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State of Washington

Quality Assurance Project Plan

Assessment for Chemical Contaminants in Northeastern Washington Area Lakes

November 2010

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Author and Contact Information

Art Johnson
P.O. Box 47600
Environmental Assessment Program
Washington State Department of Ecology
Olympia, WA 98504-7710

For more information contact: Communications Consultant, phone 360-407-6834.

Washington State Department of Ecology - www.ecy.wa.gov/

- Headquarters, Olympia 360-407-6000
- Northwest Regional Office, Bellevue 425-649-7000
- Southwest Regional Office, Olympia 360-407-6300
- Central Regional Office, Yakima 509-575-2490
- Eastern Regional Office, Spokane 509-329-3400

Quality Assurance Project Plan

Assessment for Chemical Contaminants in Northeastern Washington Area Lakes

November 2010

Approved by:

Signature: _____ Date: November 2010
John Roland, Client, Toxics Cleanup Program, ERO

Signature: _____ Date: October 2010
Mike Hibbler, Regional Manager, Toxics Cleanup Program, ERO

Signature: _____ Date: October 2010
Arianne Fernandez, Client, HWTR Program, ERO

Signature: _____ Date: November 2010
Lisa Brown, Regional Manager, HWTR Program, ERO

Signature: _____ Date: October 2010
Jim Bellatty, Regional Manager, Water Quality Program, ERO

Signature: _____ Date: October 2010
Art Johnson, Project Manager, Toxics Studies Unit, EAP

Signature: _____ Date: October 2010
Dale Norton, Unit Supervisor, Toxics Studies Unit, EAP

Signature: _____ Date: November 2010
Gary Arnold, Section Manager, Eastern Operations Section, EAP

Signature: _____ Date: October 2010
Michael Friese, EIM Data Engineer, EAP

Signature: _____ Date: October 2010
Stuart Magoon, Director, Manchester Environmental Laboratory, EAP

Signature: _____ Date: October 2010
Bill Kammin, Ecology Quality Assurance Officer

Signatures are not available on the Internet version.
EAP: Environmental Assessment Program.
EIM: Environmental Information Management system.
HWTR: Hazardous Waste and Toxics Reduction Program.
ERO: Eastern Regional Office.

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Abstract

Each study conducted by the Washington State Department of Ecology (Ecology) must have an approved Quality Assurance (QA) Project Plan. The plan describes the objectives of the study and the procedures to be followed to achieve them. After completing the study, Ecology will post the final report to the Internet.

This QA Project Plan is for a study to characterize levels of potentially toxic metals and organic compounds in sediments and fish from selected lakes in the northeastern Washington area. The data are needed to support cleanup decisions in Ecology's Eastern Region. Bottom sediments, fish fillets, and whole fish samples from up to 15 waterbodies will be analyzed for mercury, other metals, polychlorinated biphenyls (PCBs), polychlorinated dibenzo-p-dioxins and -dibenzofurans (PCDDs/PCDFs), and polybrominated diphenyl ethers (PBDEs). Sensitive analytical methods will be used to achieve low detection limits for the target chemicals.

Introduction

In 2007-2008, the Washington State Department of Ecology (Ecology) conducted a statewide assessment of PCB and dioxin levels in fish from background lakes and rivers in Washington (Johnson et al., 2010). Ecology needed this information to help prioritize the state's resources for cleaning up 303(d) listed waterbodies that do not meet human health criteria specified in the EPA National Toxics Rule. The study showed that background levels of these chemicals were often low in the far eastern counties (Figure 1).

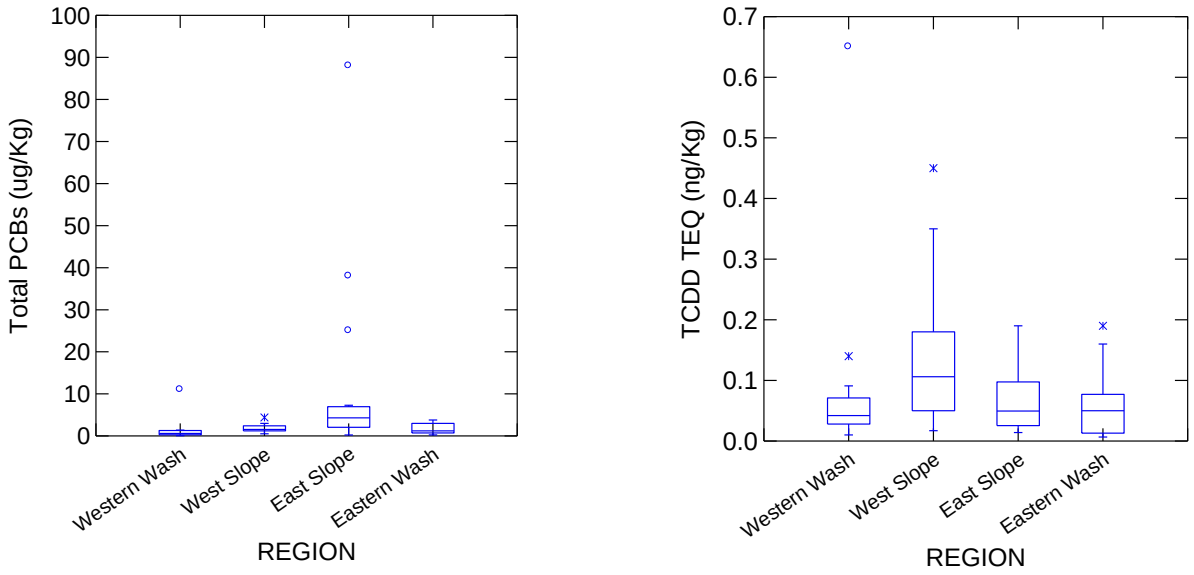

Figure 1. PCB and Dioxin Levels in Fish from Background Lakes and Rivers in Washington. *(Fillet data from Johnson et al., 2010; TCDD TEQs are dioxin toxicity equivalents.)*

Another Ecology study by Sloan and Blakley (2009) focused on selecting appropriate reference areas for freshwater sediment investigations. Sloan and Blakley noted a general lack of information on baseline chemical and biological conditions for aquatic sediments in eastern Washington.

The regional variability and data gaps came to the attention of Ecology's Eastern Regional Office (ERO). ERO's Toxics Cleanup Program (TCP), Water Quality Program (WQ), and Hazardous Waste & Toxic Reduction Program (HWTR) expressed concern that the use of statewide-based background values for decision-making purposes would tend to inappropriately bias outcomes, particularly for cleanup actions. They saw a need for additional reference data specific to northeastern Washington. In view of these concerns, TCP, WQ, and HWTR initiated a project to assess levels for a range of metallic and organic contaminants in fish and sediments from northeastern Washington area lakes. The results will inform a range of cleanup decisions in the Eastern Region. TCP, WQ, and HWTR are hereafter collectively referred to as ERO.

Project Description

Present understanding of area or regional-scale background conditions for chemical contaminants in aquatic environments in the northeastern Washington area is limited. Ecology will therefore pursue a field study to achieve enhanced testing of selected waterbodies in Ferry, Stevens, and Pend Oreille Counties, as well as two representative waterbodies in northern Idaho. An anticipated 13 lakes will be sampled in Washington. One lake and one river will be sampled in Idaho.

The objective of this study is to characterize the levels of selected metals and organic compounds in bottom sediment and fish tissue from waterbodies that exhibit relatively low impact from human activities. Factors considered in lake selection included land-use development, proximity to mining and industry, general local watershed conditions, and known management history. Results will be coordinated with data from selected eastern Washington lakes previously obtained associated with the state-wide surveys.

Field work will take place during the late summer and fall of 2010. Bottom sediments, fish fillets, and whole fish samples will be analyzed for mercury, ten other potentially toxic metals, polychlorinated biphenyls (PCBs), polychlorinated dibenzo-p-dioxins and -dibenzofurans (PCDDs/PCDFs), and polybrominated diphenyl ethers (PBDEs). Sensitive analytical methods will be used to achieve low detection limits for the target chemicals.

The study will be conducted by the Ecology Environmental Assessment Program (EA Program) with the assistance of ERO. The samples will be analyzed by the Ecology Manchester Environmental Laboratory (MEL) and their contractors. A final project report is scheduled for July 2011. This Quality Assurance (QA) Project Plan follows the Ecology guidance in Lombard and Kirchmer (2004).

Study Design

Study Area

ERO has defined the study area for this project as encompassing Ferry, Stevens, and Pend Oreille counties. This region includes or is adjacent to the majority of the cleanup and hazardous waste sites in northeastern Washington and provides several waterbodies exhibiting relatively low impact by human activities. Within the study area, however, potential exists for atmospheric depositional influences caused by historic emissions from smelter operations in British Columbia.

ERO requested that Upper Priest Lake and the upper St. Joe River in northern Idaho be included in the study, in view of their natural condition and proximity to Washington. Upper Priest Lake lies in the Pend Oreille basin. The St. Joe River flows into Lake Coeur D'Alene, which drains to the Spokane River.

Target Chemicals

The study will analyze the following chemicals of primary concern to ERO (Table 1):

Table 1. Target Chemicals for the Northeastern Washington Area Study.

Antimony
Arsenic
Barium
Cadmium
Chromium
Copper
Iron
Lead
Manganese
Mercury
Zinc
Polychlorinated biphenyls (PCBs; 209 congeners)[1]
Polychlorinated dibenzo-p-dioxins (PCDDs; 7 congeners)
Polychlorinated dibenzofurans (PCDFs; 10 congeners)
Polybrominated diphenylethers (PBDEs; 36 congeners)

[1] The term congener refers to different variants or configurations of a common chemical structure. All 209 PCB congeners are being analyzed in the present study. The subset of PCDD, PCDF, and PBDE congeners being analyzed is listed in Appendix A

Mercury, cadmium, lead, PCBs, PCDDs, PCDFs, and PBDEs are persistent, bioaccumulative toxics (PBTs) that are a hazard for fish and other aquatic life, wildlife, and human health (www.ecy.wa.gov/programs/swfa/pbt). The other metals being analyzed also have toxic properties and can bioaccumulate but are not classed as PBTs. Detailed profiles on the target chemicals for this study - describing health effects, physical/chemical properties, production and use, environmental occurrence, regulations, and analysis methods - have been prepared by the Agency for Toxic Substances & Disease Registry (www.atsdr.cdc.gov/toxprofiles/index.asp). This site profiles hazardous substances found at National Priorities List (Superfund) sites.

Ancillary parameters to be analyzed will include total organic carbon (TOC) and grain size in sediment, and percent lipids in fish tissue. These parameters may be of use in normalizing the data for comparing contaminant levels between waterbodies.

Waterbody Selection

For purposes of this study, the term "background" denotes a waterbody currently appearing to be characterized by near-natural conditions or minimally influenced by non-atmospheric human activities. ERO and the EA Program developed a preliminary list of potential background lakes by examining Washington state maps and GIS coverages showing population density, agricultural land use, industrial and municipal outfalls, surface mines, and public lands. Recommendations were also provided by Bill Baker of the Washington Department of Fish and Wildlife (WDFW) and Sheri Sears, Resident Division Fish Manager for the Colville Confederated Tribes. This effort identified lakes that appeared to have a low probability of local sources of contamination.

The appropriateness of each lake for inclusion in the study was then checked against Ecology's Facility Site Identification System, found at www.ecy.wa.gov/fs/index.html). Facility Site identifies sites known to Ecology as having an active or potential impact on the environment. Facility Site showed several mines or mining-related sites in the Cedar Lake watershed (Lucky Four Mine, Redtop Mine, Northport Minerals). It was decided, however, to retain Cedar Lake in the study following a reconnaissance visit, with the understanding that its appropriateness may need to be re-evaluated for some target chemicals once the results are in.

Ecology and WDFW staff were contacted to verify that the lakes considered for study had not been chemically treated to control aquatic plants, algae, or undesirable fish species. Records showed some of these lakes had been treated with rotenone in the past as part of a WDFW program to eliminate spiny-rayed fish and rehabilitate the trout fishery. The historic use of rotenone, a natural product derived from derris root, was not viewed as compromising a lake's usefulness for this study.

Based on the above evaluations, 26 lakes (15 primary and 11 alternate) were tentatively selected for the background study (Table 2, Figure 2). Up to 15 of these lakes will ultimately be sampled.

Table 2. Waterbodies Being Considered for Sampling in the Northeastern Washington Area. (Lakes of primary interest in **bold** font)

Waterbody	County	WRIA No.	Elevation (feet)	Surface Area (acres)	Maximum Depth (feet)	Latitude	Longitude
Swan L	Ferry	52	3,641	52	95	48.512	118.839
Long L		52	3,250	14	58	48.496	118.813
Renner L		52	2,525	9.6	no data	48.781	118.189
Davis L		60	4,550	17	no data	48.739	118.231
Trout L		58	3,000	8	no data	48.627	118.241
Ellen L		58	2,300	78	34	48.501	118.256
South Twin L		58	2,572	973	57	48.264	118.387
Summit L	Stevens	60	2,600	7	no data	48.959	118.127
Pierre L		60	2,012	106	75	48.905	118.139
Cedar L		61	2,135	52	28	48.943	117.594
Pepoon L		60	2,450	11	32	48.901	117.893
Ansaldo L		60	3,050	15	no data	48.897	117.922
Phalon L		61	2,380	23	25	48.784	117.898
Williams L		61	1,980	38	47	48.755	117.968
Bayley L		59	2,400	17	12	48.420	117.664
Crater L	Pend Oreille	62	4,400	no data	no data	48.882	117.262
Sullivan L		62	1,380	1,290	330	48.816	117.292
Muskegon L		62	3,450	7	no data	48.797	117.038
Leo L		62	2,588	39	37	48.648	117.495
Yocum L		62	2,875	42	60	48.613	117.331
Browns L		62	3,450	88	23	48.439	117.191
N. Skookum L		62	3,550	39	20	48.406	117.180
No Name L		62	2,850	18	30	48.297	117.136
Bead L		62	2,850	720	170	48.299	117.116
Upper Priest L	Bonner	Idaho	2,441	1,338	no data	48.786	116.889
St. Joe R	Clearwater	Idaho	na	na	na	above Avery	

WRIA: Water Resource Inventory Area.
na: not applicable.

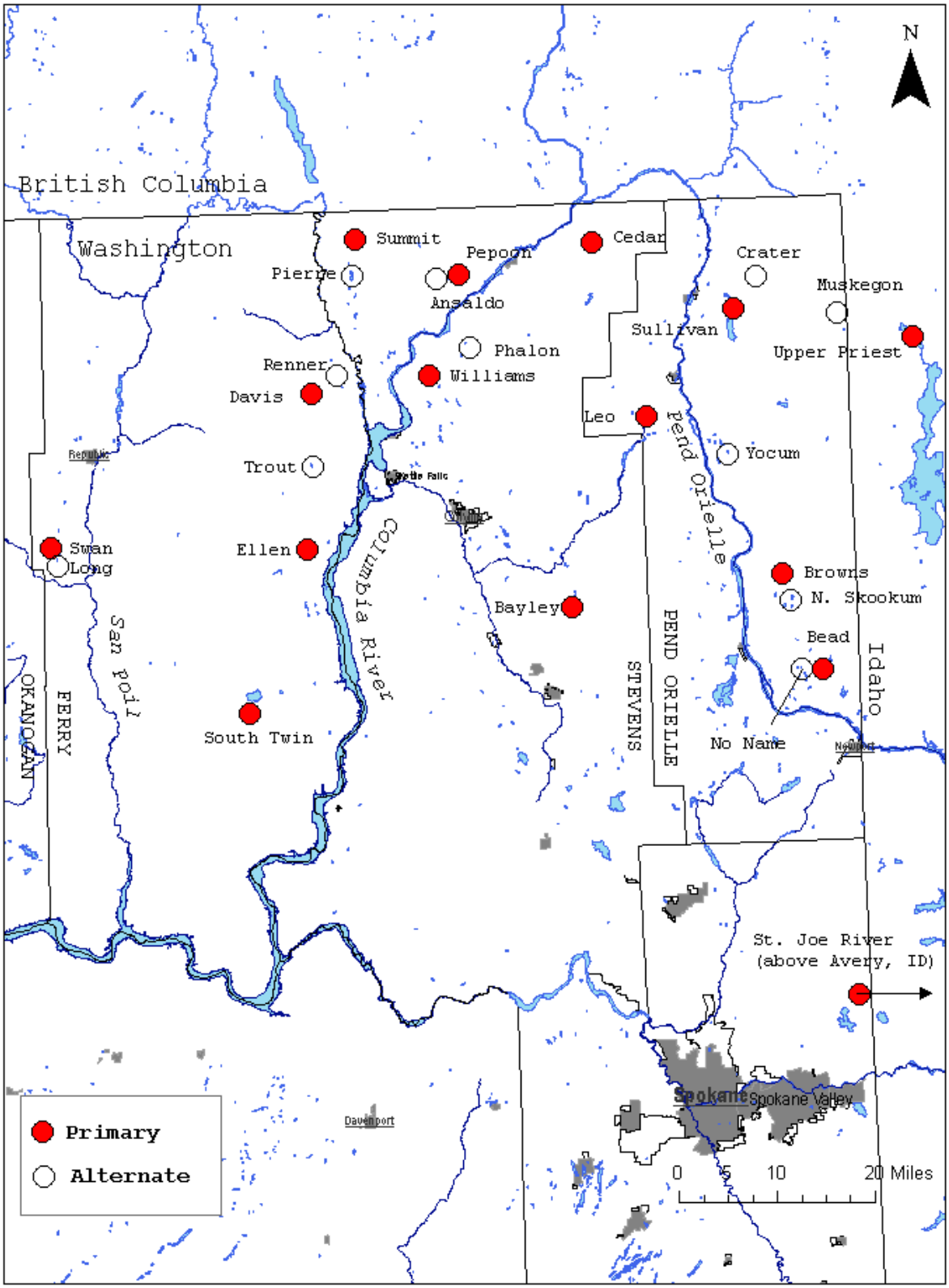

Figure 2. Waterbodies Being Considered for Sampling in the Northeastern Washington Area.

An attempt was made to distribute the sampling effort more or less evenly across the study area, although this was not always possible. Most of the lakes lie in a north-south gradient along the Columbia and Pend Oreille rivers. More emphasis was placed on lakes near the Columbia due to significant transboundary pollution issues.

Size was not an important factor in selecting the lakes, absence of local pollutant sources and geographic location being considered more important. Larger lakes tend to have longer food chains which may result in some species attaining higher levels of bioaccumulative chemicals in their tissues.

Elevation was generally applied in order to select waterbodies in non-mountainous zones, since Ecology regulatory cleanup actions are rarely associated with alpine locations. High mountain lakes are subject to enhanced atmospheric deposition of synthetic organic compounds due to colder temperatures and larger amounts of precipitation (Wania and Mackay, 1993; Gillian and Wania, 2005). High lakes also typically have a low diversity of fish species.

The present study will sample a diverse range of lake sizes and elevations to obtain an area-wide estimate of the chemical background. The lakes selected for study range in size from less than 10 to over 1,000 acres, with maximum depths of 12 to 180 feet. Elevations are between about 2,000 and 4,500 feet.

Appropriate background lakes could not be located in the southern parts of Ferry and Stevens counties, which are more highly developed. Northwestern Ferry County is lacking in lakes of significant size except for Curlew Lake which has sources related to mining, recreation, and other development.

Prior to initiating the field sampling program, each lake will be investigated in more detail and a final selection of 15 waterbodies made for the study. This will include:

- Searching for existing chemical data in Ecology's Environmental Information Management system (EIM).
- Obtaining aerial and bathymetric maps of each waterbody.
- Identifying location and condition of boat ramps or other means of access.
- Further contacts with regional biologists.
- Determining what fish species are present.
- Reviewing fish stocking history.
- Obtaining required permits and verifying they allow collection of species of interest.
- Field reconnaissance where appropriate.

Types and Numbers of Samples

ERO has requested data on bottom sediments and fish tissue from each waterbody. The rationale for the specific types and numbers of samples to be collected follows.

Bottom Sediments

Sediment samples will be taken of the top10 cm layer. This layer is generally considered to include the biologically active zone (Ecology, 2008; Blakley, 2008).

To enhance representativeness of the data, each sediment sample will be a composite of three separate grabs. The grabs will be taken at three different lake depths: shallow, mid-depth, and maximum depth. The grabs will be placed along a longitudinal transect, with the shallow and mid-depth samples being taken at approximately 1/3 and 2/3 of the maximum lake depth. The samples will be collected away from lake inflows, boat launches, cabins, camp sites, and other such disturbances.

Sediment samples from the upper St. Joe, the only river in the study, will likewise consist of a composite from three separate locations. These will be taken from areas where deposits of sand and finer material occur, selected in such a way as to avoid bank-sloughed material and local tributary inputs. Because bed sediments in a river are continually mixed, it is not critical to adhere to a 10 cm depth increment.

The sediment samples will be analyzed for all target chemicals (see Table 1).

The budget for this project allows for one composite sediment sample to be analyzed for each waterbody. The selected waterbodies would be expected to exhibit relatively uniform chemical conditions. Thus, a single composite sample as planned should be representative of sediment quality in support of this regional assessment. Chemical gradients are potentially generated by sediment from inflows to a lake. This is being taken into account by avoiding sampling at inlets and by compositing from several depths and locations.

Fish Tissue

The study will analyze fish fillets and whole fish. Fillet data are typically more appropriate for comparative assessments associated with human health concerns. Whole fish data are applicable to ecological risk assessment.

An attempt will be made to collect two predator and one bottom-feeder species from each waterbody, with a focus, in part, on popular food fish. This is EPA's recommended approach for initial screening of contaminant levels in lakes and rivers (EPA, 2000). Use of fish from two distinct ecological groups as target species reflects a range of habits, feeding strategies, and physiological factors that can result in differences in bioaccumulation of contaminants. Predators, for example, may have higher levels of chemicals that biomagnify. Bottom-feeders may be elevated in chemicals they come in contact with through the sediments.

Fillets will be analyzed from both predators and bottom-feeders, depending on the number and type of species encountered in a given lake. Bottom-feeding species are preferred for whole body samples due to anticipated higher levels of some of the constituents being analyzed (discussed further below).

Each sample will consist of a composite of pooled tissues from up to five individual fish. Composite samples provide a more cost-efficient estimate of mean contaminant concentrations than single fish samples.

The fish species known to occur in northeastern Washington lakes (excluding very small species) are listed below (Sears, 2010). The species listed in bold font are most commonly analyzed in fish tissue studies in the Pacific Northwest and are preferred for the present study.

Eastern Brook Trout (P)	**Smallmouth Bass** (P)
Rainbow Trout (P)	Pumpkinseed (P)
Brown Trout (P)	Bluegill (P)
Cutthroat Trout (P)	**Yellow Perch** (P)
Kokanee (P)	Tench (B)
Lahontan Cutthroat Trout (P)	**Carp** (B)
Pygmy Whitefish (P)	**Largescale Sucker** (B)
Lake Whitefish (P)	**Bridgelip Sucker** (B)
Mountain Whitefish (upper St. Joe River only)	Peamouth (B)
Northern Pike Minnow (P)	Chiselmouth (B)
Largemouth Bass (P)	Brown Bullhead (B)

P - predator, B - bottom-feeder

Although the species list is relatively long, difficulties are likely to be encountered in obtaining both a predator and a bottom-feeder at all sites. This is especially true for some of the smaller lakes which have been managed as a trout monoculture. Where a bottom-feeder cannot be caught, a predator species should be substituted. Wild fish will be preferred.

Planted fish have been shown to accumulate PCBs and other target chemicals during hatchery rearing (e.g., Serdar et al., 2006). Because the chemical residues may not be representative of the surrounding environment, planted fish will only be analyzed as a last resort and then only if planted as small fish which then resided in that waterbody for at least one year.

Due to the likely event that the desired sample size will not be achieved for all lakes, the field crew should retain three or four species from each lake whenever possible. A decision on which species to analyze and whether fillet or whole will be made after the fish collection is completed.

Differences in chemical concentrations due to age and size class are of interest in this study. Therefore, an effort will be made to collect two distinct size classes when the opportunity presents itself. A decision on which samples to analyze by size class will be made after the fish collection is completed.

The fillets will be analyzed for mercury and the complete suite of organic compounds. Metals analysis of the fillets is being limited to mercury because fish muscle is a poor accumulator for the other metals of concern in this study. Whole fish, on the other hand, are indicators for a range of metals (e.g., Lowe et al., 1985). The metals to be analyzed in whole fish will include arsenic, cadmium, copper, lead, mercury, and zinc.

For budget reasons, organics analyses are not currently planned for whole fish. If fewer fish or sediment samples are collected than planned for the study, organic compounds will be analyzed in selected whole fish samples, with PCBs being the first priority. Excess from all project samples will be archived frozen in the event that other analyses are wanted in the future.

The budget for this project assumes two fish fillet and one whole fish sample will be analyzed for each waterbody. This sample size is comparable to other fish tissue surveys that have assessed levels of chemical contaminants over a large number of waterbodies (Lowe et al., 1985; Schmitt et al., 1990; EPA, 1992, 2009; Seiders and Deligeannis, 2009).

Timing of Sample Collection

ERO requires that this project be completed by July 2011. This stipulation, coupled with the timing of the project request, dictates that the field work be conducted during the September – October 2010 timeframe.

A late summer-fall sampling program is appropriate for this type of study. Seasonality is not a concern for the sediment task. The 10 cm depth increment being analyzed will cover a depositional history spanning multiple years. Most surveys for chemical contaminants in resident Pacific Northwest freshwater fish are conducted during late summer or fall. This is done primarily for reasons of logistics and endangered species concerns (high summer water temperatures), and to include the recreational fishing season (generally April – October for eastside lakes).

The literature does not provide clear and consistent conclusions about seasonal cycles of chemical contaminants in fish. Several researchers have recommended that fish be sampled for mercury during the summer or fall when uptake is most rapid and methylmercury production greatest (e.g., Cope et al., 1990; Slotton et al., 1995). Others have found the highest mercury levels in the spring (Ward and Neumann, 1999).

The organic compounds being analyzed are lipophilic, although many fish tissue studies have failed to find a correlation between bioaccumulative organic compounds and lipid (fat) content (Herbert and Keenleyside, 1995; Stow et al., 1997). During late summer and fall, spring spawners are rebuilding their lipid reserves and the winter spawners are approaching their highest lipid levels. In terms of an overall species average, late summer-fall probably represents a period of generally elevated lipid levels. Percent lipids are being determined for all samples analyzed for organic compounds.

Seasonal differences for metals and organic compounds in fish can often be attributed to an age/size effect. As previously described, the study will obtain some estimates of the importance of these factors by sampling different age classes in several lakes.

Analytical Methods and Detection Limits

Low-level methods will be used to minimize the number of non-detects in the data.

Metals will be analyzed by MEL using inductively-coupled plasma/mass spectrometry (ICP/MS) and cold vapor atomic absorbance techniques (CVAA, mercury). Reporting limits are anticipated to be in the range of 0.005 mg/Kg for mercury and 0.l - 0.5 mg/Kg for other metals, except 2.5 mg/Kg for iron and 5 mg/Kg for zinc. These reporting limits should allow concentrations of target metals to be quantified in all or nearly all sediment and tissue samples (Sloan and Blakley, 2009; Seiders, 2010; Dowling, 2010).

The organic compounds will be analyzed by MEL contract laboratories using high resolution gas chromatography/mass spectrometry (HR-GC/MS). The contract will require detection limits down to 20 ng/Kg for PCBs, 0.03 ng/Kg for PCDDs/PCDFs, and 2 ng/Kg for PBDEs, depending on the congener in question. These are the lowest detection limits currently available through laboratories accredited by Ecology for these methods. Concentrations will be reported down to the detection limit, with concentrations between the detection and quantitation limits being reported as estimates (J flag). Based on other Ecology sediment and fish tissue investigations, a minimal number of non-detects is anticipated (Johnson et al., 2010; Sloan and Blakley, 2009; Seiders, 2010; Dowling, 2010).

Summary of Study Design

Table 3. Summary of Types and Numbers of Samples to be Analyzed.

Sample Type	Number of Waterbodies	Number of Composite Samples	Mercury	Metals (10)*	Metals (5)†	PCBs	PCDDs	PCDFs	PBDEs	TOC	Grain Size	Lipids
Sediment	15	15	✓	✓		✓	✓	✓	✓	✓	✓	
Fish Fillets	15	30	✓			✓	✓	✓	✓			✓
Whole Fish	15	15	✓		✓							

*Sb, As, Ba, Cd, Cr, Cu Fe, Pb, Mn, Zn
†As, Cd, Cu, Pb, Zn

Quality Objectives

Quality objectives for this project are to obtain data of sufficient quality so that uncertainties are minimized and results are comparable to similar data from other studies. Achieving low detection limits is of particular importance for a successful study outcome. These objectives will be achieved through careful attention to the sampling, measurement, and quality control (QC) procedures described in this plan.

Measurement Quality Objectives

MEL and their contractors are expected to meet all QC requirements of the analytical methods being used for this project. Specific measurement quality objectives (MQOs) selected for the project are shown in Tables 4 and 5. These MQOs correspond to MEL's QC limits (metals and ancillary parameters) or the acceptance limits specified in the analytical methods (organic compounds). The lowest concentrations of interest shown in the tables are the lowest currently attainable by MEL and its contract laboratories. Data outside these MQOs will be evaluated for appropriate corrective action.

Table 4. Measurement Quality Objectives: Metals and Ancillary Parameters.
(Analyses by MEL.)

Analysis	Laboratory Control Samples (recovery)	Laboratory Duplicates (RPD)	Matrix Spikes (recovery)	Matrix Spike Duplicates (recovery)	Lowest Concentration of Interest (tissue, ww)	(sediment, dw)
Antimony	85-115%	±20%	75-125%	±20%	NA	0.05 mg/Kg
Arsenic	85-115%	±20%	75-125%	±20%	0.1 mg/Kg	0.1 mg/Kg
Barium	85-115%	±20%	75-125%	±20%	NA	0.1 mg/Kg
Cadmium	85-115%	±20%	75-125%	±20%	0.01 mg/Kg	0.05 mg/Kg
Chromium	85-115%	±20%	75-125%	±20%	NA	0.5 mg/Kg
Copper	85-115%	±20%	75-125%	±20%	0.1 mg/Kg	0.1 mg/Kg
Iron	85-115%	±20%	75-125%	±20%	NA	2.5 mg/Kg
Lead	85-115%	±20%	75-125%	±20%	0.05 mg/Kg	0.05 mg/Kg
Manganese	85-115%	±20%	75-125%	±20%	NA	0.5 mg/Kg
Mercury	80-120%	±20%	75-125%	±20%	0.02 mg/Kg	0.005 mg/Kg
Zinc	85-115%	±20%	75-125%	±20%	5 mg/Kg	5 mg/Kg
Percent Lipids	80-120%	±20%	NA	NA	0.1%	NA
Total Organic Carbon	80-120%	±20%	NA	NA	NA	0.1%
Grain Size	na	±15%	NA	NA	NA	1%

RPD: relative percent difference.
NA: not analyzed or not applicable.
ww: wet weight.
dw: dry weight.
mg/Kg: parts per million.

Table 5. Measurement Quality Objectives: Organic Compounds.
(Analyses by accredited contract laboratories.)

Analysis	Laboratory Control Samples (recovery)	Laboratory Duplicates (RPD)	Labeled Compound Recovery (%)	Lowest Concentration of Interest
PCBs	50-150%	±20%	25-150%*	20 ng/Kg
PCDDs	50-150%	±20%	25-164%†	0.03 ng/Kg
PCDFs	50-150%	±20%	24-169%**	0.03 ng/Kg
PBDEs	50-150%	±20%	25-150%††	2 ng/Kg

*Applies to most congeners, see EPA Method 1668A.
†Applies to 2,3,7,8-TCDD; other congeners as per EPA Method 1613.
**Applies to 2,3,7,8-TCDF; other congeners as per EPA Method 1613.
††Except 20-200% for $^{13}C_{12}$DeBDE; see EPA Method 1614.
RPD: relative percent difference.
ng/Kg: parts per trillion.

Laboratory control samples contain known amounts of analyte and indicate bias due to sample preparation and/or calibration. Results on laboratory duplicates (split samples) provide estimates of analytical precision. Matrix spikes may indicate bias due to matrix effects and provide an estimate of the precision of the results. The organics analyses will be done by isotopic dilution methods where each sample is spiked with labeled congeners. The concentration of target compounds is corrected for recovery of labeled congeners (some congeners are quantitated by an internal standards technique).

Organization and Schedule

The following people will contribute to this project. All are Ecology employees.

Table 6. Organization of Project Staff and Responsibilities.

Staff	Title	Responsibilities
John Roland Toxics Cleanup Program ERO Phone: (509) 329-3581	TCP ERO Client	Clarifies scope of the project. Provides internal review of the QAPP and approves the final QAPP. Reviews project report.
Arianne Fernandez HWTR Program ERO Phone: (509) 329-3498	HWTR ERO Client	Provides internal review of the QAPP and approves the final QAPP. Reviews project report.
Art Johnson Toxics Studies Unit SCS, EAP (360) 407-6766	Project Manager	Writes the QAPP. Oversees field and laboratory work. Conducts QA review of data, analyzes and interprets data. Writes the draft and final report.
Michael Friese Toxics Studies Unit SCS, EAP (360) 407-6737	Field Lead, EIM Data Engineer	Leads field work. Enters project data into EIM.
Casey Deligeannis Toxics Studies Unit SCS, EAP (360) 407-7395	Field Lead	Assists with field work.
Dale Norton Toxics Studies Unit SCS, EAP (360) 407-6765	Unit Supervisor for the Project Manager	Provides internal review of the QAPP. Approves the budget and approves the final QAPP. Reviews project report.
Gary Arnold Eastern Operations Section, EAP (509) 454-4244	Section Manager for the Study Area	Reviews the project scope and budget. Tracks progress, reviews the draft QAPP, and approves the final QAPP.
Stuart Magoon Manchester Environmental Laboratory Phone: (360) 871-8801	Director	Approves the final QAPP.
William R. Kammin Phone: (360) 407-6964	Ecology Quality Assurance Officer	Reviews the draft QAPP and approves the final QAPP.

ERO: Eastern Regional Office.
TCP: Toxics Cleanup Program.
SCS: Statewide Coordination Section.
EAP: Environmental Assessment Program.
HWTR: Hazardous Waste and Toxics Reduction.
EIM: Environmental Information Management database.
QAPP: Quality Assurance Project Plan.

Table 7. Proposed Schedule for Completing Field and Laboratory Work, Data Entry into EIM, and Reports.

Field and laboratory work	Due date	Lead staff
Field work completed	October 2010	Michael Friese
Laboratory analyses completed	February 2011	
Environmental Information System (EIM) database		
EIM user study ID	AJOH0063	
Product	Due date	Lead staff
EIM data loaded	May 2011	Michael Friese
EIM quality assurance	June 2011	Dale Norton
EIM complete	July 2011	Michael Friese
Final report		
Author lead / Support staff	Art Johnson	
Schedule		
Draft due to supervisor	April 2011	
Draft due to client/peer reviewer	May 2011	
Final (all reviews done) due to publications coordinator	June 2011	
Final report due on web	July 2011	

Sampling Procedures

Sediment

Sediment collection and handling will follow the EAP SOP for freshwater sediment samples (Blakley, 2008). The lake samples will consist of composites of three grabs taken with a 0.02 m^2 Ponar sampler. A grab will be considered acceptable if not over-filled with sediment, overlying water is present and not excessively turbid, the sediment surface is relatively flat, and the desired depth penetration has been achieved. Stainless steel scoops will be used to sample sediments from the upper St. Joe River.

For Ponar collected samples, overlying water will be siphoned off and the top 10 cm of sediment removed with a stainless steel scoop, placed in a stainless steel bowl, and homogenized by stirring. Material touching the side walls of the grab will not be taken. The scoop samples from the upper St. Joe will be homogenized in the same way.

Subsamples of the homogenized sediment will be put into 4 oz. glass jars (Teflon lid liners) that have been cleaned to EPA (1990) QA/QC specifications and placed on ice immediately upon collection. The samples will be returned to Ecology headquarters and held frozen until transport with chain-of-custody record to MEL.

Stainless steel implements used to collect and manipulate the sediments will be cleaned by washing with Liquinox detergent, followed by sequential rinses with tap water, deionized water, and pesticide-grade acetone. The equipment will then be air dried and wrapped in aluminum foil. Between-sample cleaning of the Ponar at each lake will consist of thorough brushing with on-site water.

Field data to be collected in conjunction with sediment sampling are to include date, sample site description, latitude and longitude, water depth, penetration depth of the grab, and observations on the type of material obtained.

Fish

Fish will be collected by electroshocking, gill nets, or hook and line, following the EA Program's SOP for fish collection (Sandvik, 2006a). To the extent possible, only those fish large enough to reasonably be retained for consumption will be taken.

Field data to be collected in conjunction with fish sampling are to include date, sample site description, latitude and longitude, and collection method.

Fish selected for analysis will be killed by a blow to the head. Each fish will be given a unique identifying number and its length and weight recorded. The fish will be individually wrapped in aluminum foil, put in plastic bags, and placed on ice for transport to Ecology headquarters, where the samples will be frozen pending preparation of tissue samples.

Tissue samples will be prepared by the study team at Ecology headquarters following the EA Program's SOP for resecting finfish (Sandvik, 2006b). Techniques to minimize potential sample contamination will be used. People preparing the samples will wear non-talc nitrile gloves and work on heavy duty aluminum foil or a polyethylene cutting board. The gloves and foil will be changed between samples; the cutting board will be cleaned between samples as described below.

The fish will be thawed enough to remove the foil wrapper and rinsed with tap water, then deionized water to remove any adhering debris. For fillet samples, the fish will be scaled and the entire fillet from one or both sides removed with stainless steel knives and homogenized to uniform color and consistency in a Kitchen-Aid blender. The fillets will be analyzed skin-on. For whole body samples, the fish will be rinsed with tap water and deionized water and homogenized in a Hobart commercial grinder.

The sex of each fish will be recorded. The homogenates will be placed in precleaned 4 oz. glass jars (Teflon lid liners) cleaned to EPA (1990) QA/QC specifications. The tissue samples will be refrozen for later shipment to MEL. Excess will be retained for all samples and archived frozen at Ecology headquarters.

Each tissue sample will be a composite of up to five individual fish. To the extent possible, the length of the smallest fish in a composite will be no less than 75% of the length of the largest fish (EPA, 2000). The composites will be prepared using equal weights from each fish.

Cleaning of resecting instruments, cutting boards, and blender/grinder parts will be done by washing in tap water with Liquinox detergent, followed by sequential rinses with tap water, de-ionized water, and pesticide-grade acetone. The items will then be air dried on aluminum foil in a fume hood before use.

Sample Analysis

Tables 8 and 9 show the anticipated number of sediment and fish samples to be analyzed, expected range of results, required reporting limits, and analysis methods to be used for the background study.

As previously noted, MEL will analyze metals and ancillary parameters. Organic compounds will be analyzed by accredited contract laboratories selected by MEL.

Table 8. Laboratory Procedures: Sediment Samples.

Analysis	Number of Samples	Expected Range of Results	Reporting Limit	Analytical Method
Antimony	15	0.1-0.5 mg/Kg	0.1 mg/Kg	EPA 200.8
Arsenic	15	1-10 mg/Kg	0.1 mg/Kg	EPA 200.8
Barium	15	50-200 mg/Kg	0.1 mg/Kg	SW 6010
Cadmium	15	0.1-1 mg/Kg	0.1 mg/Kg	EPA 200.8
Chromium	15	10-50 mg/Kg	0.5 mg/Kg	EPA 200.8
Copper	15	1-50 mg/Kg	0.1 mg/Kg	EPA 200.8
Iron	15	5,000-50,000 mg/Kg	2.5 mg/Kg	SW 6010
Lead	15	1-50 mg/Kg	0.1 mg/Kg	EPA 200.8
Manganese	15	100-500 mg/Kg	0.5 mg/Kg	SW 6010
Mercury	15	0.005 - 0.1 mg/Kg	0.005 mg/Kg	EPA 245.5
Zinc	15	20-100 mg/Kg	5 mg/Kg	EPA 200.8
PCBs	15	20 - 200 ng/Kg	20 ng/Kg*	EPA 1668A
PCDDs/PCDFs	15	<0.03 - 1 ng/Kg	0.03 ng/Kg*	EPA 1613B
PBDEs	15	2-20 ng/Kg	2 ng/Kg*	EPA 1614
Total Organic Carbon	15	0.1-10%	0.1%	PSEP-TOC
Grain Size	15	10-90% fines	1%	PSEP (1986)

*Varies with congener.
mg/Kg: parts per million.
ng/Kg: parts per trillion.

Table 9. Laboratory Procedures: Fish Tissue Samples.

Analysis	Number of Samples	Expected Range of Results	Reporting Limit	Analytical Method
Arsenic	15	0.1 - 0.5 mg/Kg	0.1 mg/Kg	EPA 200.8
Cadmium	15	0.05 - 0.1 mg/Kg	0.05 mg/Kg	EPA 200.8
Copper	15	0.1 - 1 mg/Kg	0.1 mg/Kg	EPA 200.8
Lead	15	0.05 - 1 mg/Kg	0.05 mg/Kg	EPA 200.8
Mercury	45	0.02 - 0.5 mg/Kg	0.02 mg/Kg	EPA 245.5
Zinc	15	5-50 mg/Kg	5 mg/Kg	EPA 200.8
PCBs	30	20 - 3,000 ng/Kg	20 ng/Kg*	EPA 1668A
PCDDs/PCDFs	30	<0.03 - 1 ng/Kg	0.03 ng/Kg*	EPA 1613B
PBDEs	30	2-20 ng/Kg	2 ng/Kg*	EPA 1614
Percent lipids	30	0.1-10%	0.1%	MEL SOP #730009

*Varies with congener.
mg/Kg: parts per million.
ng/Kg: parts per trillion.

Quality Control Procedures

Field

The sediment and fish tissue samples for this study are being collected as composites to enhance representativeness of the data.

Duplicate (split) samples will be used to assess variability in the data due to sample preparation and laboratory procedures. Duplicate sediment samples will be prepared in the field at two of the study lakes. Three duplicate fillet samples and two duplicate whole body samples will be prepared when the fish are being resected for chemical analyses.

Laboratory

Laboratory QC samples to be used in assessing the precision and bias of data obtained through this project are shown in Table 10. The samples for duplicate analysis are those prepared by the study team, as described above. Additional laboratory duplicates are not requested.

Table 10. Laboratory Quality Control Samples.

Analysis	Duplicate (split) Samples	Laboratory Control Samples	Method Blanks	OPR Standards	Labeled Compounds
Metals	2-3/batch	1/batch	1/batch	NA	NA
PCBs	2-3/batch	1/batch	1/batch	all samples	all samples
PCDDs/PCDFs	2-3/batch	1/batch	1/batch	all samples	all samples
PBDEs	2-3/batch	1/batch	1/batch	all samples	all samples
Percent Lipids	2-3/batch	1/batch	1/batch	NA	NA
TOC	2/batch	1/batch	1/batch	NA	NA
Grain Size	2/batch	1/batch	NA	NA	NA

OPR: ongoing precision and recovery.
NA: not applicable.

Laboratory Cost Estimate

A cost estimate for the laboratory analyses being conducted for this project is provided in Table 11. The contract laboratory prices shown for organic compounds are based on past studies. Actual prices will be set by bids.

Table 11. Laboratory Cost Estimate.

Matrix/Analysis	Samples	QC	Cost	Subtotals
Sediment				
TOC	15	2	35	595
Grain Size	15	2	90	1,530
Metals (10)	15	2	168	2,856
Mercury	15	2	50	850
PCB Congeners	15	2	800	13,600
PCDDs/PCDFs	15	2	700	11,900
PBDEs	15	2	800	13,600
				44,931
Fish Fillets				
% Lipids	30	3	85	2,805
Mercury	30	3	48	1,584
PCB Congeners	30	3	800	26,400
PCDDs/PCDFs	30	3	700	23,100
PBDEs	30	3	800	26,400
				80,289
Whole Fish				
Mercury	15	2	48	816
As, Cd, Pb, Zn, Cu	15	2	118	2,006
				2,822
			Total Lab =	**$128,042**

These costs include the 50% discount for MEL. MEL's 25% surcharge for contracting and data review is included in the per sample cost for organics.

Data Management Procedures

Field data will be recorded in a bound notebook of waterproof paper.

The data packages from contract laboratories will include case narratives discussing any problems encountered with the analyses, corrective actions taken, changes to the referenced method, and an explanation of data qualifiers. The narrative should address condition of the samples on receipt, holding time, methods of analysis, sample preparation, instrument calibration, recovery data, and results on QC samples. This information is needed to evaluate the accuracy of the data and to determine whether the MQOs were met.

The contract laboratory will provide the sample results to MEL in Excel spreadsheet format and include calculation of PCB homologue totals, total PCBs, and TEQs for PCDDs/PCDFs. TEQs will be calculated using the TEFs (toxic equivalency factors) in Van den Berg et al. (1998).

MEL will provide results and case narratives for their analyses following MEL standard procedures.

All project data will be entered into Ecology's Environmental Information Management System (EIM). Data entered into EIM follow a formal data review procedure where the data are reviewed by the project lead, the person entering the data, and an independent reviewer.

Data Verification

MEL will conduct a review of all contract laboratory data and case narratives. MEL will verify that methods and protocols specified in this QA Project Plan were followed; that all calibrations, checks on quality control, and intermediate calculations were performed for all samples; and that the data are consistent, correct, and complete, with no errors or omissions. Evaluation criteria will include the acceptability of holding times, procedural blanks, calibration, matrix spike recoveries, labeled compound and internal standard recoveries, ion abundance ratios, duplicates, laboratory control samples, and appropriateness of data qualifiers assigned. MEL will prepare written data verification reports based on the results of their data review.

A case narrative will meet the requirements for a data verification report for MEL's chemical data.

The project lead will examine the data reviews, case narratives, and data packages. To determine if project MQOs have been met, results for laboratory control samples, sample duplicates, matrix spikes, and labeled compound recoveries will be compared to QC limits. The method blanks results will be examined to verify there was no significant contamination of the samples. To evaluate whether the targets for reporting limits have been met, the results will be examined for "non-detects" and to determine if any values exceed the lowest concentration of interest. Based on these assessments, the data will be either accepted, accepted with appropriate qualifications, or rejected and re-analysis considered.

Data Analysis

Once the data have been verified, the project lead will determine if they can be used to make the calculations, determinations, and decisions for which the project was conducted. If the results are satisfactory, data analysis will proceed.

Summary statistics will be calculated for each chemical and outliers identified. The data will be plotted to compare contaminant concentrations among waterbodies and identify spatial patterns. If a correlation exists between chemical concentrations and ancillary parameters such as fish lipid content or sediment TOC or percent fines, the data will also be examined on a normalized basis. The fish tissue data will be evaluated for differences among samples analyzed by size class.

Results from other surveys of chemical contaminants in fish and sediment in Washington background or reference areas will be included in the data analysis as appropriate. A comparison will be provided with available human health, aquatic life, and wildlife criteria.

Audits and Reports

Audits

MEL participates in performance and system audits of their routine procedures. Results of these audits are available on request. No audits will be conducted of MEL contract laboratories.

Reports

The following reports will be prepared for this project:

1. A draft technical report for review by ERO and other interested parties; the tentative date for the draft is May 2011. Responsible staff: Art Johnson.
2. The final technical report for this project will be completed by July 2011.
   Responsible staff: Art Johnson.
3. The project data will be entered into EIM on or before July 2011.
   Responsible staff: Michael Friese.

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Appendices

Appendix A. PCDD, PCDF, and PBDE Congeners Being Analyzed in the Northeastern Washington Area Lake Study

Polychlorinated Dibenzo-p-dioxins

2,3,7,8-TCDD
1,2,3,7,8-PeCDD
1,2,3,4,7,8-HxCDD
1,2,3,6,7,8-HxCDD
1,2,3,7,8,9-HxCDD
1,2,3,4,6,7,8-HpCDD

Polychlorinated Dibenzofurans (PCDFs)

2,3,7,8-TCDF
1,2,3,7,8-PeCDF
2,3,4,7,8-PeCDF
1,2,3,4,6,7,8-HpCDF
1,2,3,4,7,8,9-HpCDF
1,2,3,4,7,8-HxCDF
1,2,3,6,7,8-HxCDF
1,2,3,7,8,9-HxCDF
2,3,4,6,7,8-HxCDF
OCDF

Polybrominated Diphenyl Ethers (PBDEs)

2,6-DiBDE
2,2',3,4,4',6-HxBDE
2,2',3,4,4',6'-HxBDE
2,2',3,4,4',5'-HxBDE
2,3,3',4,4',5,-HxBDE
2,2',3,4,4',6,6'-HpBDE
2,2',3,4,4',5',6-HpBDE
2,3,3',4,4',5',6-HpBDE
2,2',3,4,4',5,5'-HpBDE
2,2',3,3',4,4',6-HpBDE
2,2',3,3',4,5',6,6'-OcBDE
2,2',3,3',4,4',6,6'-OcBDE
2,2',3,4,4',5,5',6-OcBDE
2,2',3,3',4,4',5,6'-OcBDE
2,3,3',4,4',5,5',6-OcBDE
2,2',3,3',4,5,5',6,6'-NoBDE
2,2',3,3',4,4',5,6,6'-NoBDE
2,2',3,3',4,4',5,5',6-NoBDE
DeBDE

Appendix B. Acronyms and Units of Measurement

Acronyms

Ecology	Washington State Department of Ecology
EIM	Environmental Information Management database
EPA	U.S. Environmental Protection Agency
ERO	Eastern Regional Office
GIS	Geographic Information System software
HWTR	Hazardous Waste and Toxics Reduction
MEL	Manchester Environmental Laboratory
MQO	Measurement quality objective
PBDEs	Polybrominated diphenyl ethers
PBT	Persistent, bioaccumulative, and toxic substance
PCBs	Polychlorinated biphenyls
PCDDs	Polychlorinated dibenzo-dioxins
PCDFs	Polychlorinated dibenzofurans
QA	Quality assurance
QC	Quality control
RPD	Relative percent difference
SOP	Standard operating procedures
TCP	Toxics Cleanup Program
TOC	Total organic carbon
WDFW	Washington Department of Fish and Wildlife
WQ	Water Quality
WRIA	Water Resources Inventory Area

Units of Measurement

dw	dry weight
mg/Kg	milligrams per kilogram (parts per million)
ng/Kg	nanograms per kilogram (parts per trillion)
ww	wet weight