

# Quality Assurance Project Plan

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## Verification of 303(d) Listings for Fish Tissue in the Skagit and Pend Oreille Rivers

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November 2004

### 303(d) Listings Addressed in this Study

Skagit River (SV53RP) – 4,4’-DDE, 4,4’-DDT, Alpha BHC, and Total PCBs  
Pend Oreille River (DS54SI) - Aldrin

User Study ID: BERA0002

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- B. Individual 303(d) (Category 5) and Category 2 Listings
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## Abstract

The Skagit and Pend Oreille Rivers are listed on the Federal Clean Water Act, Section 303(d) List for violations of water quality standards for the following parameters in fish tissue:

- Skagit River – 4,4'-DDE, 4,4'-DDT, Alpha BHC, and Total PCBs.
- Pend Oreille River – Aldrin.

By request of the Washington State Department of Ecology's Water Quality Program, these rivers will be assessed to determine whether or not they should remain on the 303(d) list. The listed parameters will be analyzed in fish tissue and the results compared to the criteria specified by Ecology's Water Quality 303(d) Listing Policy.

## Background and Problem Statement

The Washington State Department of Ecology's (Ecology) Water Quality (WQ) Program has requested that the Skagit and Pend Oreille Rivers be re-assessed for violations of water quality standards. These rivers are listed on the Federal Clean Water Act, Section 303(d) List, Category 5, for exceeding the National Toxics Rule (NTR) Human Health Criteria for several chemical contaminants in fish tissue. Both rivers also have Category 2 listings for chemical contaminants in fish tissue and in the water column.

Category 5 is the formal 303(d) list where U.S. Environmental Protection Agency (EPA) approval and Total Maximum Daily Load (TMDL) assessment are required. Category 2 is an informal category that allows for waterbodies suspected of having contamination to be tracked by Ecology ([Appendix A](#) contains more detailed descriptions of the 303(d) water quality assessment categories). All of the individual listings for the Skagit and Pend Oreille Rivers are shown in [Table 1](#) and more detailed descriptions of the listings are given in [Appendix B](#).

**Table 1. Individual 303(d) Listings Addressed by the Verification Study.**

River	Matrix	Listing Category	303(d) Listed Parameter
Skagit	fish tissue	2/ 5	4,4'-DDE
Skagit	fish tissue	5	4,4'-DDT
Skagit	fish tissue	5	Alpha-BHC
Skagit	fish tissue	2/ 5	Total PCBs
Skagit	fish tissue	2	Dieldrin
Skagit	fish tissue	2	Bis(2-ethylhexyl)phthalate
Pend Oreille	fish tissue	5	Aldrin
Pend Oreille	water	2	4,4'-DDE
Pend Oreille	water	2	4,4'-DDD
Pend Oreille	water	2	4,4'-DDT
Pend Oreille	water	2	Heptachlor Epoxide
Pend Oreille	water	2	Heptachlor
Pend Oreille	water	2	Dieldrin
Pend Oreille	water	2	Endrin

The 303(d) listings are based on older data that may not be indicative of the current river conditions. The data collected from this study will assist the WQ Program in determining the appropriateness of the listings. Ecology's Environmental Assessment (EA) Program will investigate both rivers and make recommendations on listing status to the WQ Program.

## Skagit River

The Skagit River is located in the northwestern portion of Washington State (Figure 1). It has a drainage basin of approximately 3,093 square miles. It is the largest tributary to Puget Sound and has the largest drainage basin in Washington outside the Columbia River (Pickett, 1997).

The river originates in British Columbia, flows through Ross Lake, and then by the three main population centers: Sedro Woolley, Burlington, and Mount Vernon. Just before the Skagit enters the Puget Sound at Skagit Bay, it splits into the North and South Forks which bound Fir Island. The North Fork, South Fork, and Mainstem Skagit River are subject to tidal influence extending about 15 miles upstream to Mount Vernon. At high tide, flow is stopped and at times reversed in the North and South Forks (Pickett, 1997).

On average, more than 15 million acre-feet of water cycles through the river basin annually (Butkus et al., 2000). The flows of the Skagit River and its tributaries exhibit a complex hydrology influenced by several sources. Peak flows are in the early summer. Summertime flows are maintained by groundwater inflow in the tributary drainages and are also strongly influenced by glacial outflow and snowmelt. Wintertime flows are dominated by the amount of rainfall, with peak flows that may include snowmelt (Pickett, 1997).

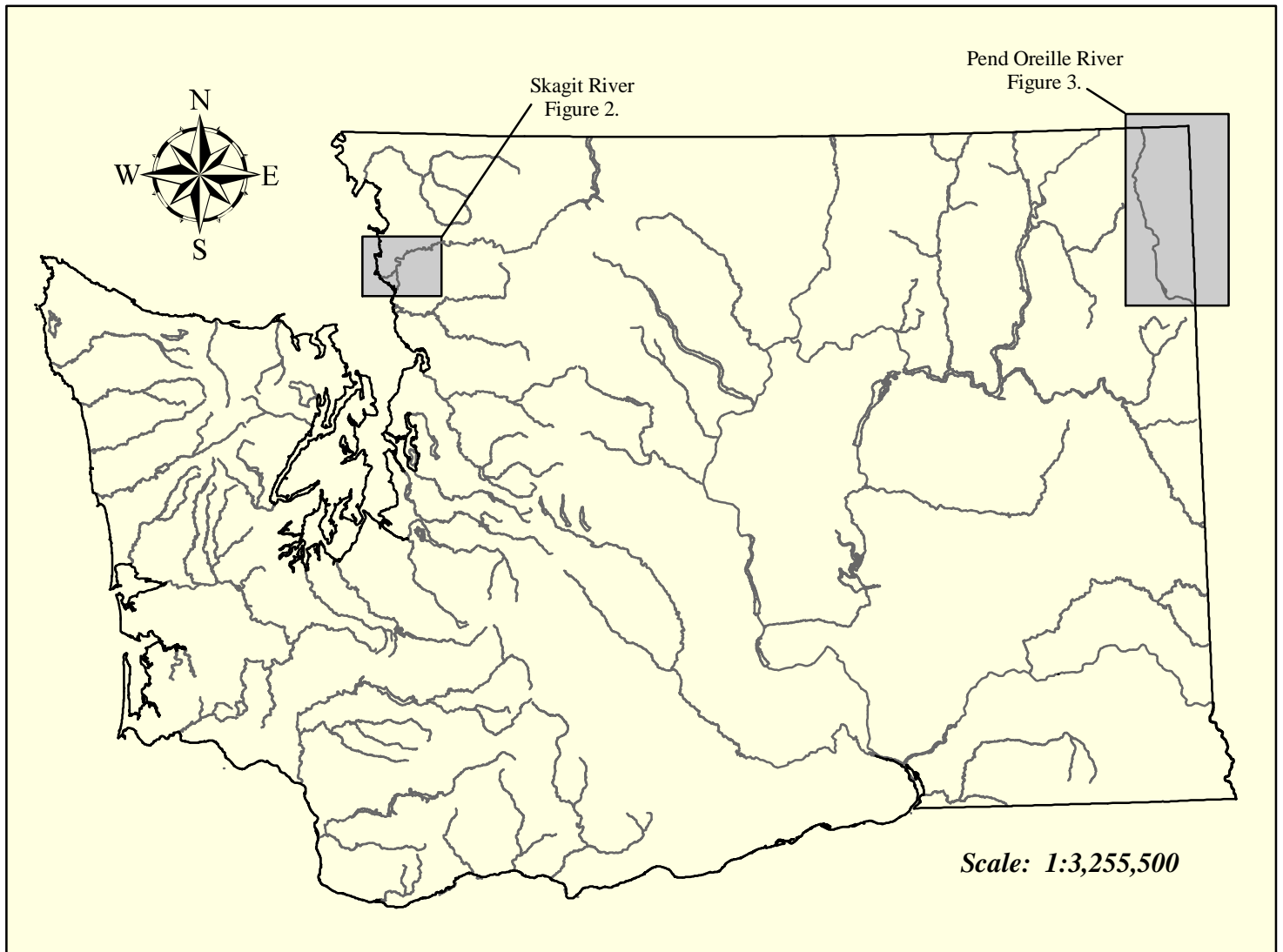


Figure 1. Map of Washington State Showing the General Location of the Skagit River and Pend Oreille River Sampling Areas

The Skagit River provides hydroelectric power, drinking water, irrigation, fish and wildlife habitat, and recreational opportunities. The main land uses in the basin are agriculture, forestry and urban. The Skagit River system is contained within Water Resource Inventory Areas (WRIAs) 3 and 4.

The Lower Skagit River has Category 5 listings for 4,4'-DDE, 4,4'-DDT, alpha-BHC, and total PCBs and Category 2 listings for 4,4'-DDE, total PCBs, dieldrin and bis (2-ethylhexyl) phthalate (DEHP) in fish tissue. [Appendix C](#) gives a brief background on all of these compounds. They are classed by EPA as probable human carcinogens. More detailed profiles for these compounds can be found at <http://www.atsdr.cdc.gov/toxpro2.html>.

[Figure 2](#) shows the location of the 303(d) listings for the Skagit River. The Category 5 listed segment is located within the city limits of Mt. Vernon. The data used as the basis for this listing came from a screening study conducted by Ecology in 1984 (Hopkins et al., 1985). Data from the 1984 study are shown in [Table 2](#). In a single composite sample of muscle tissue from bridgelip suckers (*Catostomus columbianus*) concentrations of 4,4'-DDE, 4,4'-DDT, alpha-BHC, and total PCBs exceeded NTR human health criteria. Concentrations of alpha-BHC and total PCBs from a mountain whitefish (*Prosopium williamsoni*) composite also exceeded criteria.

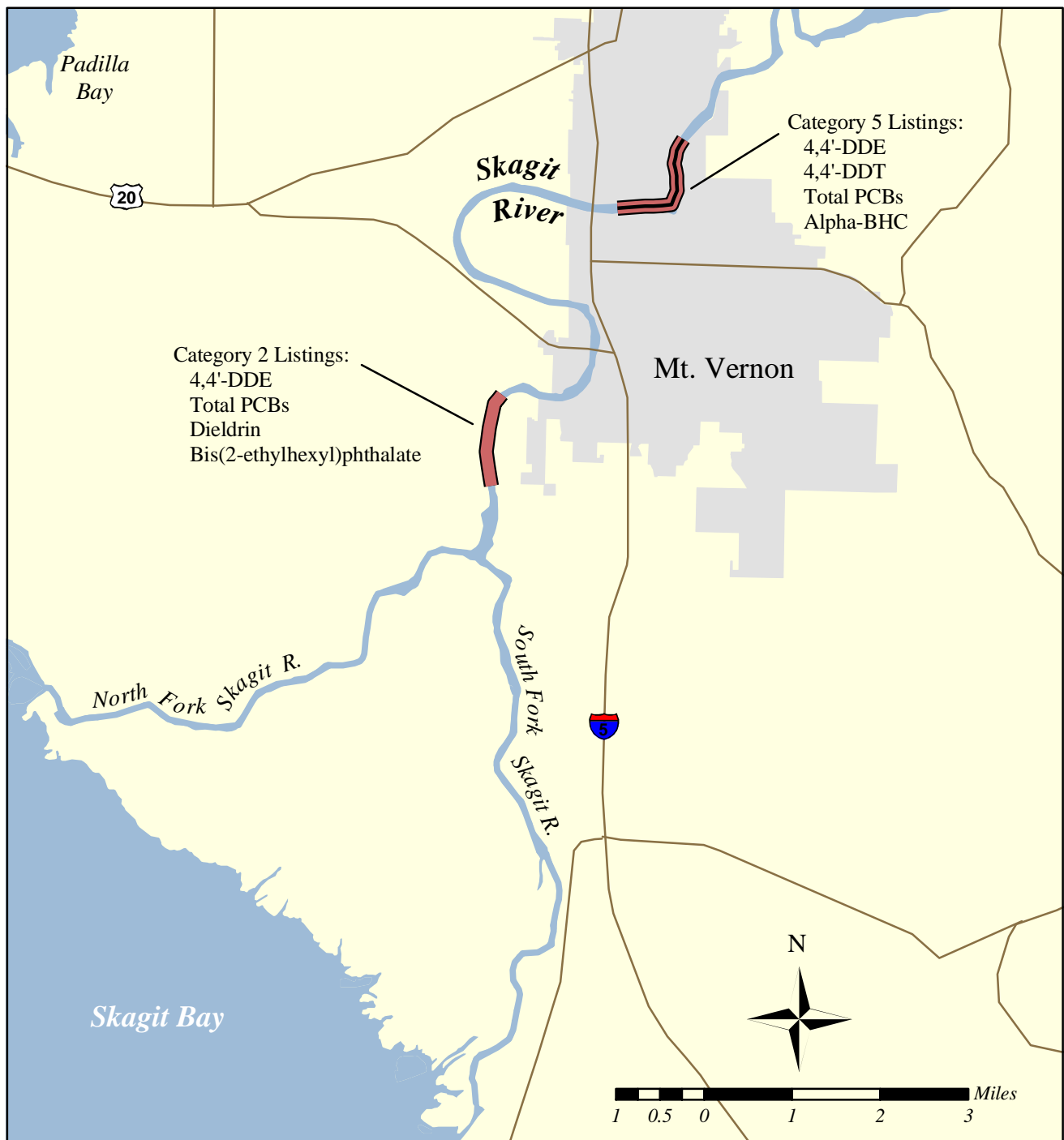


Figure 2. Lower Skagit River Showing 303(d)-Listed River Segments



**Table 2. Chlorinated Pesticide and PCB Concentrations (ug/Kg, Wet Weight) in Fish Muscle Tissue from the Skagit River – 1984 Data (Hopkins et al., 1985).**

Parameter	Bridgelip Sucker (1 Composite)	Mountain Whitefish (1 Composite)	National Toxics Rule Criteria*
4,4'-DDT	<b>47</b>	19	32
4,4'-DDE	<b>33</b>	28	32
4,4'-DDD	31	5	45
Total DDT	111	52	
Alpha-BHC	<b>4</b>	<b>4</b>	1.7
PCB-1260	36	28	
Total PCBs	<b>36</b>	<b>28</b>	5.3

\* Based on EPA bioconcentration factors and water column criteria established under the National Toxics Rule (40 CFR Part 131). Applies to edible fish tissue only.

**Bolded** values exceed NTR criteria.

The data for the Category 2 listing came from the Washington State Department of Fish and Wildlife's PSAMP database. The results were from several composites of different species of salmon collected from 1992 through 2000. The data were not used for a Category 5 listing because salmon are anadromous and, as such, the contaminants could not be traced back to the Skagit River with confidence.

## Pend Oreille River

The Pend Oreille River is located in the northeastern corner of Washington State (Figure 1). It is part of the Pend Oreille/Clark Fork Watershed which is contained in Idaho, Montana, Washington, and Canada. Less than 4% of the watershed lies within Washington State (Dames and Moore Inc., 1995)

The Pend Oreille River begins at the outlet of Lake Pend Oreille, which is fed by the Clark Fork River. The headwaters of the Clark Fork River are in the Rocky Mountains in Montana. The Pend Oreille enters Washington State at Newport, along the Idaho border, and then flows northward toward the border with Canada. Downstream of Newport, the river passes through land of the Kalispel Tribe of Indians. A short reach of the river flows through Canada to its confluence with the Columbia River just upstream of the international border.

The Pend Oreille Watershed is located in WRIA 62. The land within WRIA 62 is primarily federally managed forest (93%), with areas of rangeland (2%) and agriculture (4%) located adjacent to the river corridor (Ecology, 2003). The agriculturally based areas within the Pend Oreille Watershed are composed of a variety of uses including fruit orchards, cultivated crops, grazing, and animal husbandry. The major urban area in the watershed is the town of Newport. Land uses within the watershed have not changed significantly within the past several decades (Dames and Moore Inc., 1995).

The Pend Oreille River has a Category 5 listing for aldrin in fish tissue and Category 2 listings for dieldrin, endrin, DDT analogs, heptachlor, and heptachlor epoxide in the water column. All of these compounds are classed by EPA as probable human carcinogens. Descriptions of these compounds can be found in [Appendix C](#) and at <http://www.atsdr.cdc.gov/toxpro2.html>.

[Figure 3](#) shows the locations of the 303(d) listings in the Pend Oreille. The data used as the basis for the Category 5 aldrin listing came from a screening study conducted by Ecology in 1989 (Hopkins, 1991). A review of the 1989 data indicates that the listing for aldrin was a mistake. As shown in [Table 3](#), aldrin was not detected at or below 7.8 ug/Kg in either a composite sample of whole largescale suckers (*Catostomus macrocheilus*) or a composite of largemouth bass (*Micropterus salmoides*) muscle tissue.

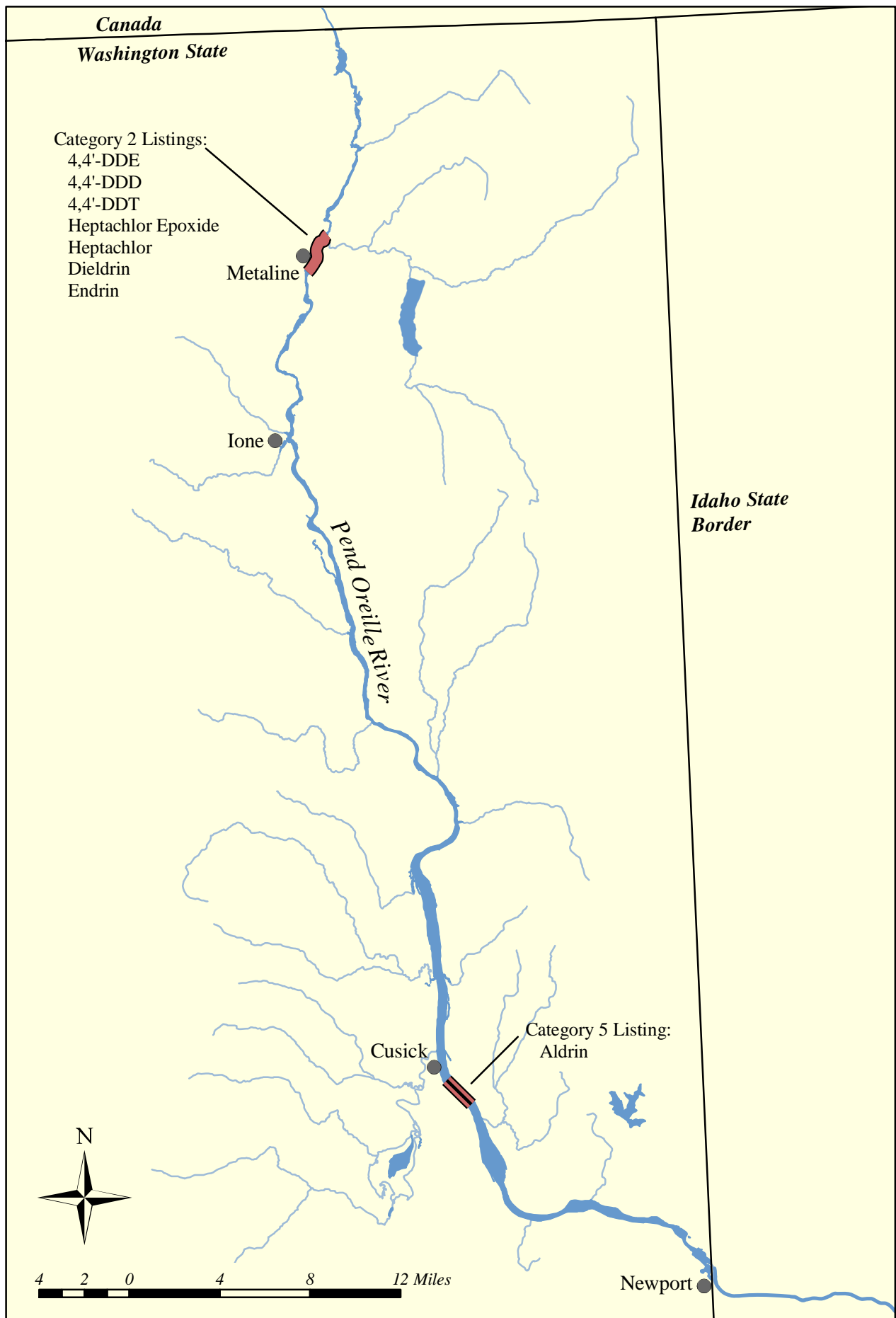


Figure 3. Pend Oreille River Showing 303(d)-Listed River Segments

**Table 3. Chlorinated Pesticide and PCB Concentrations (ug/Kg, Wet Weight) in Fish Tissue from the Pend Oreille River.**

Species Tissue No. in Comp.	1989 <sup>1</sup>		2002 <sup>2</sup>		National Toxics Rule Criteria*
	Largescale Sucker Whole	Largemouth Bass Muscle	Largescale Sucker Whole	Brown Trout Muscle	
	1	1	2	2	
4,4'-DDT	8 U	8 U	2 U	2 U	32
4,4'-DDE	5 J	8 U	8.9	2 U	32
4,4'-DDD	8 U	8 U	2 U	2 U	45
Total DDT	5 J	8 U	8.9	2 U	
Alpha-BHC	--	--	10 U	10 U	1.7
Aldrin	8 U	7.8 U	4 U	<b>3.2 J</b>	
Dieldrin	16 U	15 U	1 U	1 U	
PCB-1260	150 U	150 U	--	--	
Total PCBs	150 U	150 U	84 J	4.5 J	5.3

\* Based on EPA bioconcentration factors and water column criteria established under the National Toxics Rule (40 CFR Part 131). Applies to edible fish tissue only.

-- Data not analyzed for.

<sup>1</sup> = Hopkins, 1991.

<sup>2</sup> = EPA, 2004 (unpublished data).

J = Estimated value.

U = Not detected at or above reported result.

**Bolded** values exceed NTR criteria.

Interestingly, some recent and unpublished data from EPA's National Study of Chemical Residues in Lake Fish Tissue (EPA, 2004) found that aldrin in a composite sample of brown trout (*Salmo trutta*) muscle tissue from the Pend Oreille River exceeded NTR human health criteria (Table 3). This new data would justify a Category 5 listing for aldrin in the Pend Oreille River. The mean total PCB concentration in largescale sucker composites analyzed by EPA was elevated at 84 ug/Kg, but does meet listing criteria under the NTR because the data came from whole fish and not muscle (edible) tissue.

Both the 1989 Ecology and 2002 EPA fish samples were taken from the same river segment, upstream of the town of Cusick (Figure 3). The Category 2 water column data came from the northern portion of the Pend Oreille, near Metaline. The water column data came from EPA's STORET database, was collected between 1969 and 1971, and is of questionable accuracy.

## Project Description

Fish tissue composites will be collected from the Category 2 and Category 5 303(d) listed segments of the Skagit and Pend Oreille Rivers. Tissue will be analyzed for the Category 2 and Category 5 contaminants to evaluate current status of the 303(d) listings. Category 2 water column contaminants for the Pend Oreille River will be addressed through the fish tissue analysis. The concentrations of these contaminants are likely to be very low and difficult to detect in water. They are known to bioaccumulate in fish tissue; and, if present in the water column, will have a better chance of being detected through the analysis of fish tissue.

Fish tissue samples will be analyzed by Ecology's Manchester Environmental Laboratory (MEL), by Gas Chromatography Mass Spectrometry (GC/MS) for DEHP, and by Gas Chromatography Electron Capture Detection (GC/ECD) for the rest of the parameters.

Detection limits for the GC/ECD analysis will be sufficient to meet the 303(d) listing criteria for most of the contaminants of interest. However, several will require very low detection limits in order to meet the 303(d) listing criteria. As a contingency, if MEL can't meet the desired detection limits for these several contaminants through GC/ECD, then a subset of samples will be contracted to AXYS laboratory. AXYS will analyze these samples through High Resolution Gas Chromatography Mass Spectrometry (HR GC/MS) in order to achieve very low detection limits.

## Responsibilities

*Sally Lawrence* (Ecology) – Client and staff contact for the Northwest Regional Office. Responsible for reviewing the Quality Assurance Project Plan and draft study report.

*Paul Turner* (Ecology) – Client and staff contact for the Eastern Regional Office. Responsible for reviewing the QA Project Plan and draft study report.

*Brandee Era-Miller* (Ecology) – Toxics Studies Unit Project Manager. Responsible for study design and preparation of the QA Project Plan, field sampling, interpretation of results, and authoring the study report.

*Kristin Kinney* (Ecology) – Assistance with preparation of the QA Project Plan, field sampling, sample processing, and entering the project data into the EIM database.

*Casey Deligeannis* (Ecology) – Assistance with field sampling and sample processing.

*Kalispel Tribe of Indians* – Assistance with field sampling for the Pend Oreille River.

*Dale Norton* (Ecology) – Toxics Studies Unit Supervisor. Responsible for review of the QA Project Plan and draft study report.

*Will Kendra* (Ecology) – Watershed Ecology Section Manager. Responsible for review of the QA Project Plan and draft study report.

*Cliff Kirchmer* (Ecology) – Quality Assurance Officer. Responsible for review of the QA Project Plan and assistance on quality assurance issues during the implementation of the study.

*Stuart Magoon and MEL Personnel* (Ecology) – Responsible for review of the QA Project Plan pertaining to laboratory analysis and the analysis and reporting of project data to the project manager. Responsible for setting up the contract with AXYS Laboratory.

*AXYS Laboratory (Contract Laboratory)* – Responsible for conducting the laboratory analysis specified in the QA Project Plan and reporting the data to MEL and the project manager. AXYS will be given a copy of the QA Project Plan.

## Schedule and Budget

**Approval of QA Project Plan**                      October 2004

### Field Work

- Fish Collection                                      October 2004
- Fish Tissue Processing                              November 2004
- Samples to Laboratories                              November 2004

### Data Reporting

- Laboratory Data Completed                      February 2005
- Draft Report for Client Review                      April 2005
- EIM Data Entry                                      May 2005
- Final Report    May 2005

### Laboratory Budget

	<b>FY05</b>
Fish Tissue Analysis (MEL)	\$ 7,165
<u>Fish Tissue Analysis (AXYS)</u>	<u>\$ 6,675</u>
Total Cost*	<b>\$ 13,840</b>

\*If MEL is unable to meet desired detection limits with GC/ECD analysis, a set of subsamples may be sent to AXYS for HR GC/MS analysis at additional cost.

## Decision Criteria

In order to make recommendations on whether waterbodies should be removed or retained on the 303(d) list, data must meet the listing criteria of Ecology's Water Quality 303(d) Listing Policy (Ecology, 2002). Listing recommendations for this study will be based on the following:

The listing criteria for contaminants in fish includes fin fish muscle tissue from at least three single-fish samples or a single composite sample made up of at least five separate fish of the same species. If the average of the three single-fish samples with the highest contaminant concentration or the contaminant concentration of composite fish sample exceeds criteria for human health impacts based on EPA's bio-concentration factors and water column criteria established under the NTR, then the waterbody should be listed (Ecology, 2002).

## Data Quality Objectives

In order to limit potential sources of bias prior to laboratory analysis, fish tissue processing will follow EPA guidance (PSEP, 1996; EPA, 2000). These protocols are explained in further detail in the Field Quality Control section and the Field Procedures and Sample Preparation section of this QA Project Plan.

The laboratories conducting the analyses are expected to meet all quality control (QC) requirements of the analytical methods selected for this study. The analytical Measurement Quality Objectives (MQOs) that will be used are shown in [Table 4](#).

**Table 4. Analytical Measurement Quality Objectives.**

Parameter	Laboratory Control Samples	Duplicate Samples	Matrix Spikes	Matrix Spike Duplicates	Surrogate Standards
	% Recovery Limits	RPD	% Recovery Limits	RPD	% Recovery Limits
GC/ECD Chlorinated Pesticides <sup>1</sup> & PCB aroclors	50-150	≤ 50	50-150	≤ 50	10-140
HR GC/MS Chlorinated Pesticides <sup>2</sup>	30-150	≤ 50	60-130	≤ 50	30-150
DEHP	50-150	≤ 50	50-150	≤ 50	50-150
Percent Lipids	n/a	≤ 20	n/a	n/a	n/a

RPD = Relative Percent Difference.

<sup>1</sup> = GC/ECD parameters: 4,4'-DDT, 4,4'-DDE, 4,4'-DDD, alpha-BHC, aldrin, dieldrin, endrin, heptachlor and heptachlor epoxide.

<sup>2</sup> = HR GC/MS parameters: alpha-BHC, aldrin, dieldrin, heptachlor and heptachlor epoxide.

The percent recoveries of the laboratory control samples (LCS), matrix spikes, and surrogate standards are used as a measure of accuracy. Matrix spikes and surrogate standards also give an indication of any bias that may result from the laboratory analysis. Surrogate standards are added to every sample prior to extraction, while matrix spikes are added to only one sample within a sample batch. Matrix spike recoveries are more reliable than surrogate standard recoveries when it comes to chlorinated pesticide analysis in fish tissue and, therefore, greater weight will be given to matrix spike recoveries over surrogate standard recoveries as measures of analytical accuracy and bias (MEL, 2004). The Relative Percent Difference (RPD) of laboratory duplicates and matrix spike duplicates is used as a measure of precision.

The MQOs for contaminant lowest concentrations of interest in fish tissue are shown in [Table 5](#). These lowest concentrations of concern are the NTR human health criteria for contaminants in fish tissue. The laboratories must report down to these concentrations in order to meet the objectives of the study.



**Table 5. Lowest Concentrations of Interest for Fish Tissue Contaminant Levels.**

<b>Parameter</b>	<b>Lowest Conc. of Interest ug/Kg Wet Weight (ppb)</b>
4,4'-DDT	32
4,4'-DDE	32
4,4'-DDD	45
Aldrin	0.65
Dieldrin	0.65
Endrin	3216
Alpha-BHC	1.7
Heptachlor	2.4
Heptachlor Epoxide	1.2
Total PCB Aroclors	5.3
DEHP	767

## **Sampling Design**

All fish tissue samples for the study will be composites of five individual fish. Fish of at least legal size will be selected. To the extent possible, the length of the smallest fish within a composite will be no less than 75% of the length of the largest fish, as recommended by EPA (2000).

Two types of composites will be collected from each sampling section: predator species and bottom-feeding species. This sampling method will ensure that contaminant concentrations within the food chain are represented.

Table 6 shows the number of composites to be analyzed for the study.

**Table 6. Number of Composite Fish Tissue Samples.**

	<b>Skagit River</b>	<b>Pend Oreille River</b>
No. of Composites (Predator)	2	4
No. of Composites (Bottom feeder)	2	4
No. of Sampling Sections	1	2
Total No. of Composites	4	8

## Skagit River

Four composite samples, consisting of muscle fillet tissue from the lower section of the Skagit River (near the Category 5 listed segment) will be analyzed. Two of the samples will be from a predator species and two from a bottom-feeding species.

Target species will be mountain whitefish (*Prosopium williamsoni*) and bridgelip suckers (*Catostomus columbianus*). Mountain whitefish are considered to be a predator and bridgelip suckers a bottom-feeder. These species will also be targeted because they are resident to the Skagit River and, therefore, will be more representative of the ambient conditions. Salmonids and other anadromous species will be avoided.

Fish collection methods in the Skagit River will include either hook and line or boat electrofishing. Angling is preferred over boat electrofishing because it is much less invasive. If angling is unsuccessful, then electrofishing will be used.

## Pend Oreille River

Four muscle tissue composite samples will be analyzed each from both of the 303(d) listed segments of the Pend Oreille River. The Category 2 listed segment is located at the northern/downstream extent of the Washington portion of the river near the town of Metaline. The Category 5 listed segment is located at the southern/upstream extent of the Washington portion of the river near the town of Cusick (Figure 3).

Two predator and two bottom-feeding species will be targeted at each segment. Brown trout, mountain whitefish, and northern pike minnow (*Ptychocheilus oregonensis*) are examples of predator species found in the Pend Oreille River. Suckers will be targeted as the bottom-feeder. Species chosen for analysis will depend upon availability during sampling.

The Kalispel Tribe will collect the fish samples from the Pend Oreille River. They will use various methods for fish collection including fyke nets, gill nets, and boat electrofishing. If they are unable to collect all of the required fish samples, Ecology will use boat electrofishing to finish the sample collection.

## **Field Procedures and Sample Preparation**

All necessary state and federal permits have been obtained for fish collection. Fishing locations will be recorded by GPS. Fish selected for analysis will be quickly killed with a sharp blow to the head, given an ID number, and the weight and length recorded. The fish will be individually wrapped in heavy aluminum foil and put in plastic bags, kept cold in coolers, and frozen immediately upon return from the field.

Preparation of fish tissue samples will follow EPA (2000) guidance and will take place at Ecology's Headquarters building in Lacey, Washington. Precautions will be taken to minimize contamination during sample processing. Persons preparing samples will wear non-talc nitrile gloves and aprons. Work surfaces will be covered with heavy grade aluminum foil. Gloves, aluminum foil, and dissection tools will be changed between composite samples.

Samples for analysis will be prepared by partially thawing the fish to remove the foil wrapper and rinsing in deionized water to remove adhering debris. The scales will be removed and the entire skin-on muscle fillet from either one or both sides of each fish will be taken with stainless steel knives and homogenized by several passes through a Kitchen-Aid food processor.

To avoid contamination with tissue samples for DEHP analysis, plastics will be avoided. For these samples, a stainless steel Hobart commercial blender will be used instead of the kitchen-Aid food processor.

Composite samples will consist of equal-weight aliquots from each of the five fish. The samples will be homogenized to uniform color and consistency and placed in jars, specifically-cleaned for low-level organic analyses, and sent to the laboratories. Excess sample will be retained from each composite and stored frozen in the event that additional analysis is required by the laboratories. Containers and holding times for the fish tissue samples are shown in [Table 7](#).

**Table 7. Containers and Holding Times for Fish Tissue Samples.<sup>1</sup>**

Parameter	Container	Preservation	Holding Time*
GC/ECD Chlorinated Pesticides & PCB Aroclors	Certified 4-oz glass jar w/ Teflon lid liner	Refrigerate, 4° C Freeze, -18° C	7 day extraction 14 day analysis
HR GC/MS Chlorinated Pesticides	Certified 8-oz amber glass jar w/ Teflon lid liner	Refrigerate, 4° C Freeze, -18° C	7 day extraction 14 day analysis
DEHP	Certified 4-oz glass jar w/ Teflon lid liner	Refrigerate, 4° C Freeze, -18° C	7 day extraction 14 day analysis
Percent Lipids	Taken from the pesticide jars	Refrigerate, 4° C Freeze, -18° C	7 day extraction 14 day analysis

\* Frozen tissue samples can be held for up to 1 year.

<sup>1</sup> MEL, 2003 and PSEP, 1996.

All resecting instruments will be washed thoroughly with Liquinox detergent, followed by sequential rinses of hot tap water, de-ionized water, pesticide-grade acetone, and pesticide-grade hexane. The same decontamination procedure will be repeated between each composite sample.

The sex of each fish will be recorded during processing and aging structures (scales, otoliths, opercles, and/or dorsal spines as appropriate for each species) will be saved for age determination by the Washington Department of Fish and Wildlife (WDFW) in Olympia, Washington.

## Laboratory Procedures

Target parameters, anticipated reporting limits, and analytical methods for the 303(d) Fish Tissue Verification Study are shown in [Table 8](#). The lowest concentrations of interest for fish tissue were included in the table to give a clear comparison to the anticipated reporting limits.

**Table 8. Reporting Limits and Analytical Methods.**

Parameter	Anticipated Reporting Limits (ug/Kg ww)	Lowest Conc. of Interest (ug/Kg ww)	Sample Preparation Method	Analytical Method	Laboratory
<i>GC/ECD analysis</i>					
4,4'-DDT	0.60	32	EPA 3540/3620	EPA 8081	MEL
4,4'-DDE	0.60	32	EPA 3540/3620	EPA 8081	MEL
4,4'-DDD	0.60	45	EPA 3540/3620	EPA 8081	MEL
Aldrin	0.60	0.65	EPA 3540/3620	EPA 8081	MEL
Dieldrin	0.60	0.65	EPA 3540/3620	EPA 8081	MEL
Endrin	0.60	3216	EPA 3540/3620	EPA 8081	MEL
Alpha-BHC	0.60	1.7	EPA 3540/3620	EPA 8081	MEL
Heptachlor	0.60	2.4	EPA 3540/3620	EPA 8081	MEL
Heptachlor Epoxide	0.60	1.2	EPA 3540/3620	EPA 8081	MEL
Total PCB Aroclors	5.0	5.3	EPA 3540/3620/3665	EPA 8082	MEL
<i>HR GC/MS analysis</i>					
Aldrin	0.02	0.65	SOP MLA-028		AXYS
Dieldrin	0.02	0.65	SOP MLA-028		AXYS
Alpha-BHC	0.02	1.7	SOP MLA-028		AXYS
Heptachlor	0.02	2.4	SOP MLA-028		AXYS
Heptachlor Epoxide	0.02	1.2	SOP MLA-028		AXYS
DEHP	500	767	EPA 3540/3620	EPA 1625 & 8270	MEL
Lipids	0.1 %	--	Extraction	EPA 608.5	MEL

The anticipated reporting limits are what the laboratories expect to achieve with the methods selected for the study. The anticipated laboratory reporting limits for GC/ECD are very close to the lowest concentrations of interest for several of the compounds (aldrin, dieldrin, alpha-BHC, heptachlor, and heptachlor epoxide). To ensure that the lowest concentrations of interest are achieved for the study, a subset of samples may be sent to AXYS for analysis. AXYS will use HR GC/MS methods to achieve lower reporting limits.

Total laboratory costs and number of samples to be analyzed for the study are shown in [Table 9](#).

**Table 9. Estimated Laboratory Costs for the 303(d) Fish Tissue Verification Study.**

Analysis	No. of Samples	No. of QC Samples <sup>1</sup>	Total No. of Samples	Cost per Analysis	Cost Subtotals
Percent Lipids	12	3	15	31	\$ 465
GC/ECD Chlorinated Pesticides & PCB Aroclors	12	4	16	325	\$ 5,200
DEHP	4	2	6	250	\$ 1,500
HR GC/MS Chlorinated Pesticides	4	2	6	890	\$ 6,675
25% MEL surcharge for contract analysis					\$ 1,335
<b>Fish Tissue Analysis Total</b>					<b>\$ 13,840 *</b>

<sup>1</sup> Includes laboratory duplicates, standard reference material, and matrix spikes.

\* Costs include 50% discount for samples analyzed by MEL.

## Quality Control Procedures

### Field

Field work will be conducted in order to avoid cross-contamination. Field sampling procedures described in the Field Procedures and Sample Preparation section of this QA Project Plan will be carefully followed. A copy of the QA Project Plan will be taken into the field for reference.

Natural variability in contaminant concentrations between individual fish will be accounted for by analyzing fish in composite and by analyzing a field replicate at each site (composite sample of the same species).

## Laboratory

As part of their Standard Operating Procedures (SOPs), MEL routinely analyzes quality control samples for all analytical methods. These SOPs are described in MEL’s Quality Assurance Manual (MEL, 2002). Additional quality control samples are sometimes requested by the project manager. Quality control samples selected for this study are shown in [Table 10](#).

Laboratory duplicates will provide estimates of variability in the data (processing + laboratory). Laboratory duplicates will be composite sample splits. One laboratory duplicate each will be analyzed for the Skagit and Pend Oreille Rivers’ fish.

A standard reference material (SRM) will be analyzed for determining accuracy of the DDT, dieldrin, and heptachlor epoxide results. MEL will analyze the National Institute of Standards & Technology (NIST) SRM 1946 – Lake Superior Fish Tissue.

**Table 10. Laboratory Quality Control Samples for the 303(d) Fish Tissue Verification Study.**

Analysis	Method Blank	Lab Duplicate	Lab Control Sample	Surrogate Spikes	SRM	Matrix Spike	Matrix Spike Duplicate
GC/ ECD Chlorinated Pesticides <sup>1</sup>	1	2	1	all samples	1	1	1
PCB Aroclors	1	2	1	all samples	--	1	1
DEHP	1	1	1	all samples	--	1	1
Percent Lipids	1	2	1	--	--	1	1
HR GC/MS Chlorinated Pesticides <sup>2</sup>	1	1	1	all samples	--	1	1

<sup>1</sup> Analytes include: 4,4'-DDT, 4,4'-DDE, 4,4'-DDD, alpha-BHC, aldrin, dieldrin, endrin, heptachlor, and heptachlor epoxide.

<sup>2</sup> Analytes include: alpha-BHC, aldrin, dieldrin, heptachlor, and heptachlor epoxide.

## Data Review and Validation

The Manchester Environmental Laboratory will verify laboratory results and prepare a data verification report, including a case summary for their data. MEL will validate laboratory results from the contract laboratory (AXYS), including any changes to data qualifiers. Upon receipt of the laboratory data packages from both MEL and AXYS, the project manager will validate the results by determining if the measurement quality objectives were met for the study.

The project manager will provide a draft report of the study results to the clients in **April 2005**. At a minimum the final report will contain the following:

- A map of the study areas that shows sampling sites.
- Latitude/longitude and other location information for each sampling site.
- Descriptions of field and laboratory methods.
- A discussion of data quality and the significance of any problems encountered during sampling and analysis.
- Summary tables of biological and chemical data.
- A summary of significant findings.
- Recommendations for continued listing or de-listing on the 303(d) list and potential follow-up work.

A final report will be prepared after receiving review comments from the clients (Water Quality Program – NWRO and ERO) and internal comments from the EA Program. The final report is slated for completion in **May 2005**. All the chemistry data generated from the study will be entered into Ecology's Environmental Information Management (EIM) system.

## Data Quality Assessment

Once the data have been reviewed, verified, and validated, the project manager will determine if the quality and quantity of the data are usable and whether the data can be used to make decisions for which the study was designed.



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# Appendices

# Appendix A

## Appendix A

### 303(d) List Water Quality Assessment Categories (Ecology, 2002 Draft)

Category 1. Meets Tested Standards	Not impaired, or not known to be impaired	EPA approved and TMDL not required
Category 2. Water of Concern		
Category 3. No Data		
Category 4. Impaired But Does Not Require a TMDL 4a. Has a TMDL 4b. Has a Pollution Control Plan 4c. Impaired by a Non-Pollutant	Impaired	EPA approved and TMDL required
Category 5. The 303(d) List		

## Appendix B

# Water Quality Assessment for Washington

2002/2004 Candidate List

Water Body Name: SKAGIT RIVER

Parameter: 4,4'-DDE

Medium: Tissue

Category: 5

Listed 98?: N

Listed96?: N

Listing ID #: 14034

Township: 34N

Range: 04E

Section: 08

Latitude:

Longitude:

## Remarks

## Basis

Hopkins et al. 1985. show an excursion beyond the National Toxic Rule criterion in a multiple fish composite of edible tissue of Bridgelip sucker samples collected in 1984.

# Water Quality Assessment for Washington

2002/2004 Candidate List

Water Body Name: SKAGIT RIVER

Parameter: 4,4'-DDT

Medium: Tissue

Category: 5

Listed 98?: N

Listed96?: N

Listing ID #: 14032

Township: 34N

Range: 04E

Section: 08

Latitude:

Longitude:

## Remarks

## Basis

Hopkins et al. 1985. show an excursion beyond the National Toxic Rule criterion in a multiple fish composite of edible tissue of Bridgelip sucker samples collected in 1984.



# Water Quality Assessment for Washington

2002/2004 Candidate List

Water Body Name: SKAGIT RIVER

Parameter: ALPHA-BHC

Medium: Tissue

Category: 5

Listed 98?: N

Listed96?: N

Listing ID #: 14035

Township: 34N

Range: 04E

Section: 08

Latitude:

Longitude:

## Remarks

### Basis

Hopkins et al. 1985. show excursions beyond the National Toxic Rule criterion in a multiple fish composite of edible tissue of Bridgelip sucker and Mountian whitefish samples collected in 1984.

# Water Quality Assessment for Washington

2002/2004 Candidate List

Water Body Name: SKAGIT RIVER

Parameter: Total PCBs

Medium: Tissue

Category: 5

Listed 98?: N

Listed96?: N

Listing ID #: 14036

Township: 34N

Range: 04E

Section: 08

Latitude:

Longitude:

## Remarks

### Basis

Hopkins et al. 1985. show excursions beyond the National Toxic Rule criterion in a multiple fish composite of edible tissue of Bridgelip sucker and Mountian whitefish samples collected in 1984.

# Water Quality Assessment for Washington

## 2002/2004 Candidate List

Water Body Name: SKAGIT RIVER	Listing ID #: 35541
Parameter: 4,4'-DDE	Township: 34N
Medium: Tissue	Range: 03E
Category: 2	Section: 25
Listed 98?: N	Latitude:
Listed96?: N	Longitude:

### Remarks

Tissue samples are from anadromous or nonresident fish and do not include information on the likely source of the toxic pollutant as it relates to the waterbody segment. Since no evidence is available to connect the pollutant to the segment, it has been placed in the Waters of Concern Category.

### Basis

Washington Department of Fish and Wildlife PSAMP database show the National Toxic Rule Criterion was exceeded in a composite of more than 5 muscle samples collected in 1992 from chinook salmon (*Oncorhynchus tshawytscha*) samples from station SKAGIT. Washington Department of Fish and Wildlife PSAMP database show the National Toxic Rule Criterion was met in 1992-2000 from coho salmon (*Oncorhynchus kisutch*) samples from station SKAGIT.

# Water Quality Assessment for Washington

## 2002/2004 Candidate List

Water Body Name: SKAGIT RIVER	Listing ID #: 35548
Parameter: Bis(2-ethylhexyl)phthalate	Township: 34N
Medium: Tissue	Range: 03E
Category: 2	Section: 25
Listed 98?: N	Latitude:
Listed96?: N	Longitude:

### **Remarks**

Tissue samples are from anadromous or nonresident fish and do not include information on the likely source of the toxic pollutant as it relates to the waterbody segment. Since no evidence is available to connect the pollutant to the segment, it has been placed in the Waters of Concern Category.

### **Basis**

Washington Department of Fish and Wildlife PSAMP database show the National Toxic Rule Criterion was exceeded in a composite of more than 5 muscle samples collected in 1994 from coho salmon (*Oncorhynchus kisutch*) samples from station SKAGIT. Washington Department of Fish and Wildlife PSAMP database show the National Toxic Rule Criterion was met in 1992-1994 from chinook salmon (*Oncorhynchus tshawytscha*) samples from station SKAGIT.

# Water Quality Assessment for Washington

## 2002/2004 Candidate List

Water Body Name: SKAGIT RIVER

Parameter: Dieldrin

Medium: Tissue

Category: 2

Listed 98?: N

Listed96?: N

Listing ID #: 35550

Township: 34N

Range: 03E

Section: 25

Latitude:

Longitude:

### Remarks

Tissue samples are from anadromous or nonresident fish and do not include information on the likely source of the toxic pollutant as it relates to the waterbody segment. Since no evidence is available to connect the pollutant to the segment, it has been placed in the Waters of Concern Category.

### Basis

Washington Department of Fish and Wildlife PSAMP database show the National Toxic Rule Criterion was exceeded in a composite of more than 5 muscle samples collected in 1992 from chinook salmon (*Oncorhynchus tshawytscha*) samples from station SKAGIT. Washington Department of Fish and Wildlife PSAMP database show the National Toxic Rule Criterion was met in 1992-1994 from coho salmon (*Oncorhynchus kisutch*) samples from station SKAGIT.

# Water Quality Assessment for Washington

## 2002/2004 Candidate List

Water Body Name: SKAGIT RIVER	Listing ID #: 35570
Parameter: Total PCBs	Township: 34N
Medium: Tissue	Range: 03E
Category: 2	Section: 25
Listed 98?: N	Latitude:
Listed96?: N	Longitude:

### Remarks

Tissue samples are from anadromous or nonresident fish and do not include information on the likely source of the toxic pollutant as it relates to the waterbody segment. Since no evidence is available to connect the pollutant to the segment, it has been placed in the Waters of Concern Category.

### Basis

Washington Department of Fish and Wildlife PSAMP database show the National Toxic Rule Criterion was exceeded in a composite of more than 5 muscle samples collected in 1992-1995 from chinook salmon (*Oncorhynchus tshawytscha*) samples from station SKAGIT. Washington Department of Fish and Wildlife PSAMP database show the National Toxic Rule Criterion was exceeded in a composite of more than 5 muscle samples collected in 1992-1996 from coho salmon (*Oncorhynchus kisutch*) samples from station SKAGIT. Washington Department of Fish and Wildlife PSAMP database show the National Toxic Rule Criterion was exceeded in a composite of 4 muscle samples collected in 2000 from coho salmon (*Oncorhynchus kisutch*) samples from station SKAGIT.

# Water Quality Assessment for Washington

2002/2004 Candidate List

Water Body Name: PEND OREILLE RIVER

Parameter: Aldrin

Medium: Tissue

Category: 5

Listed 98?: N

Listed96?: N

Listing ID #: 9080

Township: 33N

Range: 44E

Section: 32

Latitude:

Longitude:

## Remarks

### Basis

Hopkins, 1991. , estimated tissue concentration exceeds the national toxic rule criterion at Usk during 1989.

# Water Quality Assessment for Washington

## 2002/2004 Candidate List

Water Body Name: PEND OREILLE RIVER	Listing ID #: 9079
Parameter: 4,4'-DDD	Township: 39N
Medium: Water	Range: 43E
Category: 2	Section: 28
Listed 98?: N	Latitude:
Listed96?: Y	Longitude:

### **Remarks**

The EPA data downloaded from STORET were challenged as not meeting the quality assurance criteria of the Water Quality Program policy on listing. The listed STORET contact (Ray Peterson) was asked to verify that these criteria were met for the data used as a basis for listing. EPA did not verify that these data meet the quality assurance criteria. Therefore, these data from STORET should not be used as a basis for listing (from 1998 list database).

### **Basis**

2 excursions beyond National Toxics Rule (40 CFR Part 131) criterion at USEPA station 543012 (at Metaline Falls) in 1971.



# Water Quality Assessment for Washington

## 2002/2004 Candidate List

Water Body Name: PEND OREILLE RIVER	Listing ID #: 9078
Parameter: 4,4'-DDE	Township: 39N
Medium: Water	Range: 43E
Category: 2	Section: 28
Listed 98?: N	Latitude:
Listed96?: Y	Longitude:

### **Remarks**

The EPA data downloaded from STORET were challenged as not meeting the quality assurance criteria of the Water Quality Program policy on listing. The listed STORET contact (Ray Peterson) was asked to verify that these criteria were met for the data used as a basis for listing. EPA did not verify that these data meet the quality assurance criteria. Therefore, these data from STORET should not be used as a basis for listing (from 1998 list database).

### **Basis**

4 excursions beyond National Toxics Rule (40 CFR Part 131) criterion at USEPA station 543012 (at Metaline Falls) in between 1969 and 1971.

# Water Quality Assessment for Washington

2002/2004 Candidate List

Water Body Name: PEND OREILLE RIVER

Parameter: DDT

Medium: Water

Category: 2

Listed 98?: N

Listed96?: Y

Listing ID #: 9077

Township: 39N

Range: 43E

Section: 28

Latitude:

Longitude:

## Remarks

The EPA data downloaded from STORET were challenged as not meeting the quality assurance criteria of the Water Quality Program policy on listing. The listed STORET contact (Ray Peterson) was asked to verify that these criteria were met for the data used as a basis for listing. EPA did not verify that these data meet the quality assurance criteria. Therefore, these data from STORET should not be used as a basis for listing (from 1998 list database).

## Basis

3 excursions beyond National Toxics Rule (40 CFR Part 131) criterion at USEPA station 543012 (at Metaline Falls) in between 1969 and 1971.

# Water Quality Assessment for Washington

## 2002/2004 Candidate List

Water Body Name: PEND OREILLE RIVER	Listing ID #: 9073
Parameter: Aldrin	Township: 39N
Medium: Water	Range: 43E
Category: 2	Section: 28
Listed 98?: N	Latitude:
Listed96?: Y	Longitude:

### **Remarks**

The EPA data downloaded from STORET were challenged as not meeting the quality assurance criteria of the Water Quality Program policy on listing. The listed STORET contact (Ray Peterson) was asked to verify that these criteria were met for the data used as a basis for listing. EPA did not verify that these data meet the quality assurance criteria. Therefore, these data from STORET should not be used as a basis for listing (from 1998 list database).

### **Basis**

2 excursions beyond National Toxics Rule (40 CFR Part 131) criterion at USEPA station 543012 (at Metaline Falls) in 1971.

# Water Quality Assessment for Washington

## 2002/2004 Candidate List

Water Body Name: PEND OREILLE RIVER

Parameter: Dieldrin

Medium: Water

Category: 2

Listed 98?: N

Listed96?: Y

Listing ID #: 9074

Township: 39N

Range: 43E

Section: 28

Latitude:

Longitude:

### **Remarks**

The EPA data downloaded from STORET were challenged as not meeting the quality assurance criteria of the Water Quality Program policy on listing. The listed STORET contact (Ray Peterson) was asked to verify that these criteria were met for the data used as a basis for listing. EPA did not verify that these data meet the quality assurance criteria. Therefore, these data from STORET should not be used as a basis for listing (from 1998 list database).

### **Basis**

4 excursions beyond National Toxics Rule (40 CFR Part 131) criterion at USEPA station 543012 (at Metaline Falls) in between 1969 and 1971.

# Water Quality Assessment for Washington

2002/2004 Candidate List

Water Body Name: PEND OREILLE RIVER

Parameter: Endrin

Medium: Water

Category: 2

Listed 98?: N

Listed96?: N

Listing ID #: 9072

Township: 39N

Range: 43E

Section: 28

Latitude:

Longitude:

## Remarks

## Basis

1 excursion beyond the chronic criterion at USEPA station 543012 (at Metaline Falls) on 2/11/70.

# Water Quality Assessment for Washington

## 2002/2004 Candidate List

Water Body Name: PEND OREILLE RIVER	Listing ID #: 9075
Parameter: Heptachlor	Township: 39N
Medium: Water	Range: 43E
Category: 2	Section: 28
Listed 98?: N	Latitude:
Listed96?: Y	Longitude:

### **Remarks**

The EPA data downloaded from STORET were challenged as not meeting the quality assurance criteria of the Water Quality Program policy on listing. The listed STORET contact (Ray Peterson) was asked to verify that these criteria were met for the data used as a basis for listing. EPA did not verify that these data meet the quality assurance criteria. Therefore, these data from STORET should not be used as a basis for listing (from 1998 list database).

### **Basis**

3 excursions beyond National Toxics Rule (40 CFR Part 131) criterion at USEPA station 543012 (at Metaline Falls) in between 1970 and 1971.

# Water Quality Assessment for Washington

## 2002/2004 Candidate List

Water Body Name: PEND OREILLE RIVER	Listing ID #: 9076
Parameter: Heptachlor epoxide	Township: 39N
Medium: Water	Range: 43E
Category: 2	Section: 28
Listed 98?: N	Latitude:
Listed96?: Y	Longitude:

### **Remarks**

The EPA data downloaded from STORET were challenged as not meeting the quality assurance criteria of the Water Quality Program policy on listing. The listed STORET contact (Ray Peterson) was asked to verify that these criteria were met for the data used as a basis for listing. EPA did not verify that these data meet the quality assurance criteria. Therefore, these data from STORET should not be used as a basis for listing (from 1998 list database).

### **Basis**

3 excursions beyond National Toxics Rule (40 CFR Part 131) criterion at USEPA station 543012 (at Metaline Falls) in between 1970 and 1971.

## Appendix C



## Appendix C

### Background Information on Skagit River and Pend Oreille River 303(d) Pesticides and PCBs\*

Aldrin – Broad spectrum insecticide primarily used on termites, other soil-dwelling insects, and on corn, cotton, and citrus. Production, and most major uses of aldrin, was banned in 1974. All uses were voluntarily cancelled by industry in 1987.

Alpha-BHC – Prior to 1977, alpha-BHC was a component of lindane, an insecticide used to control pests including flies, aphids, and grain weevils. Alpha-BHC is no longer produced in the United States.

Bis(2-ethylhexyl)phthalate (DEHP) – A manufactured chemical commonly added to plastics, principally PVC products, to make them flexible. It is in use today and is ubiquitous in the environment because of its use in plastics. It does not evaporate or dissolve in water easily but readily binds to soils.

DDT – Insecticide used on a variety of crops and for control of insect borne diseases. DDT was banned in 1972. DDE and DDD are toxic breakdown products. DDD also had some use as the insecticide Rothane.

Dieldrin – Aldrin and dieldrin have similar chemical structures and commercial uses. Aldrin rapidly breaks down to dieldrin in plants and animals and when exposed to sunlight or bacteria.

Endrin – An organochlorine compound, broad spectrum pesticide that was first used in the U.S. in 1951. Its use was gradually phased out through restrictions until 1984, when it's production ended. Endrin was used as a foliar treatment for agricultural crops as well as to control birds and rodents.

Heptachlor Epoxide – A breakdown product of heptachlor and a contaminant in heptachlor and chlordane formulations. Heptachlor was used to control soil insects and as a seed protectant and household insecticide. Major uses of heptachlor were suspended in 1978.

PCBs – Widely used in industrial applications as insulating fluids, plasticizers, in inks and carbonless paper, and as heat transfer and hydraulic fluids, but had a variety of other uses. EPA restricted manufacture of PCBs to sealed systems in 1977. In 1979, EPA banned PCB manufacture, processing, and distribution but allowed continued use in closed electrical systems. EPA phased out use of electrical equipment containing PCBs through regulations in 1982 and 1985.

\* Summarized from information in EPA (1992) and the Agency for Toxic Substances and Disease Registry (ATSDR) Website <http://www.atsdr.cdc.gov/toxpro2.html>.