix spike and laboratory control sample analyses. The DQI for accuracy varies, depending on the analyte (Table 3-10). The equation used to express accuracy for spiked samples is as follows:

 $Percent recovery = \frac{spike sample result - unspiked sample result}{amount of spike added} \times 100$ 

# 3.4.3.3 Representativeness

Representativeness expresses the degree to which data accurately and precisely represent an environmental condition. The sampling approach was designed to address the specific objectives described in Section 2.2. Assuming those objectives are met, the samples collected should be considered adequately representative of the environmental conditions they are intended to characterize.

# 3.4.3.4 Comparability

Comparability expresses the confidence with which one data set can be evaluated in relation to another data set. Therefore, the sample collection and chemical and physical testing will adhere to the most recent Puget Sound Estuary Program (PSEP) QA/QC procedures (PSEP 1997) and EPA and PSEP analysis protocols.

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#### 3.4.3.5 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 will be calculated as follows:

 $Completeness = \frac{number of valid measurements}{total number of data points planned} \times 100$ 

The DQI for completeness for all components of this project is 95%. 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.

#### 3.4.3.6 Sensitivity

Analytical sensitivity is the minimum concentration of an analyte above which a data user can be reasonably confident that the analyte was reliably detected and quantified. For this study, the MDL will be used as the measure of sensitivity of each measurement process. Results will be reported at or below the target detection limits presented in Table 3-10. Appendix D presents a detailed evaluation of whether these MDLs are sufficiently sensitive to meet the needs of the Phase 2 ecological and human health risk assessments. Based on that evaluation, the analytical MDLs specified in Table 3-10 for all chemicals except selenium are sufficiently sensitive for sculpin tissue samples to meet the needs of the ERA. MDLs for some analytes in English sole, perch, and crab tissue will exceed risk-based analytical concentration goals (ACGs) for the protection of human health. These MDLs include nine SVOCs, six PCB Aroclors, one PCB congener, six organochlorine pesticides, total and inorganic arsenic, chromium, mercury, selenium, and 2,3,7,8-TCDD. It should be noted that for three SVOCs, chromium, mercury and four pesticides, only the ACGs based on the total seafood consumption rate of 98g/day are below the MDL, not the ACGs calculated based on consumption rates specific for benthic fish, pelagic fish, and crab.

Elevated MDLs relative to ACGs are only problematic when chemicals are not detected. The laboratory will make additional efforts to achieve ACGs for Aroclors in samples if no Aroclors are detected in a sample. The lab will also make additional efforts to achieve the ACG for PCB congener 126 based on the consumption rate specific to the tissue type if it is not detected in a sample. Additional efforts may include additional sample clean-up, extracting more sample, using a lower concentration for the lowest standard in the initial calibration, adjusting the final volume, or adjusting the amount of extract injected into the instrument. For the other chemicals with MDLs above the ACGs, the ramifications for the Phase 2 HHRA will be discussed in the uncertainty assessment.

All ACGs for sculpin, which are not consumed by humans, are greater than or equal to the MDLs shown in Table D-11, with the exception of selenium, indicating that all analytical methods cited, except EPA Method 7742 for selenium, are sufficiently

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Standard tissue mass requirements are specified to meet MDLs for each particular analytical method. Because collecting the standard tissue mass may be difficult for crab tissue samples, an analysis was conducted to determine if a lower tissue mass could be collected and still meet the risk-based ACGs described in Appendix D. Based on this analysis, crab tissue mass could be lowered only for the TBT analysis, from 10 g to 2 g of sample (see Appendix D for determination of required tissue mass). <sup>33</sup> Table 3-11 summarizes the tissue mass needed for each sample type.

ANALYTE	TISSUE MASS (g) FOR CRABS <sup>a</sup>	TISSUE MASS (g) For Fish
PCB congeners and dioxins/furans	25 <sup>b</sup>	25 <sup>b</sup>
PCB Aroclors and organochlorine pesticides	20 <sup>c</sup>	20 <sup>c</sup>
SVOCs	10	10
PAHs (ultra-low extraction)	10	10 <sup>d</sup>
Mercury	2	2
Other metals <sup>e</sup>	2	2
Inorganic arsenic <sup>f</sup>	2	2
Tributyltin	2	10
Total Mass	73	81

 Table 3-11.
 Tissue mass required per sample type

na - not analyzed

- <sup>a</sup> Separate tissue mass will be collected for edible meat and for hepatopancreas
- <sup>b</sup> Tissue mass will be archived for samples not initially analyzed for PCB congeners. Also, a portion of the extract from samples analyzed for PCB congeners will be heat-sealed and frozen for potential dioxin/furan analysis.
- <sup>c</sup> A portion of the extract will be used for lipid analysis. Therefore, no additional tissue is required for lipid analysis
- <sup>d</sup> Low-level PAH analyses are not required for English sole (whole-body) and sculpin samples, because these samples are either not required for the HHRA or are of low importance. PAHs in these samples will be measured as part of the SVOC analysis, so the total mass required for these samples is 71 g
- e Tissue mass is sufficient for metals analyses using Methods 6010, 6020, and 7742
- <sup>f</sup> Inorganic arsenic will be analyzed in a subset of samples, as described in the text of this section

# 3.5 QUALITY ASSURANCE/QUALITY CONTROL

This section discusses the types of samples analyzed and procedures conducted for QA/QC in the field and laboratory.

<sup>&</sup>lt;sup>33</sup> Standard and modified tissue mass requirements do not include the amount needed for laboratory quality control samples, thus an additional 10 g tissue mass will need to be collected for each matrix spike and matrix spike duplicate sample (i.e., one of each for every 20 field samples).



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#### 3.5.1 Field quality control samples

Field QA/QC samples, such as field duplicates and rinsate blanks, are generally used to evaluate the efficiency of field decontamination procedures and variability attributable to sample handling. For the fish and crab tissue sampling, rinsate blanks are not relevant. Field duplicate fish composite samples will not be collected, although matrix replicates of homogenized tissue samples will be analyzed as described in the following section.

# 3.5.2 Laboratory quality control

Before analyzing the samples, the laboratory must provide written protocols for the analytical methods to be used, calculate MDLs for each analyte in each matrix of interest, and establish an initial calibration curve for all analytes. The laboratory must demonstrate their continued proficiency by participation in inter-laboratory comparison studies and through repeated analysis of certified reference materials, calibration checks, laboratory reagent and rinsate blanks, and spiked samples.

#### 3.5.2.1 Determination of MDLs

The MDL is defined as the lowest concentration of an analyte or compound that a method can detect in either a sample or a blank with 99% confidence. The laboratories determine MDLs using standard procedures outlined in 40 CFR§136, where seven replicate samples are fortified at 1 to 5 times (but not to exceed 10 times) the expected MDL concentration. The MDL is then determined by calculating the standard deviation of the replicates and multiplying by a factor of 3.14.

#### 3.5.2.2 Sample delivery group

Project and/or method-specific quality control measures such as matrix spikes and matrix spike duplicates will be analyzed per sample delivery group (SDG) or sample batch. An SDG is defined as no more than 20 samples or a group of samples received at the laboratory within a two-week period. Although a SDG may span two weeks, all holding times specific to each analytical method will be met for each sample in the SDG.

#### 3.5.2.3 Laboratory quality control samples

The analyst will review results of QC analyses from each sample group immediately after a sample group has been analyzed. The QC sample results will then be evaluated to determine whether control limits have been exceeded. If control limits are exceeded in the sample group, the Project QA/QC Coordinator will be contacted immediately, and corrective action, such as method modifications followed by reprocessing of the affected samples, will be initiated before processing a subsequent group of samples.

All primary chemical standards and standard solutions used in this project will be traceable to the National Institute of Standards and Technology, Environmental Resource Associates, National Research Council of Canada, or other documented, reliable, commercial sources. Standards will be validated to determine their accuracy

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by comparison with an independent standard. Laboratory QC standards are verified a multitude of ways. Second source calibration verifications are run (i.e., same standard, two different vendors) for calibrations. New working standard mixes (calibrations, spikes, etc.) are verified against the results of the original solution and must be within 10%. Newly purchased standards are verified against current data. Any impurities found in the standard will be documented. The following sections summarize the procedures that will be used to assess data quality throughout sample analysis. Table 3-12 summarizes the QC procedures to be performed by the laboratory. The associated control limits for precision and accuracy are summarized in Table 3-11.

ANALYSIS TYPE	INITIAL CALIBRATION	CONTINUING CALIBRATION	MATRIX REPLICATES	MATRIX SPIKES	MATRIX SPIKE DUPLICATES	Method Blanks	STANDARD REFERENCE MATERIAL	SURROGATE SPIKES
PCBs (Aroclors)	prior to analysis	Every 10-20 analyses or 12 hrs	na	1 per batch or SDG	1 per batch or SDG	Each batch or SDG	naª	Each sample
PCB congeners	prior to analysis	Every 10-20 analyses or 12 hrs	na	1 per batch or SDG	1 per batch or SDG	Each batch or SDG	Each batch or SDG (Radian EDF 2524)	Each sample
Dioxins and furans	prior to analysis	Every 10-20 analyses or 12 hrs	na	1 per batch or SDG	1 per batch or SDG	Each batch or SDG	Each batch or SDG(Radian EDF 2524)	Each sample
Organochlorine pesticides <sup>b</sup>	prior to analysis	Every 10-20 analyses or 12 hrs	na	1 per batch or SDG	1 per batch or SDG	Each batch or SDG	Each batch or SDG (NIST 1945)	Each sample
Mercury	prior to analysis	Every 10 samples	na	1 per batch or SDG	1 per batch or SDG	Each batch or SDG	Each batch or SDG (Dolt 3)	na
Other metals	prior to analysis	Every 10 samples	1 per batch or SDG	1 per batch or SDG	na	Each batch or SDG	Each batch or SDG (Dorm 2)	na
Semivolatiles, including PAHs	prior to analysis	Every 10-20 analyses or 12 hours	na	1 per batch or SDG	1 per batch or SDG	Each batch or SDG	Each batch or SDG (NIST 1947b for PAHs)	Each sample
Tributyltin	prior to analysis	Every 10 samples	na	1 per batch or SDG	1 per batch or SDG	Each batch or SDG	Each batch or SDG (CRM 477)	Each sample
Percent solids	na	na	1 per 20 samples	na	na	na	na	na
Lipids	na	na	1 per 20 samples	na	na	na	na	na

 Table 3-12.
 Laboratory quality control sample analysis summary

<sup>a</sup> LCS sample will be used to assess accuracy because no tissue SRM is available for Aroclors

<sup>b</sup> Aroclor standards will be run as interference check samples for this analysis

na - not applicable or not available

#### Matrix Replicates

Analytical replicates provide information on the precision of the analysis and are useful in assessing potential sample heterogeneity and matrix effects. Analytical replicates are subsamples of the original sample that are prepared and analyzed as a separate sample, assuming sufficient sample matrix is available. A minimum of one

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replicate will be analyzed for each sample group or for every 20 samples, whichever is more frequent. If insufficient material is available for matrix replicates and spikes, standard reference materials will be substituted.

#### Matrix Spikes and Matrix Spike Duplicates

The analysis of matrix spike samples provides information on the extraction efficiency of the method on the sample matrix. By performing duplicate matrix spike analyses, information on the precision of the method is also provided for organic analyses. A minimum of one matrix spike will be analyzed for each sample group or for every 20 samples, whichever is more frequent, when possible. A standard reference material will be used to assess method accuracy for those parameters that cannot be spiked.

#### Method Blanks

Method blanks are analyzed to assess possible laboratory contamination at all stages of sample preparation and analysis. A minimum of one method blank will be analyzed for each extraction/digestion batch or for every 20 samples, whichever is more frequent.

#### Standard Reference Material

SRMs are samples of similar matrix and of known analyte concentration, processed through the entire analytical procedure and used as an indicator of method accuracy. A minimum of one SRM will be analyzed for each sample group or for every 20 samples, whichever is more frequent.

#### Surrogate Spikes

All project samples analyzed for organic compounds will be spiked with appropriate surrogate compounds as defined in the analytical methods. Surrogate recoveries will be reported by the laboratories; however, no sample results will be corrected for recovery using these values.

#### Interference Check Samples

In order to identify specific organochlorine pesticides that may coelute with PCB congeners, single point mid-concentration PCB standards (Aroclors 1248, 1254, and 1260) will be run with single-component pesticides in the initial calibration. Additional Aroclor standards will be run if they are detected in sediment or tissue samples. The resulting data will be reviewed by the data validators in order to assess potential coelution issues affecting the reported pesticide results.

# 3.6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

Prior to each field event, measures will be taken to test, inspect, and maintain all field equipment. All equipment used, including the GPS unit and digital camera, will be tested for use before leaving for the field event.

The FC will be responsible for overseeing the testing, inspection, and maintenance of all field equipment. The laboratory PM will be responsible for ensuring laboratory



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equipment testing, inspection, and maintenance requirements are met. The methods used in calibrating the analytical instrumentation are described in the following section.

# 3.7 INSTRUMENT/EQUIPMENT CALIBRATION AND FREQUENCY

Multipoint initial calibration will be performed on each instrument at the start of the project, after each major interruption to the analytical instrument, and when any continuing calibration does not meet the specified criteria. The number of points used in the initial calibration is defined in each analytical method. Continuing calibrations will be performed daily for organic analyses and every 10 samples for the inorganic analyses, and with every sample batch for conventional parameters to ensure proper instrument performance.

In addition, if an Aroclor is detected in a sample, then the standard for that Aroclor must be analyzed in the continuing calibration within 72 hrs of the original detection of the Aroclor. Gel permeation chromatography (GPC) calibration verifications will be performed at least once every 7 days and corresponding raw data will be submitted by the laboratory with the data package. In addition, florisil performance checks will be performed for every florisil lot and the resulting raw data will be submitted with the data package. Calibration of analytical equipment used for chemical analysis includes instrument blanks or continuing calibration blanks, which provide information on the stability of the baseline established. Continuing calibration blanks will be analyzed immediately after the continuing calibration verification at a frequency of one blank for every 10 samples analyzed for inorganic analyses and one blank for every 12 hours for organic analyses. If the continuing calibration does not meet the specified criteria, the analysis must stop. Analysis may resume after corrective actions have been taken to meet the method specifications. All project samples analyzed by an instrument found to be out of compliance must be reanalyzed.

Two different global positioning system (GPS) receiver units will be employed for the various sampling methods outlined in this QAPP, a Trimble NT300D and a Magellan SporTrak GPS unit. GPS receivers will be calibrated daily to ensure they are accurately recording positions from known benchmarks, within the individual unit's factory specifications.

Analytical scales will be used in the field and at Windward for weighing fish and crabs. The scales will be calibrated using the scale's internal calibration before weighing samples at each sampling event. Scales will be tared before each sample is weighed.

# 3.8 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

The field team leaders for each sampling event will have a checklist of supplies required for each day in the field (see Section 3.2.5). The FC will gather and check these supplies daily for satisfactory conditions before each field event. Batteries used

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in the GPS unit and digital camera will be checked daily and recharged as necessary. Supplies and consumables for the field sampling effort will be inspected upon delivery and accepted if the condition of the supplies is satisfactory.

# 3.9 NON-DIRECT MEASUREMENTS

Tide stage data will be obtained from the Harbor Tides website (<u>http://www.saltwatertides.com/dynamic.dir/washingtonsites.html</u>), which provides daily tide tables for a station at the Lockheed Shipyard on Harbor Island, Seattle, WA.

# 3.10 DATA MANAGEMENT

All field data will be recorded on field forms, which will be checked for missing information by the FC at the end of each field day and amended. After sampling is completed, all data from field forms will be entered into a Microsoft Excel® spreadsheet. A QC check will be done to ensure that all data were properly transferred from the field forms to the spreadsheet. This spreadsheet will be kept on the Windward network drive, which is backed up daily. Field forms will be archived in the Windward library.

The analytical laboratories are expected to submit data in an electronic format as described in Section 2.6.2 and Table 2-4. The laboratory PM will contact the Project QA/QC Coordinator prior to data delivery to discuss specific format requirements.

A library of routines will be used to translate typical electronic output from laboratory analytical systems and to generate data analysis reports. The use of automated routines ensures that all data are consistently converted into the desired data structures and that operator time is kept to a minimum. In addition, routines and methods for quality checks will be used to ensure such translations are correctly applied.

Written documentation will be used to clarify how field and analytical laboratory duplicates and QA/QC samples were recorded in the data tables and to provide explanations of other issues that may arise. The data management task will include keeping accurate records of field and laboratory QA/QC samples so that project team members who use the data will have appropriate documentation. Data management files will be stored on a secure computer.

# 4.0 Assessment and Oversight

# 4.1 COMPLIANCE ASSESSMENTS AND RESPONSE ACTIONS

EPA, Ecology, or their designees may observe field activities during each sampling event, including the background area sampling, as needed. If situations arise where

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# 4.1.1 Compliance assessments

Laboratory and field performance assessments consist of on-site reviews conducted by EPA of QA systems and equipment for sampling, calibration, and measurement. EPA personnel may conduct a laboratory audit prior to sample analysis. Any pertinent laboratory audit reports will be made available to the Project QA/QC Coordinator upon request. Analytical laboratories are required to have written procedures addressing internal QA/QC; these procedures will be submitted for review by the Project QA/QC Coordinator to ensure compliance with the QAPP. All laboratories and QA/QC Coordinators are required to ensure that all personnel engaged in sampling and analysis tasks have appropriate training.

# 4.1.2 Response actions for field sampling

The FC, or a designee, will be responsible for correcting equipment malfunctions throughout field sampling and for resolving situations in the field that may result in nonconformance or noncompliance with the QAPP. All corrective measures will be immediately documented in the field logbook, and protocol modification forms will be completed.

# 4.1.3 Corrective action for laboratory analyses

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

The Project QA/QC Coordinator will be notified immediately if any QC sample exceeds the project-specified control limits (Table 3-9). The analyst will identify and correct the anomaly before continuing with the sample analysis. The laboratory PM will document the corrective action taken in a memorandum submitted to the Project QA/QC Coordinator within 5 days of the initial notification. A narrative describing the anomaly, the steps taken to identify and correct the anomaly, and the treatment of the relevant sample batch (i.e., recalculation, reanalysis, reextraction) will be submitted with the data package using a corrective action form (Appendix B).

# 4.2 **REPORTS TO MANAGEMENT**

Progress reports will be prepared by the FC for submittal to LDWG following each sampling event. The project QA/QC coordinator will also prepare progress reports for submittal to LDWG after the sampling is completed and samples have been submitted for analysis, when information is received from the laboratory, and when analysis are complete. The status of the samples and analyses will be indicated with emphasis on

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any deviations from the QAPP. A data report will be written after validated data are available, as described in Section 2.6.4.

# 5.0 Data Validation and Usability

# 5.1 DATA VALIDATION

Data are not considered final until validated. Data validation for both LDW and background area samples will be conducted following EPA (1999b, 2002b) guidance.

The data validation process begins within the laboratory with the review and evaluation of data by supervisory personnel or QA specialists. The laboratory analyst is responsible for ensuring that the analytical data are correct and complete, that appropriate procedures have been followed, and that QC results are within the acceptable limits. The project QA/QC coordinator is responsible for ensuring that all analyses performed by the laboratories are correct, properly documented, and complete, and that they satisfy the project DQOs specified in this QAPP.

Independent third-party data review and summary validation of the analytical chemistry data will be conducted by Cari Sayler of Sayler Data Solutions, Inc or a suitable alternative. A minimum of 10% or a single sample delivery group will undergo full data validation. Full data validation parameters include:

- quality control analysis frequencies
- analysis holding times
- laboratory blank contamination
- instrument calibration
- surrogate recoveries
- ♦ LCS recoveries
- matrix spike recoveries
- matrix spike/matrix spike duplicate RPDs
- compound identifications verification of raw data with the reported results (10% of analytes)
- compound quantitations verification of calculations and reporting limits (10% of analytes)
- instrument performance check (tune) ion abundances
- internal standard areas and retention time shifts

If no discrepancies are found between reported results and raw data in the set that undergoes full data validation, then validation can proceed as a summary validation on the rest of the data using all of the QC forms submitted in the laboratory data

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package. Quality assurance review of the tissue chemistry data will be performed in accordance with the QA requirements of the project, the technical specifications of the analytical methods indicated in Table 3-9, and EPA (1999b, 2002b) guidance for organic and inorganic data review. The EPA PM may have EPA peer review the third-party validation or perform data assessment/validation on a percentage of the data.

All discrepancies and requests for additional, corrected data will be discussed with the laboratories prior to issuing the formal data validation report. All contacts with the laboratories will be documented in a communication report. Review procedures used and findings made during data validation will be documented on worksheets. Sayler Data Solutions will prepare a data validation report that will summarize QC results, qualifiers, and possible data limitations. This data validation report will be appended to the tissue data report. Only validated data with appropriate qualifiers will be released for general use.

# 5.2 RECONCILIATION WITH DATA QUALITY OBJECTIVES

Data quality assessment will be conducted by the project QA/QC Coordinator in accordance with EPA guidelines. The results of the third-party independent review and validation will be reviewed and cases where the projects DQOs were not met will be identified. The usability of the data will be determined in terms of the magnitude of the DQO exceedance.

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