DEPARTMENT OF
ECOLOGY
State of Washington

Quality Assurance Project Plan

Analyzing Chlorinated Pesticide Residues in Fish from Washington Background Lakes

September 2011

Publication No. 11-03-108

Publication Information

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

The plan for this study is available on Ecology's website at www.ecy.wa.gov/biblio/1103108.html.

Data for this project will be available on Ecology's Environmental Information Management (EIM) website at www.ecy.wa.gov/eim/index.htm. Search User Study ID, AJOH0065.

Ecology's Activity Tracker Code for this study is 12-044.

Waterbody Number: Statewide

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Quality Assurance Project Plan

Analyzing Chlorinated Pesticide Residues in Fish from Washington Background Lakes

September 2011

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Signatures are not available on the Internet version.
EAP: Environmental Assessment Program.
EIM: Environmental Information Management database.

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Abstract

This QA Project Plan is for a study that will characterize chlorinated pesticide residues in edible tissues of fish from background lakes in Washington. The data are needed to: (1) prioritize waterbodies currently listed as water quality limited for fish consumption due to pesticides and (2) determine the potential for new waterbody listings for these chemicals if Ecology adopts human health criteria based on higher fish consumption rates.

Ecology will collect fish from 24 lakes in four regions statewide during the summer and fall of 2011. Approximately 50 composite fillet samples will be analyzed for 29 chlorinated pesticides or breakdown products using low-level methods. The following legacy insecticides are of primary interest: DDT, dieldrin, chlordane, heptachlor, hexachlorobenzene, aldrin, alpha BHC, and toxaphene.

Background

In 2008, the Washington State Department of Ecology (Ecology) conducted a study to assess PCB and dioxin levels in edible tissues of fish from background lakes and rivers in Washington (Johnson et al., 2010). The data were needed to aid in prioritizing waterbodies for water cleanup plans to address PCB and dioxin contamination.

This Quality Assurance (QA) Project Plan is for a study that will collect similar data on chlorinated pesticides. The pesticides of primary interest are the legacy insecticides DDT, dieldrin, chlordane, heptachlor, hexachlorobenzene, aldrin, alpha BHC, and toxaphene. Like PCBs, chlorinated pesticides have become ubiquitous in the environment due to global use, long-term persistence, and bioaccumulation.

Chlorinated pesticides are routinely detected in Washington state freshwater fish (e.g., Seiders and Deligeannis, 2009). There are currently over 100 freshwater listings on Washington's 2010 (proposed) section 303(d) list for pesticide-impaired waterbodies that exceed Washington's human health criteria for fish consumption. Many of the listings are for lakes and rivers with no obvious local sources of these compounds. The Clean Water Act requires that waterbodies on the 303(d) list be cleaned up by pollution control programs or that Total Maximum Daily Loads (TMDLs) be developed (www.ecy.wa.gov/programs/wq/links/wq_assessments.html). A TMDL determines the loading capacity of a waterbody for a pollutant and allocates the load among the various point and non-point sources in the watershed.

Without better information on what constitutes a background level for these contaminants in fish, it is difficult to determine the feasibility and best approach for bringing listed waterbodies into compliance with water quality standards. The information collected through this project will help prioritize the state's resources and accelerate pollution control actions related to chlorinated pesticides in freshwaters statewide. The data will also be useful in evaluating progress toward meeting cleanup targets for waterbodies where pollution control programs or TMDLs have already been established.

Additionally, Ecology's Water Quality Program (WQP) is considering adopting new human health-based water quality criteria for a range of toxic chemicals that include chlorinated pesticides. WQP wants background data to compare with possible criteria concentrations calculated using different fish consumption rates. Implementation of new criteria will be an important part of the rule-making discussion. Background fish tissue data will help determine the potential for new 303(d) listings.

Project Description

The objective of this project is to characterize chlorinated pesticide residues in edible fish tissue from background lakes in Washington. This information will be used in conjunction with existing data to recommend approaches for prioritizing 303(d) listings for these compounds and determining the potential for new listings if Ecology were to adopt human health criteria based on higher fish consumption rates.

Approximately 50 fish samples will be collected from 24 background lakes in four regions of Washington during the summer and fall of 2011. One-to-three species will be sampled in each waterbody, depending on availability. Composite fillets from five to ten individuals of each species will be analyzed for 29 chlorinated pesticides or breakdown products and lipid content. Sensitive analytical methods will be employed to achieve low detection limits.

The study will be conducted by Ecology's Environmental Assessment Program (EA Program). Pesticides will be analyzed by AXYS Analytical Services, Sidney B.C. (tentative) through a contract with the Ecology Manchester Environmental Laboratory (MEL). A final report is anticipated by March 2012. This QA Project Plan follows the Ecology guidance in Lombard and Kirchmer (2004).

Organization and Schedule

Table 1. Organization of Project Staff and Responsibilities.

Staff	Title	Responsibilities
Cheryl Niemi Water Quality Program	Client	Clarifies scope of the project. 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.
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.
Will Kendra Statewide Coordination Section, EAP (360) 407-6698	Section Manager	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 EAP Phone: (360) 407-6964	Ecology Quality Assurance Officer	Reviews the draft QAPP and approves the final QAPP.

EAP: Environmental Assessment Program.
EIM: Environmental Information Management database.
QAPP: Quality Assurance Project Plan.

Table 2. Proposed Schedule.

Field and laboratory work	Due date	Lead staff
Field work completed	July – Oct. 2011	Michael Friese
Laboratory analyses completed	January 2012	
Environmental Information System (EIM) database		
EIM user study ID	AJOH0065	
Product	Due date	Lead staff
EIM data loaded	April 2012	Michael Friese
EIM quality assurance	May 2012	to be determined
EIM complete	June 2012	to be determined
Final report		
Author lead / Support staff	Art Johnson / Michael Friese	
Schedule		
Draft due to supervisor	March 2012	
Draft due to client/peer reviewer	April 2012	
Final (all reviews done) due to publications coordinator	May 2012	
Final report due on web	June 2012	

Human Health Criteria

Ecology's 303(d) listing criteria for edible fish tissue for the pesticides of primary concern in this study are shown in Table 3. The criteria are derived from EPA bioconcentration factors and human health water column criteria established for fish consumption under the EPA National Toxics Rule issued to Washington in 1992 (40 CFR Part 131; Federal Register Vol. 57, No. 246, and as updated). The Table 3 criteria provide a cancer risk protection at the 10^{-6} (one in one million) excess lifetime cancer risk level. The criteria calculations incorporate data for average fish consumption among the general public (6.5 g/day), average adult weight (70 kg), a drinking water ingestion rate of 2 liters of water per day (for freshwater), and an exposure duration of 70 years.

Table 3. Human Health Edible Fish Tissue 303(d) Listing Criteria for Selected Chlorinated Pesticides (ug/Kg, wet weight).

Pesticide Compound	Edible Fish Tissue Criteria
4,4'-DDT	32
4,4'-DDE	32
4,4'-DDD	45
toxaphene	9.6
chlordane	8.3
hexachlorobenzene	6.7
heptachlor	2.4
alpha-BHC	1.7
dieldrin	0.65
aldrin	0.65

Existing Background Data

Several recent studies have obtained data on background concentrations of chlorinated pesticides in Washington freshwater fish. The U.S. Geological Survey (USGS) analyzed organochlorine compounds in fish tissue samples from 14 Washington mountain lakes over 3,000 feet in elevation (Moran et al., 2007). The only chlorinated pesticide detected was DDE, a breakdown product of DDT. The reporting limit in the USGS pesticide analysis was relatively high at 5 ug/Kg wet weight (parts per billion).

The Ecology Washington State Toxics Monitoring Program analyzed fish fillet samples obtained from Upper Twin, Black, Amber, Leo, and Pierre Lakes in northeast Washington in 2009 (Seiders, 2011). This was done in response to a request from the Ecology Eastern Regional Office for data on chemical contaminants in local background lakes. Chlorinated pesticides were not detected in any sample at or above 0.5 ug/Kg, wet weight.

Slightly lower detection limits were achieved in a 2006 survey of chlorinated compounds in fish from the Yakima River basin (Johnson et al., 2007). The following chlorinated pesticide data was obtained on fish from the three upper Yakima River reservoirs on Snoqualmie Pass.

Table 4. Results of Analyzing Chlorinated Pesticides in Fish Fillets from Three Upper Yakima River Reservoirs (Johnson et al., 2007).

Average of 2-3 composite samples' ug/Kg, wet weight; detections in bold font.

Lake	Species	DDE		Dieldrin		Total Chlordane		Alpha-BHC		Toxaphene	
Kachess Lake	Sucker	**0.83**		0.40	U	0.40	U	0.40	U	NA	
"	Pike Minnow	**3.7**		0.40	U	0.40	U	0.40	U	NA	
Keechelus Lake	Sucker	**2.2**		0.38	U	0.38	U	0.40	U	NA	
"	Pike Minnow	**2.6**		0.40	U	0.40	U	0.40	U	NA	
"	Kokanee	**2.2**		0.40	UJ	**0.70**	**J**	0.40	U	NA	
"	Cutthroat	**0.61**		0.39	U	**0.23**	**J**	0.40	U	2.0	U
"	Whitefish	**0.73**		0.39	U	0.39	U	0.40	U	NA	
Cle Elum Lake	Sucker	**7.1**		0.39	U	**0.41**	**J**	0.40	U	5.0	U
"	Pike Minnow	**11**		0.39	U	**0.57**	**J**	0.40	U	5.0	U
"	Whitefish	**10**		0.40	UJ	**2.0**	**J**	0.40	U	2.0	U

U: not detected.
UJ: not detected; detection limit is an estimate.
J: estimated value.
NA: not analyzed.

The above findings suggest that efforts to analyze background levels of chlorinated pesticides in Washington freshwater fish are likely to encounter concentrations lower than 0.5 ug/Kg.

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

AXYS (tentative) is expected to meet all QC requirements of the analysis methods being used for this project. Specific measurement quality objectives (MQOs) selected for the project are shown in Table 5. The pesticide MQOs correspond to the acceptance limits specified in the AXYS High Resolution Gas Chromatography/Mass Spectrometry (HR-GC/MS) method for chlorinated pesticides. The lowest concentrations of interest are the lowest sample specific detection limits reported by AXYS. Data outside these MQOs will be evaluated for appropriate corrective action.

Table 5. Measurement Quality Objectives

Analysis	Laboratory Control Samples (% recovery)	Laboratory Duplicates (RPD)	Labeled Compounds* (% recovery)	Matrix Spikes* (% recovery)	Matrix Spike Duplicates (RPD)	Lowest Concentration of Interest (wet weight)
Chlorinated Pesticides	80-120	≤ 20**	30-150	70-130	≤ 20**	0.02 ug/Kg
Percent lipids	80-120	≤ 20	NA	NA	NA	0.1 %

*varies with pesticide compound, see AXYS Method MLA-028 Rev 6.
**applicable to concentrations >10 times DL.
RPD: relative percent difference.
NA: not applicable.

Study Design

Selection of Background Waterbodies

For purposes of this study, the term *background* denotes a waterbody characterized by near-natural conditions or minimal influence by local human activities. The study will focus on lakes rather than rivers because of the low diversity of fish species in most rivers that might qualify as background and the great ability of fish to move into and out of rivers as opposed to lakes. Lakes also tend to be less impacted by human development due to the location of towns, industry, and their associated discharges.

Potential background areas were selected by examining Washington state maps and GIS coverages showing population density, agricultural land use, public lands, annual precipitation, and wind direction. This exercise identified locations that had a low probability of significant local sources of contamination.

Biologists and resource managers within the Washington Department of Fish and Wildlife, National Park Service, U.S. Forest Service, and Ecology were asked to identify potential background lakes within these areas, using the following criteria:

- Elevation under approximately 3,000 feet.
- Watershed relatively undisturbed except of past logging.
- At least two resident fish species of catchable size.
- Good accessibility.

Based on the mapping exercise and recommendations, six lakes were tentatively selected for sampling in each of four regions: Western Washington, West Slope of the Cascades, East Slope of the Cascades, and Eastern Washington. The lakes were grouped by region in view of the potential for different rates of chemical deposition due to proximity to the Pacific Ocean air mass, urban/industrial sources around Puget Sound, and effects of the Cascade Range.

The appropriateness of each lake for inclusion in the background study was checked against Ecology's Facility Site Identification System (http://www.ecy.wa.gov/fs). This system identifies sites known to Ecology as having an active or potential impact on the environment. Table 6 lists 24 waterbodies proposed for sampling; Figure 1 shows their locations.

An effort was made to distribute the sampling effort along a north-south gradient within each of the four regions. The waterbodies selected include a mix of impoundments and natural waterbodies of various sizes, as is the case with the 303(d) list. It was difficult to locate appropriate background lakes within the major agricultural basins of Southeast Washington. Efforts to identify useful background lakes in these areas continue. If efforts are successful, the sampling design will be altered slightly to accommodate one or two lakes in this region.

While pristine, high mountain lakes obviously qualify as background, they are not being included in this study because of enhanced atmospheric deposition of synthetic organic compounds due to colder temperatures and larger amounts of precipitation (Gillian and Wania, 2005; Blais et al., 1998; Wania and Mackay, 1993). For example, in the previously mentioned USGS study of high mountain lakes in Washington, approximately 20% of the fish samples had total PCB concentrations estimated at 17 – 20 ug/Kg, higher levels than in a number of the waterbodies on the 303(d) list and well above current listing criteria (5.3 ug/Kg). High lakes have the additional drawbacks of difficult access and low fish diversity.

Table 6. Lakes Tentatively Selected for Sampling.

Region and Name	Surrounding Area	County	Lake Elevation (ft)	Lake Area (acres)	Max. Depth (ft)	Lat.	Long.
Western Washington							
Ozette Lake	Olympic NP	Clallam	29	7,787	331	48.100	124.640
Tarboo Lake	Olympic Peninsula	Jefferson	642	24	58	47.924	122.852
Cushman Lake	Olympic NF	Mason	731	4,003	115	47.470	123.250
Wynoochee Lake	Olympic NF	Grays Harbor	800	1,120	175	47.405	123.587
Devereaux Lake	Kitsap Peninsula	Mason	215	100	50	47.405	122.848
Silver Lake	Seaquest SP	Cowlitz	485	2996	10	46.290	122.792
West Slope Cascades							
Baker Lake	N. Cascade NP	Whatcom	724	3,616	283	48.720	121.660
Cavanaugh Lake	Baker-Snoqualmie NF	Skagit	1008	844	80	48.322	122.013
Spada Lake	Baker-Snoqualmie NF	Snohomish	1,435	1,870	?	47.970	121.650
Chester Morse Lake	Baker-Snoqualmie NF	King	1,555	1,682	116	47.390	121.700
Coldwater Lake	St. Helens National Monument	Cowlitz	~2,500	700	?	46.303	122.239
Merrill Lake	Lewis River basin	Clark	1,541	344	60	46.090	122.330
East Slope Cascades							
Patterson Lake	Okanogan NF	Okanogan	2,740	130	85	48.460	120.240
Wenatchee Lake	Wenatchee NF	Chelan	2,257	513	300	47.830	120.700
Cle Elum Lake	Wenatchee NF	Kittitas	2,224	4,810	140+	47.290	121.110
Bumping Lake	Wenatchee NF	Yakima	3,426	1,310	89	46.850	121.320
Clear Lake	Wenatchee NF	Yakima	3,615	265	54	46.630	121.280
Walupt Lake	Goat Rocks Wilderness	Lewis	4,000	384	295	46.417	121.464
Eastern Washington							
Leo Lake	Colville NF	Pend Oreille	2,588	39	37	48.910	118.130
Swan Lake	Colville NF	Ferry	3,641	52	95	48.512	118.839
South Twin Lake	Colville NF	Ferry	2,572	973	57	48.264	118.387
Buffalo Lake	Colville Reservation	Okanogan	954	3,244	121	48.280	119.400
Turnbull Lake	Turnbull NWR	Spokane	~2,300	361	shallow	47.440	117.590
Evergreen Lake	Quincy Wildlife Area	Grant	~1,000	235	54	47.140	119.920

NP: National Park
NF: National Forest
SP: State Park
NWR: National Wildlife Refuge

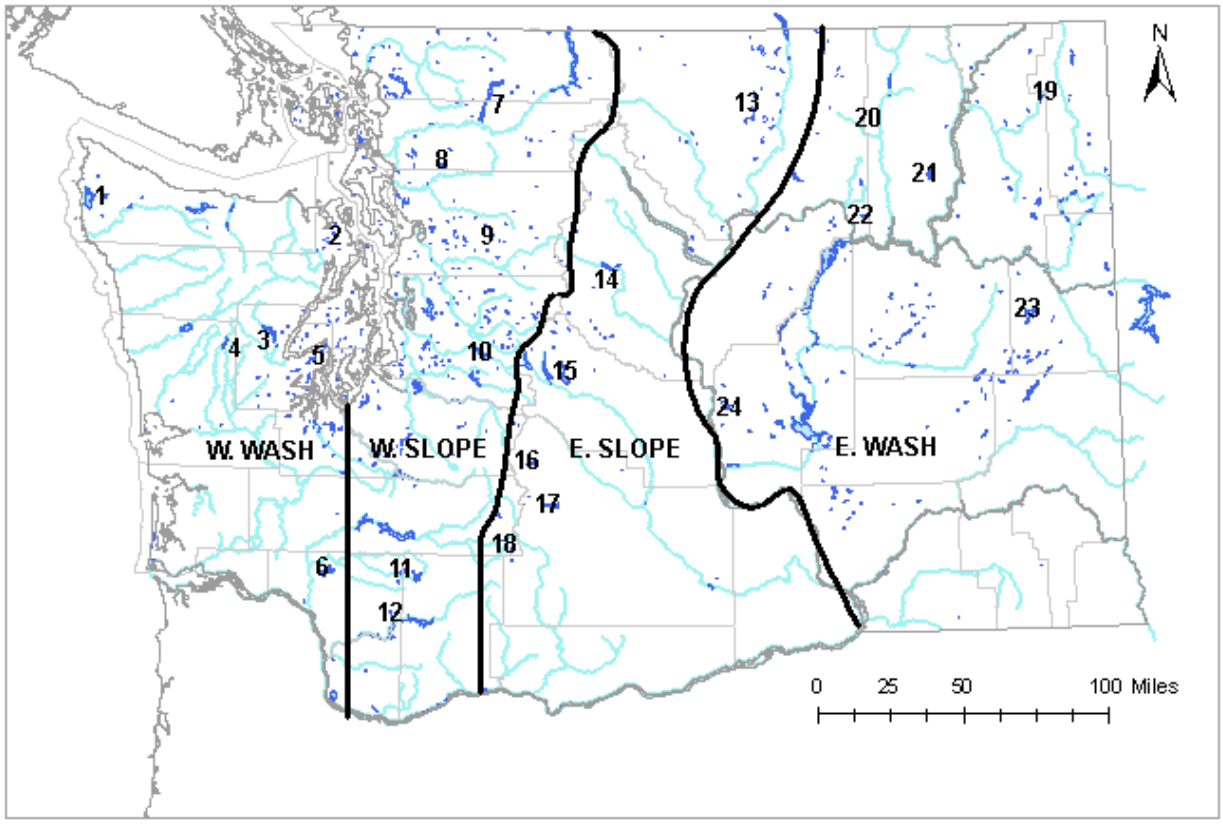

Figure 1. Lakes Tentatively Selected for Sampling

Key to Figure 1:

Western Washington	West Slope Cascades	East Slope Cascades	Eastern Washington
1 = Ozette Lake	7 = Baker Lake	13 = Patterson Lake	19 = Leo Lake
2 = Tarboo Lake	8 = Cavanaugh Lake	14 = Wenatchee Lake	20 = Swan Lake
3 = Cushman Lake	9 = Spada Lake	15 = Cle Elum Lake	21 = South Twin Lake
4 = Wynoochee Lake	10 = Chester Morse Lake	16 = Bumping Lake	22 = Buffalo Lake
5 = Devereaux Lake	11 = Coldwater lake	17 = Clear Lake	23 = Turnbull NWR
6 = Silver Lake	12 = Merrill Lake	18 = Walupt Lake	24 = Quincy Wildlife Area

Fish Samples

This study will target the larger fish species more likely to be consumed and on which most of the 303(d) listings for Washington are based. The species of primary interest are as follows:

- brook trout (*Salvelinus fontinalis*)
- carp (*Cyprinus carpio)*
- cutthroat trout (*Oncorhynchus clarki*)
- kokanee (*Oncorhynchus nerka*)

- largemouth bass (*Micropterus salmoides*)
- largescale suckers (*Catostomus macrocheilus*)
- mountain whitefish (*Prosopium williamsoni*)
- rainbow trout (*Oncorhynchus mykiss*)
- smallmouth bass (*Micropterus dolomieu*)
- yellow perch (*Perca flavescens*)

Based on past experience, it is anticipated that one-to-three species could be collected from each lake. An effort will be made to collect at least one predator and one bottom feeder from each site, as recommended by EPA (2000). No planted fish will be analyzed, unless planted as fingerlings.

Fish will be collected primarily during the late summer and fall of 2011. Due to endangered species concerns related to high summer water temperatures, most of 303(d) listings are based on fish surveys conducted around this timeframe. Lipid content of fall spawners is increasing at this time and spring spawners are rebuilding their lipid reserves. Chlorinated pesticides are primarily associated with lipids.

Fillets will be analyzed for all fish samples. Each sample will consist of a composite of pooled tissues from five to ten individual fish, depending on size. Composite samples provide a more cost-efficient estimate of mean contaminant concentrations than single fish samples. There will be one composite per species from each lake. To provide estimates of the effect fish size might have on the results, two size ranges will be analyzed for several lakes, as the opportunity presents itself. Length, weight, and sex will be recorded for each fish used in the composites.

Chemical Analysis

The fish tissue samples will be analyzed for 29 chlorinated pesticides or breakdown products (Appendix A) and percent lipids. HR-GC/MS methods will be used for pesticides to give low detection limits of 0.02 – 0.05 ug/Kg.

Table 7 shows sample size for this project and an estimate of laboratory costs. The cost estimate includes MEL's 25% surcharge for contract laboratory services. The cost per sample for pesticides is preliminary; final cost will be based on the laboratories responding to MEL's Request for Qualifications and Quote.

Table 7. Sample Size and Laboratory Cost Estimate.

Number of Waterbodies*	Ave. No. Samples per Waterbody	QC Split Samples	Total Samples†	Lab Cost (per sample)		Total Cost
				Chlorinated Pesticides	Percent Lipids	
24	2	3	51	$900	$31	$58,956

*Six lakes in each of four regions.
† Two QC split samples assumed to be free of charge.

Representativeness, Comparability, and Completeness

The intent of this sampling design is to obtain representative data on background concentrations of chlorinated pesticides in fish from Washington lakes, excluding high elevation waterbodies, as previously explained. Steps being taken to enhance representativeness include use of appropriate sampling and sample handling procedures, use of composite samples, and a statewide sampling network where a range of species and waterbody types are being sampled.

The field and laboratory methods being used are the same as or similar to recent Ecology studies of chemical contaminants in Washington freshwater fish.

The completeness goal for this project is to have valid, defensible data for all samples collected.

Sampling Procedures

Fish Collection

Fish sampling will follow the EA Program's Standard Operating Procedure (SOP) (Sandvik, 2006a). Fish will be collected by electroshocking, gill net, or hook and line. Only legal size fish will be taken. For species with no size limits, only those large enough to reasonably be kept for consumption will be retained.

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, labeled with date, species, and lake name, and placed on ice for transport to Ecology headquarters, where the samples will be frozen pending preparation of tissue samples.

Tissue Preparation

Tissue samples will be prepared following the EA Program's SOP (Sandvik, 2006b). Techniques to minimize potential for 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, scaled, and rinsed with tap water and then de-ionized water. The entire fillet from one or both sides of each fish will be removed with stainless steel knives and homogenized in a Kitchen-Aid blender. The fillets will be analyzed with the skin on.

Five to ten individual fish will be used for each composite sample. 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. The pooled tissues will be homogenized to uniform color and consistency, using a minimum of three passes through the blender. The homogenates will be placed in glass jars with Teflon lid liners which have been cleaned to EPA (1990) QA/QC specifications.

Cleaning of resecting instruments, cutting boards, and blender 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 fumehood before use.

The tissue samples will be refrozen for shipment with chain-of-custody record to MEL. The samples for pesticide analysis will be stored frozen at MEL until shipped to the contract laboratory. Excess tissue will be retained for all samples and stored frozen at Ecology Headquarters. Table 8 summarizes sample containers, preservation, and holding times.

Table 8. Sample Containers, Preservation, and Holding Time.

Parameter	Container	Preservation	Holding Time
Chlorinated Pesticides	4 oz. glass w/ Teflon lid liner	Freeze at -18°C	1 year (frozen)
Lipids	4 oz. glass w/ Teflon lid liner	Freeze at -18°C	NA

Measurement Procedures

Table 9 shows the number of samples to be analyzed, expected range of results, required reporting limits, and sample preparation and analysis methods.

Table 9. Laboratory Procedures.

Analysis	Number of Samples*	Expected Range of Results	Reporting Limit†	Analytical Method	Laboratory
Chlorinated Pesticides	51	≤0.01-10 ug/Kg	0.01 ug/Kg	MLA-028**	AXYS
Percent lipids	52	0.1-10%	0.1%	extraction	AXYS

*includes three duplicate samples.
†varies with analyte, see Appendix A.
**AXYS in-house method.

AXYS (tentative) will report down to the detection limit and flag concentrations between the detection and quantitation limit (lowest method calibration level or LMCL as estimates (see Appendix A).

Ecology has not accredited any laboratories to analyze chlorinated pesticides by HR-GC/MS, a relatively new application of this method. A waiver has been requested from the Ecology QA Officer to use AXYS in-house method MLA-028 for this project. The Request for Laboratory Services (Appendix B) details additional reporting requirements for AXYS' pesticide analysis and data reporting.

Quality Control Procedures

Field

No field QC samples are planned for this project.

Laboratory

Laboratory QC samples to be analyzed for this project are shown in Table 10.

Table 10. Laboratory Quality Control Samples.

Analysis	Duplicates	Laboratory Control Samples	Method Blanks	Ongoing Precision and Recovery Standards	Labeled Compounds
Chlorinated Pesticides	2/batch	1/batch	2/batch	all samples	all samples
Percent Lipids	2/batch	1/batch	1/batch	NA	NA

NA: not applicable.

For the laboratory duplicates, four composites will be split and analyzed separately. The duplicates will be prepared by the study team and submitted blind.

Data Management Procedures

Field data and data from preparation of tissue samples will be recorded in a bound notebook of waterproof paper.

The data packages from the laboratories will include case narratives discussing any problems encountered in 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, 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 laboratories will provide the sample results in Excel spreadsheet format. 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 laboratory data and case narratives. MEL will verify that:

- Methods and protocols specified in this QA Project Plan were followed.
- All calibrations, checks on quality control, and intermediate calculations were performed for all samples.
- Data are consistent, correct, and complete, with no errors or omissions.

Evaluation criteria will include, as appropriate, the acceptability of holding times, calibration, internal standard recoveries, labeled compound recoveries, ion abundance ratios, procedural blanks, ongoing precision and recovery, laboratory control samples, procedural blanks, matrix spikes, and appropriateness of data qualifiers assigned. MEL will prepare written data verification reports based on the results of their data review. A case summary will meet the requirements for a data verification report.

To determine if project MQOs have been met, results for laboratory control samples, laboratory duplicates, ongoing precision and recovery, matrix spikes, and labeled compound recoveries will be compared to QC limits. The method blank 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.

The project lead will review the laboratory data packages and MEL's data verification report. Based on these assessments, the data will be either accepted, accepted with additional qualification, or rejected and re-analysis considered.

Data Usability Assessment

Once the data have been verified, the project lead will determine if the data 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 tabulated for each parameter. The data will be plotted to compare contaminant concentrations between sampling region and species and to identify exceedances of current human health criteria. If a correlation exists between chemical concentrations and lipid content, the data will be normalized to percent lipids and re-examined for regional and species differences. Outliers will be identified.

Data from other recent fish tissue surveys in Washington that have analyzed chlorinated pesticides will be compiled The distribution of values in the total data set for chlorinated pesticides in Washington freshwater fish will be examined to identify and prioritize concentration levels for pollution control programs or TMDLs.

Audits and Reports

Audits

No laboratory audits are planned for this project.

Reports

The following reports will be prepared for this project:

1. A draft technical report for review by the Ecology Water Quality Program and other interested parties. The tentative date for the draft is April 2012. The responsible staff member is Art Johnson.
2. A final technical report is anticipated in May 2012. The responsible staff member is Art Johnson.
3. The project data will be entered into EIM on or before May 2012. The responsible staff member is Michael Friese.

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Appendices

Appendix A. Chlorinated Pesticides to be Analyzed in Fish Tissue Samples (tentative)

AXYS Analytical Services Ltd.

TYPICAL DETECTION LIMITS, METHOD DETECTION LIMITS (MDL)AND LOW CALIBRATION LIMITS for OC Pesticides by GC/HRMS

AXYS Method: MLA-028
Instrument Type: High Resolution GC/MS
MDL Protocol: Federal Register 40 CFR Part 136, Appendix B, no iteration

Matrix:		Tissue		
Units/Sample Size:		ng/g based on 10 g sample		
Analytes		Typical Detection Limit/SDL	MDL	LMCL based on Low Cal.
E1 Pesticides [2]				
Hexachlorobenzene		0.01	0.008	0.2
HCH, alpha		0.02	0.022	0.4
HCH, beta		0.02	0.010	0.4
HCH, gamma		0.02	0.139	0.4
Heptachlor		0.02	0.013	0.2
Aldrin		0.02	0.017	0.4
Chlordane, oxy-		0.02	0.030	0.4
Chlordane, gamma (trans)		0.02	0.011	0.4
Chlordane, alpha (cis)		0.02	0.017	0.4
Nonachlor, trans-		0.02	0.012	0.4
Nonachlor, cis-		0.02	0.023	0.4
2,4'-DDD		0.02	0.016	0.2
4,4'-DDD		0.02	0.018	0.2
2,4'-DDE		0.02	0.007	0.2
4,4'-DDE		0.02	0.008	0.2
2,4'-DDT		0.02	0.009	0.2
4,4'-DDT		0.02	0.013	0.2
Mirex		0.02	0.004	0.2
Technical Toxaphene		0.05	NA	111*
E2 Pesticides				
HCH, delta		0.05	0.012	0.2
Heptachlor Epoxide		0.05	0.009	0.16
alpha-Endosulphan		0.05	0.035	0.16

Dieldrin		0.05	0.018	0.16
Endrin		0.05	0.009	0.16
beta-Endosulphan		0.05	0.050	0.16
Endosulphan Sulphate		0.05	0.015	0.16
Endrin Aldehyde		0.05	0.017	0.16
Endrin Ketone		0.05	0.017	0.16
Methoxychlor		0.10	0.010	0.16

*Not a true LMCL; reflects effect of summing LMCLs for multiple toxaphene congeners.

Appendix B. Request for Laboratory Services for High Resolution Chlorinated Pesticide Analysis of Fish Tissue Samples. (Example, to be replaced by actual.)

REQUEST FOR LABORATORY SERVICES

PPR #. pr-32952-11
Quote: **RFQQ 1030**

Contact	Project	Date
Dave Hope	**ERO Lakes Study - Fish tissue**	**5/31/2011**
Laboratory **Pacific Rim Laboratories**	Client and Address:	☐ Enforcement
Address **#103, 19575 - 55A Avenue** **Surrey, BC V3S 8P8** **Canada**	**WA State Dept. of Ecology** **Manchester Laboratory** **7411 Beach Drive East** **Port Orchard, WA**	☐ Return to Client ☐ Dispose ☒ **Please** Return Cooler and Blue Ice!
Phone: **(604) 532-8711** FAX: (604) 532-8712	**98366-8204**	

ITEM NO.	SAMPLE NO.	PROJECT NAME AND/OR DESCRIPTION	QUANTITY	UNIT PRICE	TOTAL COST
1	**1105039-**	Perform analyses on these fish for: PCB congeners by EPA method 1668A for all 209 congeners; Dioxin/Furans by EPA method 1613B; and for PBDE congeners by EPA method 1614.		$684 $555 $600	$40,458.00
2	**1105039-**	Perform duplicate analyses on these samples for: PCB congeners, Dioxin/Furans, and PBDE congeners.	1 1 1	NC NC NC	NC NC NC
3		See Item 6D and Tables 1 and 2 for reporting limits.			
4		Perform lipids analysis on all samples, including duplicates.			
5		Deliverables shall include copies of all raw data necessary to perform an independent evaluation of the results, including, but not limited to initial calibration and verification standards, sample and QC chromatograms and spectra, benchsheets, etc. See following pages for more details on reporting.			
6		Deliverables are due within 30 calendar days from the date of receipt of samples.			
7		In addition to hard copies, submit results in Excel format according to items 7 on page 3.			
		NC = no charge		TOTAL	$40,458.00

Requested By (Your contact if any questions arise): **Karin Feddersen** Phone No. **(360) 871-8829**

CHAIN OF CUSTODY*

Relinquished By:	Received By:	Yr	Mo	Day	Hr	Min

*Signatures on this part of the form pertain to the custody of these samples and not to the cost of the analysis.
Invoice will be paid after sample analyses have passed a QA/QC review.

Items for sample analysis:

Section **9.5.1** of all three methods states "Analyze the blank immediately after analysis of the OPR (Section 15.5) to demonstrate freedom from contamination." However, we would prefer the lab not analyze the method blank directly after the calibration curve. Instead, please **analyze a solvent blank** after the calibration curve and after any highly contaminated samples that are suspected may cause carry-over to other samples.

Reporting of results:

1. Copy of the "Request for Laboratory Services", with signed and dated Chain of Custody section.

2. Case narrative and corrective action reports.

3. Description of: analytical method used; any modifications to the method, QA/QC performed and results; definitions of all data qualifiers used; and any other information that helps client understand the data package. For lipids, report the extraction solvents and ratios used.

4. A fully bookmarked PDF file on CD and/or paginated hardcopies of all raw data with a table of contents.

    A. Provide Tier IV Deliverables package: Deliverables shall include copies of all raw data necessary to perform an independent evaluation of the results, including, but not limited to initial calibration and verification standards, sample and QC chromatograms and spectra, analytical sequence (run) logs, benchsheets, etc.

    B. Clearly identify all field and QC samples with the sample number or QC name in the raw data and report.

    C. All initial calibration (ICAL) standards, CCVs, and the 209 standard shall be clearly identified in the raw data. For example: CS0, CS1, etc., for the ICAL.

5. Define all qualifiers and acronyms used in the data package in order to prevent confusion.

6. Reporting Limits: Estimated Detection Limits, Method Detection Limits, and Estimated Quantitation Limits.

    A. Provide the Estimated Quantitation Limit (EQL); aka: Practical Quantitation limit or PQL - based on the lowest validated standard in calibration curve) for each result. Report the EQL in the electronic file in the "Result Value EQL" column.

    B. Provide the most recent Method Detection Limit (MDL) study results, and date of study, for each analyte.

C. Provide the Estimated Detection Limits (EDL) or Sample Detection Limits (SDL) based on signal-to-noise ratio for High Resolution Mass Spectrometry (HRMS) analyses.

D. Reporting Limits (RL):

a. RLs are defined below. Report on a wet weight basis for fish and a dry weight basis for sediment.

b. For individual PCB congeners, Reporting Limits (RL) for individual congeners are to be equal to the EQLs, 20 – 80 ng/kg, depending on the congener.

c. See tables below for Dioxin/Furan and PBDE RLs.

Table 1. Required Method Detection Limit for Dioxin/Furan Analyses.

Dioxin/Furan Congener	TEFs: WHO 2005	Required Method Detection Limit (ng/kg)
2,3,7,8-TCDD	1	0.03
1,2,3,7,8-PeCDD	1	0.03
2,3,4,7,8-PeCDF	0.3	0.1
1,2,3,4,7,8-HxCDD	0.1	0.1
1,2,3,4,7,8-HxCDF	0.1	0.1
1,2,3,6,7,8-HxCDD	0.1	0.1
1,2,3,6,7,8-HxCDF	0.1	0.1
1,2,3,7,8,9-HxCDD	0.1	0.1
1,2,3,7,8,9-HxCDF	0.1	0.1
2,3,4,6,7,8-HxCDF	0.1	0.1
2,3,7,8-TCDF	0.1	0.1
1,2,3,7,8-PeCDF	0.03	0.1
1,2,3,4,6,7,8-HpCDD	0.01	0.2
1,2,3,4,6,7,8-HpCDF	0.01	0.2
1,2,3,4,7,8,9-HpCDF	0.01	0.2
1,2,3,4,6,7,8,9-OCDD	0.0003	0.5
1,2,3,4,6,7,8,9-OCDF	0.0003	0.5

Note that the criterion we use for fish tissue is 0.07 ng/kg so we need reporting limits below this for congeners with higher Toxicity Equivalent Factors (TEFs) of 1 and 0.3.

Table A-2. Brominated Diphenyl Ethers.

Congeners	PBDE	RL ng/kg
2,6-DiBDE	10	2
2,4-DiBDE	7	2
4,4'-DiBDE	15	2
2,4,6-TrBDE	30	2
2,2',4-TrBDE	17	2
2,4,4'-TrBDE	28	2
2,2',4,5'-TeBDE	49	2
2,3',4',6-TeBDE	71	2
2,2',4,4'-TeBDE	47	5
2,3',4,4'-TeBDE	66	2
3,3',4,4'-TeBDE	77	2
2,2',4,4',6-PeBDE	100	5
2,3',4,4',6-PeBDE	119	2
2,2',4,4',5-PeBDE	99	5
2,2',3,4,4'-PeBDE	85	2
3,3',4,4',5-PeBDE	126	2
2,2',4,4',5',6-HxBDE	154	4
2,2',4,4',5,5'-HxBDE	153	4
2,2',3,4,4',6-HxBDE	139	4
2,2',3,4,4',6'-HxBDE	140	4
2,2',3,4,4',5'-HxBDE	138	4
2,3,3',4,4',5,-HxBDE	156	4
2,2',3,4,4',6,6'-HpBDE	184	4
2,2',3,4,4',5',6-HpBDE	183	4
2,3,3',4,4',5',6-HpBDE	191	4
2,2',3,4,4',5,5'-HpBDE	180	4
2,2',3,3',4,4',6-HpBDE	171	4
2,2',3,3',4,5',6,6'-OcBDE	201	4
2,2',3,3',4,4',6,6'-OcBDE	197	4
2,2',3,4,4',5,5',6-OcBDE	203	4
2,2',3,3',4,4',5,6'-OcBDE	196	4
2,3,3',4,4',5,5',6-OcBDE	205	4
2,2',3,3',4,5,5',6,6'-NoBDE	208	10
2,2',3,3',4,4',5,6,6'-NoBDE	207	10
2,2',3,3',4,4',5,5',6-NoBDE	206	10
DeBDE	209	25

d. Qualify detected values that lie between the RL and the EQL as estimates ("J").

e. For non-detect values: Record the reporting limit value in the "Result Reported Value" column and a "U", or "UJ" if appropriate, in the "Result Data Qualifier" column.

E. Qualify results that do not meet the ion abundance ratio criteria. Calculate and report the Estimated Maximum Possible Concentration (EMPC) value.

F. Qualifiers above are defined as:

I. "J" – The analyte was positively identified. The associated numerical result is an estimate.

II. "U" – The analyte was not detected at or above the reporting limit.

III. "UJ" – The analyte was not detected at or above the reported estimated reporting limit.

7. Sample identification:

A. Provide the client sample ID (Manchester Laboratory ID) and Field ID associated with all sample results as appropriate.

B. Provide the lab's internal sample ID associated with all results OR a table that cross-references MEL lab ID with the lab's internal sample ID.

D. Clearly identify QA/QC samples and results: blanks, SRMs, lab duplicates. If samples are reanalyzed, these results need be clearly identified as such.

E. Label analyte peaks on chromatograms with either the congener name or the retention time and scale chromatograms such that peaks are visible above the baseline.

8. Analyte identification:

A. Provide the CAS RN for all analytes.

B. PCB congeners use number using the BZ_1992 numbering convention modified to a 7-character format that uses leading zeroes for congener numbers below 100 (e.g. PCB-008). (Conversely, the value "PCB-001" appears to have 7 characters yet actually has 11 since there are 4 spaces after the 001. This complicates export into databases and statistical packages.)
See www.epa.gov/osw/hazard/tsd/pcbs/pubs/congeners.htm

C. Co-eluting congeners should be numbered in ascending order (e.g.: PCB-040/041/071), and records for co eluting congeners must have no CAS number.

D. Name PCB congeners using the IUPAC naming convention.

9. QC Analyses.

A. Perform all QC samples as specified in the method.

B. Provide results of Duplicates and Laboratory Control Samples (On-going Precision and Recovery standards) and labeled compounds, internal standards, and surrogates as % recoveries.

C. Samples for laboratory duplicate and matrix spike analyses may be designated by Ecology.

D. Provide results of certified reference material using the same units as for the samples. Provide a copy of the Certificate of Analysis.

10. Blanks.

A. Total homologs for the in-house method blank must not exceed the limits below. If these limits are exceeded, contact Ecology's Project Manager to discuss actions to take. Most likely, any blanks with individual results greater than half the EQL should be re-extracted along with any associated samples.

B. For PCB congeners, the "Total PCB" value in the method blank must not exceed 0.3 ug/kg.

C. For dioxin/furan congeners, the value of individual congeners found in the method blank must not exceed the congener-specific MDLs in Table 1 with the following exception:

TCDD, PeCDD	0.05 ng/kg

D. If sample results are less than 10 times the blank concentration; qualify sample results with "B". Discuss in the narrative whether these qualified results are included in total homologs or total PCBs results.

E. Clearly identify samples associated with each blank.

11. Treatment of result qualifiers and summing of PCB homologs.

A. Describe any qualifiers used.

B. Describe in the case narrative how totals were derived for PCB homolog groups and total PCBs (e.g. what rules are used for rounding values, dealing with non-detects, blank detects, qualifier definitions, etc.).

C. Report Total homolog results in EDD. However, do not report an EQL (i.e.: leave EQL column blank for summed values).

12. Electronic results must be in Excel-compatible format as in Table 3.

Table 3. Required Fields for Electronic Data Package from PBDEs, PCB Congeners, and Dioxins/Furans Analyses.		
Preferred Order	**Field Name**	**Example**
1	MEL (Client) Sample ID	1006021-03 (format as text)
2	Result IUPAC Name	2,3'-DiCB
3	Result Parameter Name (for PCB congeners only)	PCB-006
4	Result Parameter CAS Number	25569-80-6
5	Sample Extraction Date	11/1/09 (format as numerical date)
6	Sample Analysis Date	11/2/09 (format as numerical date)
7	Lab Duplicate Flag	"Y" if lab duplicate, blank or "N" if not
8	Re-analysis Flag	"Y" if a re-analysis, blank or "N" if not
9	Result Reported Value	0.4 (format as number)
10	Result Data Qualifier	J
11	Result Value UOM	ng/Kg
12	Result Value EQL *	20 (format as number)
13	Result Value Detection Limit**	0.2 (format as number)
14	Result Method Code	EPA 1668A
15	Result Lab Name	Laboratory Name
16	Contract Lab Sample ID	PR07954
17	Others as needed by contract lab or MEL.	If used, clearly identify field and content
	* = Estimated Quantitation Limit (Based on the lowest validated standard in the calibration curve and adjusted for weight, volume, % solids, etc., as applicable).	
	** = State which detection limit used in Case Narrative (e.g. Method Detection Limit, Estimated Detection Limit).	

Appendix C. Glossary, Acronyms, and Abbreviations

Glossary

Benchsheet: Method-specific worksheets.

Clean Water Act: A federal act passed in 1972 that contains provisions to restore and maintain the quality of the nation's waters. Section 303(d) of the Clean Water Act establishes the TMDL program.

Nonpoint source: Pollution that enters any waters of the state from any dispersed land-based or water-based activities. This includes, but is not limited to, atmospheric deposition, surface-water runoff from agricultural lands, urban areas, or forest lands, subsurface or underground sources, or discharges from boats or marine vessels not otherwise regulated under the NPDES program. Generally, any unconfined and diffuse source of contamination. Legally, any source of water pollution that does not meet the legal definition of "point source" in section 502(14) of the Clean Water Act.

Point source: Sources of pollution that discharge at a specific location from pipes, outfalls, and conveyance channels to a surface water. Examples of point source discharges include municipal wastewater treatment plants, municipal stormwater systems, industrial waste treatment facilities, and construction sites that clear more than 5 acres of land.

Total Maximum Daily Load (TMDL): A distribution of a substance in a waterbody designed to protect it from not meeting (exceeding) water quality standards. A TMDL is equal to the sum of all of the following: (1) individual wasteload allocations for point sources, (2) the load allocations for nonpoint sources, (3) the contribution of natural sources, and (4) a margin of safety to allow for uncertainty in the wasteload determination. A reserve for future growth is also generally provided.

303(d) list: Section 303(d) of the federal Clean Water Act requires Washington State to periodically prepare a list of all surface waters in the state for which beneficial uses of the water – such as for drinking, recreation, aquatic habitat, and industrial use – are impaired by pollutants. These are water quality-limited estuaries, lakes, and streams that fall short of state surface water quality standard, and are not expected to improve within the next two years.

Acronyms and Abbreviations

Ecology	Washington State Department of Ecology
EIM	Environmental Information Management database
GIS	Geographic Information System software
MDL	Method Detection Limit
MEL	Manchester Environmental Laboratory
MQO	Measurement quality objective
PCB	Polychlorinated biphenyls
QA	Quality assurance

QC	Quality control
RPD	Relative percent difference
SOP	Standard operating procedures
TMDL	(See Glossary above)
USGS	U.S. Geological Survey

Units of Measurement

ug/Kg	micrograms per kilogram (parts per billion)