



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

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

by
Brandee Era-Miller and Kristin Kinney

Environmental Assessment Program
Olympia, Washington 98504-7710

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Abstract

The Skagit River and Pend Oreille River are included on the 2002/2004 Section 303(d) list of the federal Clean Water Act for contaminants in fish tissue. The 303(d) listings are based on older data that may no longer be indicative of current river conditions. Fish from both rivers were collected and analyzed by the Washington State Department of Ecology to assess the appropriateness of the 303(d) listings.

Total PCBs, though found at concentrations below the 30th percentile when compared to statewide levels, continued to exceed NTR human health criteria in most samples. DDT metabolites and dieldrin were also detected but were present at concentrations below the NTR human health criteria. Over half of the contaminants analyzed for were not detected.

It is recommended that both the Skagit and Pend Oreille rivers should be included in Category 5 of the 303(d) list for total PCBs in fish tissue. A total of six other contaminant listings for fish tissue were recommended for removal from the list.

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Introduction

The Washington State Department of Ecology (Ecology) Water Quality Program requested that the Skagit and Pend Oreille rivers be re-assessed for violations of water quality standards. These rivers have been proposed for listing on the proposed 2002/2004 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.

Ecology has five categories for tracking 303(d) listings. Category 5 is the formal 303(d) list where U.S. Environmental Protection Agency (EPA) approvals 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 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 Present Study.

River Segment	Matrix	303(d)-Listed Parameter	1996 303(d) List	1998 303(d) List	2002/2004 303(d) List (Category)
<i>Skagit River</i>					
North	Fish Tissue	4,4'-DDE	N	N	5
North	Fish Tissue	4,4'-DDT	N	N	5
North	Fish Tissue	Total PCBs	N	N	5
North	Fish Tissue	Alpha BHC	N	N	5
South	Fish Tissue	4,4'-DDE	N	N	2
South	Fish Tissue	Total PCBs	N	N	2
South	Fish Tissue	Dieldrin	N	N	2
South	Fish Tissue	Bis(2-ethylhexyl)phthalate	N	N	2
<i>Pend Oreille River</i>					
South	Fish Tissue	Aldrin	N	N	5
North	Water	4,4'-DDE	Y	N	2
North	Water	4,4'-DDD	Y	N	2
North	Water	4,4'-DDT	Y	N	2
North	Water	Heptachlor Epoxide	Y	N	2
North	Water	Heptachlor	Y	N	2
North	Water	Aldrin	Y	N	2
North	Water	Dieldrin	Y	N	2
North	Water	Endrin	N	N	2

Bold = Category 5 Listings

The proposed 303(d) listings for the Skagit and Pend Oreille rivers are based on data that may no longer be indicative of the current river conditions. Data for these listings were collected between the late 1960s and early 1990s and could be outdated. The Category 2 listings for the Skagit River are a result of sampling salmon tissue. As anadromous (sea-run) animals, salmon do not adequately represent local freshwater conditions.

The data collected from the current study will assist the Water Quality Program in determining the appropriateness of both the Category 2 and 5 listings in the Skagit and Pend Oreille rivers for the proposed 2002/2004 303(d) list. The present verification study was conducted by Ecology's Environmental Assessment Program, Toxics Studies Unit.

Background

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 Skagit 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).

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 candidate 2002/2004 Category 5 listings for 4,4'-DDE, 4,4'-DDT, alpha-BHC, and total PCBs as well as candidate Category 2 listings for 4,4'-DDE, total PCBs, dieldrin, and bis(2-ethylhexyl)phthalate 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 www.atsdr.cdc.gov/toxpro2.html.

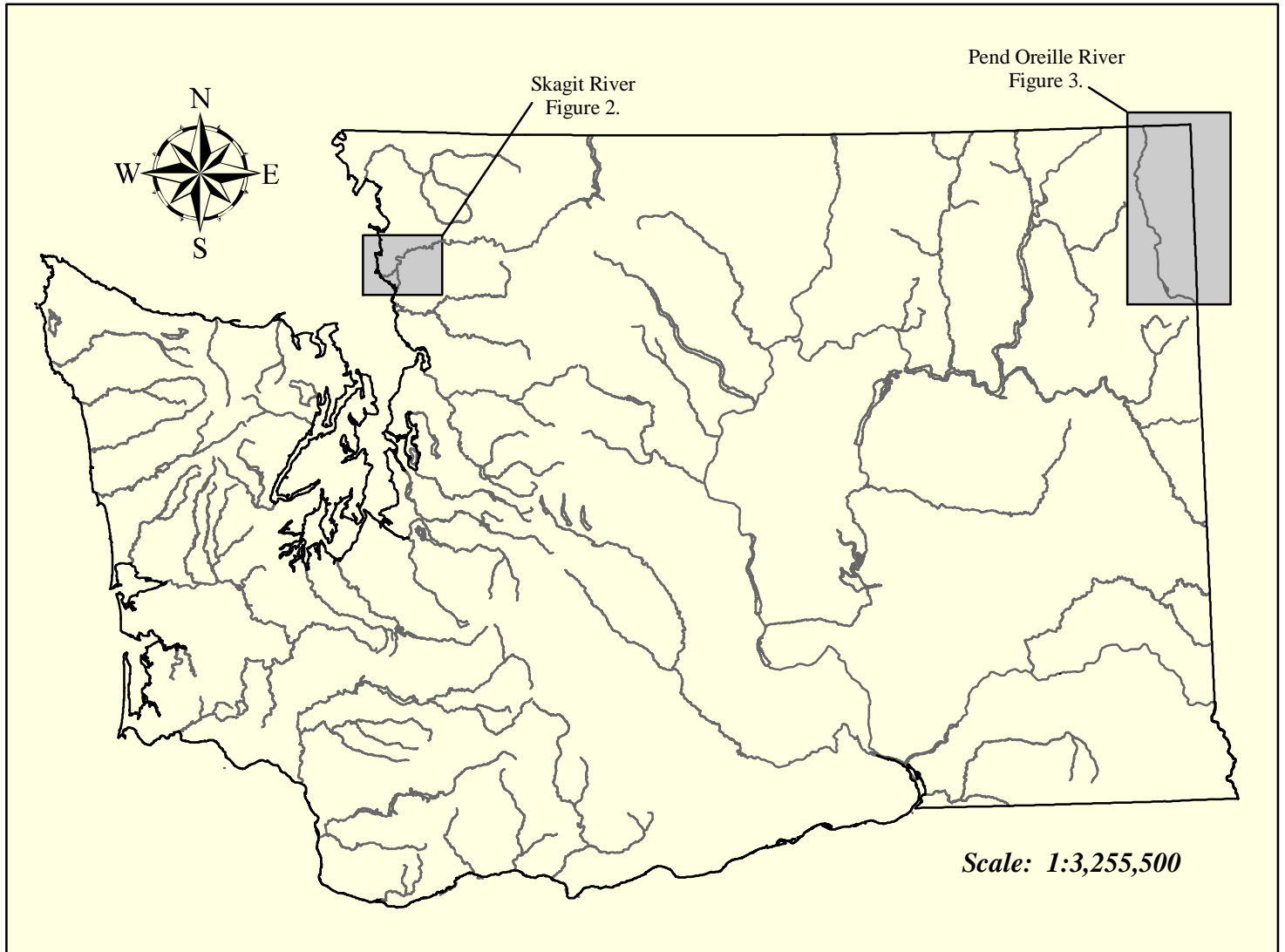


Figure 1. Map of Washington State Showing the General Locations of the Skagit River and Pend Oreille River Sampling Areas

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*), the study found that 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 sample, also exceeded criteria.

Table 2. Chlorinated Pesticide and PCB Concentrations (ug/Kg, wet weight) in Fish 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	47	19	32
4,4'-DDE	33	28	32
4,4'-DDD	31	5	45
Total DDT	111	52	
Alpha-BHC	4	4	1.7
PCB-1260	36	28	
Total PCBs	36	28	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.

Data for the Category 2 listing came from the Washington Department of Fish and Wildlife's Puget Sound Ambient Monitoring Program (PSAMP) database. The results were from several composites of Chinook salmon (*Oncorhynchus tshawytscha*) collected in 1992. The data did not result in a Category 5 listing because salmon are anadromous and the source of 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 four percent of the watershed lies within Washington State (Dames and Moore Inc. et al., 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.

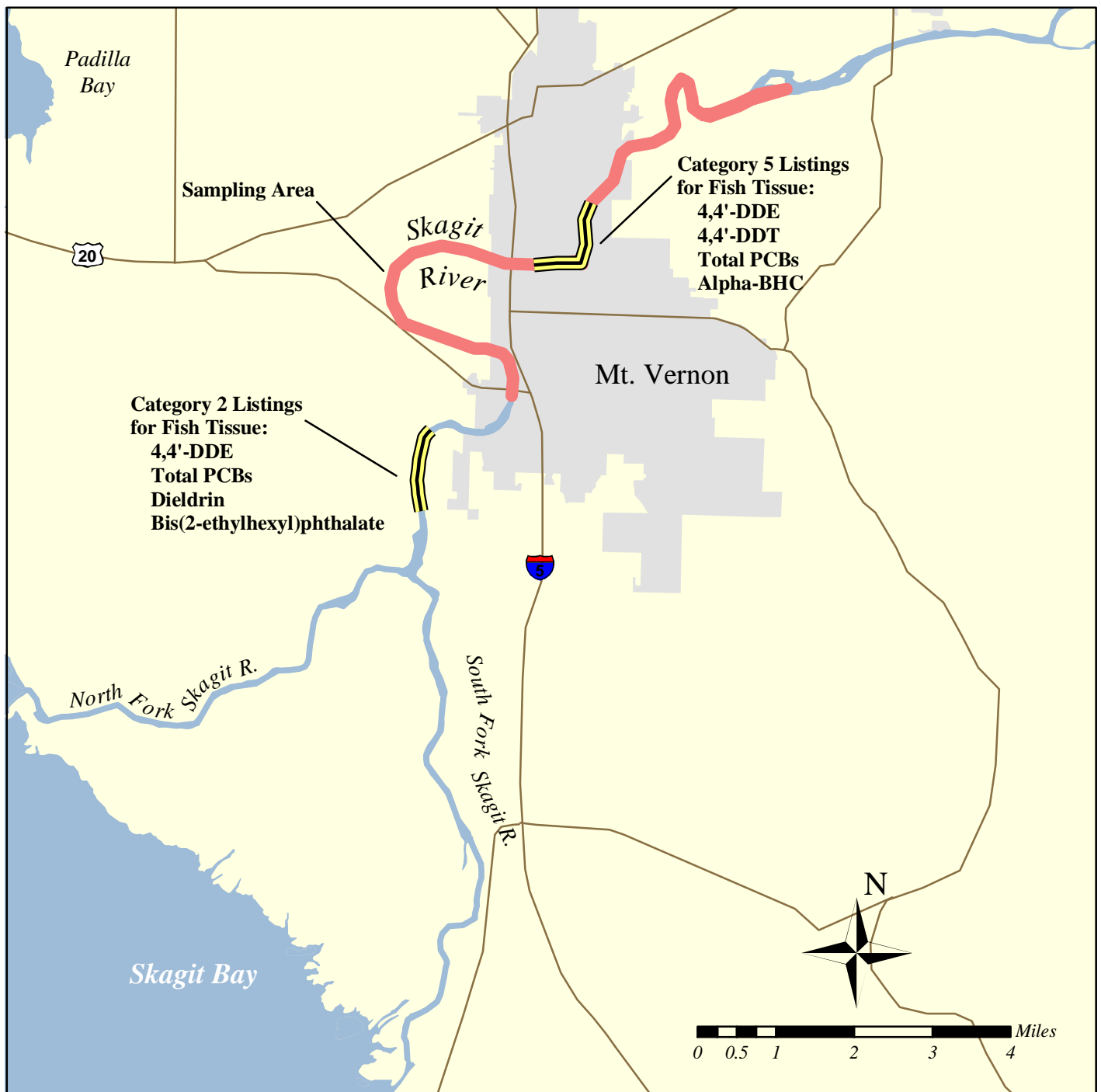


Figure 2. Lower Skagit River Showing Sampling Area for the Present Study and the 303(d)-Listed River Segments

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. et al., 1995).

The Pend Oreille River has a candidate 2002/2004 Category 5 listing for aldrin in fish tissue and candidate 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 www.atsdr.cdc.gov/toxpro2.html.

Figure 3 shows the locations of the 303(d) listings in the Pend Oreille River. 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 listing error. 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.

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

Species:	1989 ¹		2002 ²		National Toxics Rule Criteria*
	Largescale Sucker	Largemouth Bass	Largescale Sucker	Brown Trout	
Tissue:	whole	muscle	whole	muscle	
No. in Comp:	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	3.2 J	0.65
Dieldrin	16 U	15 U	1 U	1 U	0.65
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

¹ = Hopkins, 1991

² = EPA, 2004 (unpublished data)

J = Estimated value

U = Not detected at or above reported result

Bolded values exceed NTR criteria

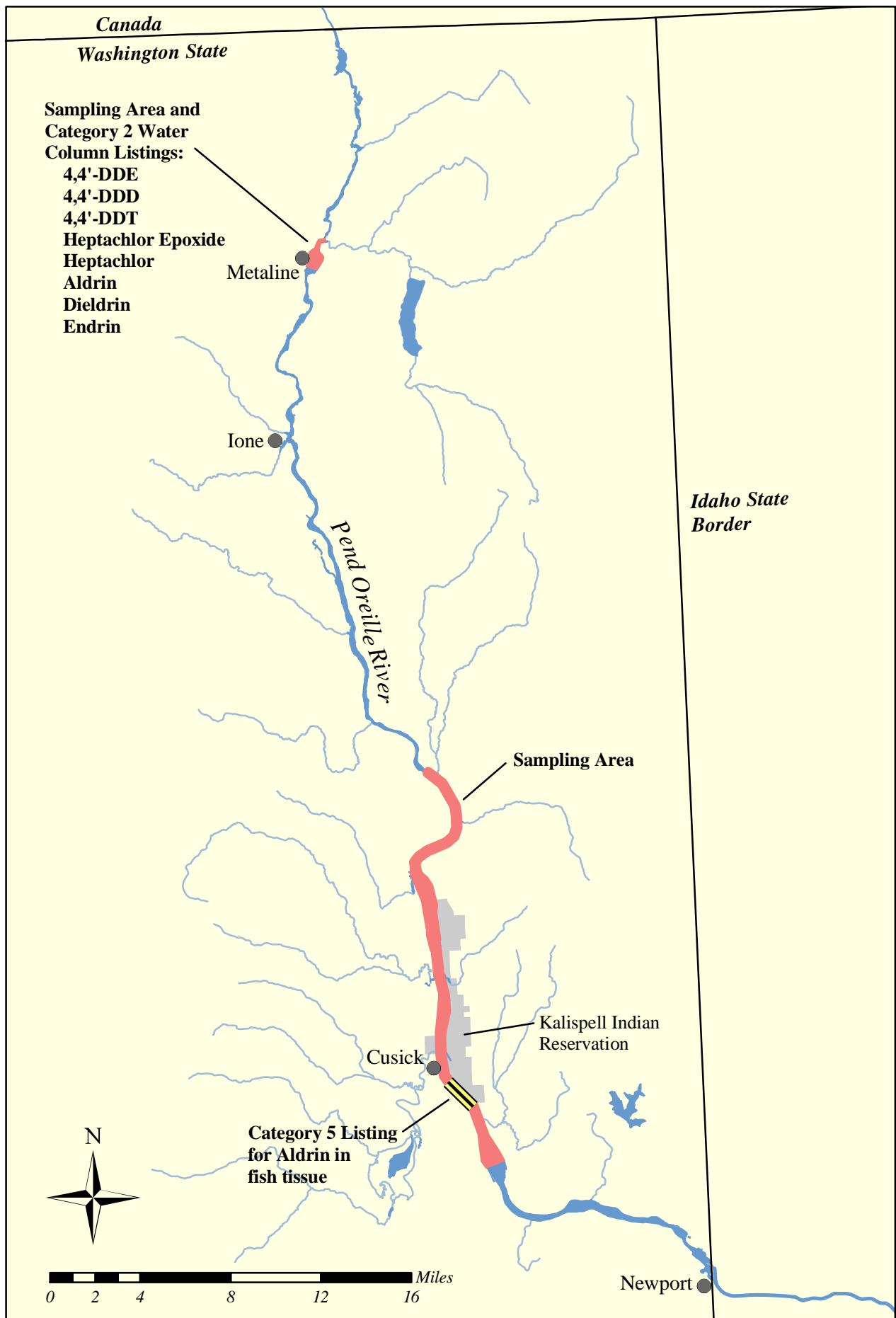


Figure 3. Pend Oreille River Showing Sampling Areas for the Present Study and the 303(d)-Listed River Segments

Interestingly, 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 collected in 2002 from the Pend Oreille River exceeded NTR human health criteria (Table 3). These data support 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 not meet 303(d) listing criteria 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 data came from EPA's STORET database and were collected between 1969 and 1971. The data were listed under Category 2 because Ecology was not able to obtain sufficient quality assurance/quality control information for the data from EPA. The data were downloaded from legacy STORET and are shown in Appendix D.

Methods

Project Description

Fish tissue composite samples were collected from selected segments of the Skagit and Pend Oreille rivers. The fish tissue was analyzed for the Category 2 and Category 5 contaminants in order to evaluate the appropriateness of the current 303(d) listings.

Category 2 water column contaminants for the Pend Oreille River were 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 be lipophilic and will bioaccumulate in fish tissue, and if present in the water column, there is a high probability that these contaminants would be detected through the analysis of fish tissue.

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 were based on the following:

A segment will be placed on the 303(d) list due to toxic pollutants in fin fish muscle or whole shellfish when either the average of three single-fish samples with the highest concentration of a given chemical or one composite sample made up of at least five fish exceeds the criteria for human health impacts based on EPA's bio-concentration factors and water column criteria established under the National Toxics Rule. A segment will be placed in the Waters of Concern Category (Category 2) when any one tissue sample exceeds the criteria.

Sampling Design

The segments on the lower Skagit River having Category 2 and 5 listings were close enough to be combined into one sampling transect. The north and south Pend Oreille River segments were sampled separately. The sampling transects and 303(d)-listed segments are shown in Figures 2 and 3. Location descriptions can be found in Appendix E, Table E-1.

All fish tissue samples for the study were composites of five individual fish. Fish of at least legal size were selected. Only resident fish species were chosen for the study.

Fish using different feeding strategies were collected from each river: predator species and bottom-feeding species. This sampling method was used to ensure that contaminant concentrations within the food chain were represented. For both the Skagit and Pend Oreille rivers, suckers were targeted for the bottom-feeding species, and predator species varied depending on species availability at the time of sampling.

Field Procedures and Sample Preparation

All required state and federal collection permits were obtained prior to fish collection. Fish from the Skagit River were collected with Ecology's electrofishing boat. Fish from the Pend Oreille River were collected by the Kalispel Tribe of Indians with assistance from Ecology through a combination of boat electrofishing, fyke nets, and gill nets. Fishing transects were recorded by GPS and can be found in Appendix E, Table E-1.

Fish selected for analysis were humanely killed with a sharp blow to the head, given an ID number, weighed, and measured. Data on fish weight, length, sex, and age are included in Appendix E, Table E-2. Specimens were individually wrapped in heavy aluminum foil, placed in plastic bags, and kept cold while in the field. Fish were then placed in a freezer (-20°C) at the Ecology Headquarters building immediately upon return from the field. Fish were frozen within 48 hours of collection.

Preparation of fish tissue samples followed EPA (2000) guidance and took place at Ecology Headquarters in Lacey, Washington. Precautions were taken to minimize contamination during sample processing. Persons preparing samples wore non-talc nitrile gloves and vinyl aprons. Work surfaces were covered with heavy grade aluminum foil. Gloves, aluminum foil, and dissection tools were changed between composite samples.

Samples for analysis were prepared by partially thawing the fish to remove the foil wrapper and rinsing in de-ionized water to remove adhering debris. The scales were removed, and the entire skin-on muscle fillet from either one or both sides of each fish were removed with stainless steel knives.

Fish fillets from the Pend Oreille River were homogenized by several passes through a Kitchen-Aid® food processor. Skagit River fish fillets were homogenized with a stainless steel homogenizer and a glass/stainless steel blender. In order to avoid contamination with tissue samples for bis(2-ethylhexyl)phthalate analysis, plastics were avoided during processing.

Composite samples were made up of equal-weight aliquots from each of five fish. The samples were homogenized to uniform color and consistency and placed in jars, specifically-cleaned for low-level organics analyses, and sent to Manchester Environmental Laboratory (MEL) for analysis. Containers and holding times for the fish tissue samples are shown in Table 4. Excess tissue was archived in freezers at Ecology Headquarters.

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

The sex of each fish was recorded during processing. Anatomical structures (scales, otoliths, and/or opercles, as appropriate for each species) were removed and sent to the Washington Department of Fish and Wildlife (WDFW) in Olympia, Washington for aging analysis. Fin clips and muscle tissue were also removed and sent to WDFW, upon their request, for potential future DNA analysis.

Table 4. Containers and Holding Times for Fish Tissue Samples.¹

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
Bis(2-ethylhexyl)phthalate	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 one year

¹ MEL, 2003 and PSEP, 1996

Laboratory Analysis

Target parameters, reporting limits, and analytical methods used for this study are shown in Table 5. The reporting limits are those that MEL achieved for the fish tissue analysis with the stated analytical methods.

Table 5. Reporting Limits and Analytical Methods Used by MEL.

Parameter	MEL Reporting Limits (ug/Kg ww)	Sample Preparation Method	Analytical Method
Bis(2-ethylhexyl)phthalate	92 – 100	EPA 3540/3620	EPA 1625 & 8270
4,4'-DDT	0.21 – 0.50	“	EPA 8081
4,4'-DDE	0.50	“	“
4,4'-DDD	0.19 – 0.50	“	“
Aldrin	0.44 – 0.50	“	“
Dieldrin	0.44 – 0.50	“	“
Endrin	0.46 – 1.2	“	“
Alpha-BHC	0.44 – 0.50	“	“
Heptachlor	0.44 – 0.50	“	“
Heptachlor Epoxide	0.44 – 0.50	“	“
PCB Aroclor 1248	4.4 – 9.8	EPA 3540/3620/3665	EPA 8082
Total PCB Aroclors	4.4 – 5.0	“	“

Data Quality

The data are useable as qualified by MEL. Most of the laboratory quality control (QC) samples met both MEL's established QC limits and the measurement quality objectives established by the Quality Assurance (QA) Project Plan (Era-Miller and Kinney, 2004) for the study, with few exceptions. A summary of MEL's QA discussion is given below. More detailed explanations are presented in the case narratives from MEL (Appendix F).

Laboratory QC samples for the study included laboratory control samples (LCS), method blanks, matrix spikes, matrix spike duplicates, laboratory duplicates, and surrogate recoveries. The percent recoveries of the LCS, matrix spikes, and surrogate standards were used as a measure of accuracy and bias that can result from the laboratory analysis. Surrogate standards were added to every sample prior to extraction, while matrix spikes were added to only one sample within a sample batch. Matrix spike recoveries are more reliable than surrogate standard recoveries for chlorinated pesticide analysis in fish tissue, and therefore greater weight should be given to matrix spike recoveries over surrogate standard recoveries as measures of analytical accuracy and bias (MEL, 2004).

The LCS and matrix spike recoveries were well within the study measurement quality objective of 50 – 150%, with the exception of heptachlor and aldrin which had low recoveries. Neither analyte was detected in any of the samples. All results for aldrin and heptachlor were qualified with a "UJ," which is defined as "The analyte was not detected at or above the reported estimated result".

Surrogate recoveries were within the study measurement quality objectives of 10 – 140% for chlorinated pesticides and PCBs, and within 50 – 150% for bis(2-ethylhexyl)phthalate. Samples 04458100 (Skagit River largescale sucker) and 04458111 (Pend Oreille River largescale sucker) had low recoveries and should be considered as biased low. Analytes detected in these samples were qualified to indicate that the results are estimates ("J") or are tentatively identified ("NJ"). Analytes not detected were qualified with a "UJ".

Most of the detected PCB aroclors and some of the DDT analogs for the Pend Oreille River fish tissue samples were qualified with a "NJ" due to differences between the two chromatographic columns used to measure the analytes. This likely occurred because another compound was interfering with the PCBs and DDT. Other results were qualified as estimated concentrations (J) when the reported results were between the reporting limit and the method detection limit.

The relative percent differences (RPD) of laboratory duplicates and matrix spike duplicates were used to measure precision. Table 6 shows the RPDs for the laboratory duplicates and matrix spike duplicates. With the exception of total PCBs in the Skagit River sample and heptachlor in the matrix spikes, all detected results were within the study measurement quality objective of $\leq 50\%$ RPD. The RPD of 113% for total PCBs in the Skagit River is high, but only as a function of low concentrations in the samples. An RPD could not be quantified for heptachlor epoxide due to the low recovery of 0% in matrix spike duplicate. RPDs were not calculated where chemicals were not detected.

Table 6. Precision of Laboratory Duplicate Results (ug/Kg, wet weight) and Percent Matrix Spike Recoveries.

Station Name: Species: Sample No:	Skagit River Largescale Sucker 04458100			Pend Oreille River Largescale Sucker 04458109			Matrix Spike/ MS Duplicate Recovery (%)		
Parameter	Result	Duplicate	RPD	Result	Duplicate	RPD	Spike	Duplicate	RPD
Bis(2-ethylhexyl)phthalate	nd	nd	nc	--	--	nc	107	112	5%
Alpha-BHC	nd	nd	nc	nd	nd	nc	61	67	9%
Aldrin	nd	nd	nc	nd	nd	nc	41	43	5%
Dieldrin	nd	nd	nc	nd	nd	nc	87	93	7%
Endrin	nd	nd	nc	nd	nd	nc	59	65	10%
Heptachlor	nd	nd	nc	nd	nd	nc	27	0	nc
Heptachlor epoxide	nd	nd	nc	nd	nd	nc	62	66	6%
4, 4' -DDE	1.2	1.6	29%	3.1	3.5	12%	91	85	7%
4, 4' -DDD	0.30	0.23	26%	0.58	0.64	10%	75	82	9%
4, 4' -DDT	0.32	0.27	17%	0.48	0.52	8%	69	76	10%
Total DDT	1.82	2.1	14%	4.16	4.66	11%	--	--	nc
PCB-1016	nd	nd	nc	nd	nd	nc	67	67	0%
PCB-1221	nd	nd	nc	nd	nd	nc	--	--	nc
PCB-1232	nd	nd	nc	nd	nd	nc	--	--	nc
PCB-1242	nd	nd	nc	nd	nd	nc	--	--	nc
PCB-1248	nd	nd	nc	nd	nd	nc	--	--	nc
PCB-1254	2.0	3.2	46%	10	12	18%	--	--	nc
PCB-1260	nd	4.0	nc	6.9	7.2	4%	95	81	16%
PCB-1262	nd	nd	nc	nd	nd	nc	--	--	nc
PCB-1268	nd	nd	nc	nd	nd	nc	--	--	nc
Total PCBs	2.0	7.2	113%	16.9	19.2	13%	--	--	nc
% lipids	1.27	1.36	7%	2.66	2.63	1%	--	--	nc

-- = not analyzed for

nc = not calculated

nd = not detected

RPD = Relative Percent Difference

A certified standard reference material (SRM)¹ was analyzed to determine how accurately MEL analyzed alpha-BHC, dieldrin, heptachlor epoxide, and DDT (Table 7). Accuracy is measured here as the percent difference between the study value and certified reference value. With the exception of Alpha-BHC and 4,4'-DDT, the results appear to be biased low, indicating that the true concentrations in the environment may be slightly higher than study concentrations show.

Table 7. Percent Difference of Standard Reference Material and Present Study Fish Tissue Concentrations for Alpha-BHC, Heptachlor Epoxide, Dieldrin, and DDT analogs (ug/Kg, ww).

Parameter	Study Value	Mean SRM Value*	% Difference
Alpha-BHC	7.8	5.72 ± 0.65	+31
Heptachlor Epoxide	4.6 NJ	5.50 ± 0.23	-18
Dieldrin	27 J	32.5 ± 3.5	-18
4,4'-DDE	330	373 ± 48	-12
4,4'-DDD	8.8 NJ	17.7 ± 2.8	-67
4,4'-DDT	58 J	37.2 ± 3.5	+44

* = The certified value is the weighted mean of the results of four to six analytical methods. The ± values quantify the uncertainty about the mean as 95% confidence intervals.

J = The analyte was positively identified. The associated numerical result is an estimate.

NJ = There is evidence that the analyte is present. The associated numerical result is an estimate.

¹ National Institute of Standards & Technology (NIST) SRM 1946 – Lake Superior Fish Tissue

Results and Discussion

Skagit River

Results from the Skagit River fish tissue samples are shown in Table 8.

The only chemical to exceed the NTR human health criteria was total PCBs. Over half of the analyzed contaminants were not detected at or above detection limits in any of the fish tissue composite samples. Those that were detected were present at low concentrations.

Detected contaminants included all three DDT analogs, PCB aroclors 1254 and 1260, and dieldrin. Dieldrin was detected in only one composite sample. PCBs were detected in all but one sample. Non-detected chemicals in the Skagit River included bis(2-ethylhexyl)phthalate, alpha-BHC, aldrin, endrin, heptachlor, heptachlor epoxide, and some of the PCB aroclors.

Based on the exceedances of NTR human health criteria, the Skagit River should be placed on the Category 5 303(d) list for total PCBs in fish tissue. The other historical chemical listings – 4,4'-DDE, 4,4'-DDT, alpha-BHC, dieldrin, and bis(2-ethylhexyl) phthalate – for fish tissue should be moved to Category 1 (Meets Tested Standards). Specific 303(d)-listing recommendations for both the Category 2 and 5 fish tissue listings are provided in the *Conclusions and Recommendations* section of this report.

Table 9 gives a comparison between the historical and current 303(d)-listed contaminants in fish tissue composite samples from the Skagit River. Contaminant levels appear to be decreasing overall. Total DDT shows the most dramatic decline with concentrations decreasing by one to two orders of magnitude.

Pend Oreille River

Results from the Pend Oreille River fish tissue samples are shown in Table 10.

The only chemical to exceed the NTR human health criteria was total PCBs. Over half of the analyzed contaminants were not detected at or above detection limits in any of the fish tissue composite samples. Those that were detected were present at low concentrations.

Detected contaminants included all three DDT analogs and PCB aroclors 1254 and 1260. They were detected in all but one sample. Non-detected chemicals in the Pend Oreille River included bis(2-ethylhexyl)phthalate, alpha-BHC, aldrin, endrin, dieldrin, heptachlor, heptachlor epoxide, and some of the PCB aroclors.

Table 8. Chemical Concentrations (ug/Kg ww) for Skagit River Fish, Skin-On Fillets (collected October 2004)

Sample ID:	SK LSS-1	SK LSS-2	SK MWF-1	SK MWF-2	SK PEA-1	NTR Human Health Criteria
Sample Lab No:	4458100*	04458101	04458102	04458103	04458104	
Species:	Largescale Sucker		Mountain Whitefish		Peamouth	
Mean fish age (yrs)	8.2	8.4	2.4	2.6	6.2	
Lipids (%)	1.32	1.05	0.25	1.82	1.65	
Bis(2-Ethylhexyl)-phthalate	98 U	98 U	100 U	92 U	99 U	767
Alpha-BHC	0.47 U	0.5 U	0.5 U	0.49 U	0.49 U	1.7
Aldrin	0.47 UJ	0.5 UJ	0.5 UJ	0.49 UJ	0.49 UJ	0.65
Dieldrin	0.47 U	0.5 U	0.5 U	0.49 U	0.27 J	0.65
Endrin	0.47 U	0.5 U	0.5 U	0.49 U	0.49 U	3216
Heptachlor	0.47 UJ	0.5 UJ	0.5 UJ	0.49 UJ	0.49 UJ	2.4
Heptachlor epoxide	0.47 U	0.5 U	0.5 U	0.49 U	0.49 U	1.2
4,4'-DDE	1.4 J	1.6	3.2	4.6	2.3	32
4,4'-DDD	0.27 J	0.5 U	0.5 U	0.58	0.27 J	45
4,4'-DDT	0.30 J	0.3 J	0.35 J	0.88	0.49 U	32
Total DDT	2.0 J	1.9 J	3.6 J	6.1	2.6	
PCB-1016	4.7 U	5 U	5 U	4.9 U	4.9 U	
PCB-1221	4.7 U	5 U	5 U	4.9 U	4.9 U	
PCB-1232	4.7 U	5 U	5 U	4.9 U	4.9 U	
PCB-1242	4.7 U	5 U	5 U	4.9 U	4.9 U	
PCB-1248	4.7 U	5 U	5 U	9.8 UJ	4.9 U	
PCB-1254	2.6 J	5 U	4 J	12	3.8 J	
PCB-1260	4 J	5 U	6.3	6.3	4.9 U	
PCB-1262	4.7 U	5 U	5 U	4.9 U	4.9 U	
PCB-1268	4.7 U	5 U	5 U	4.9 U	4.9 U	
Total PCBs	6.6 J	5 U	10.3 J	18.3	3.8 J	5.3

* = Results are the mean of laboratory duplicate analysis.

□ = Boxed values exceed National Toxics Rule (NTR) human health criteria for edible fish tissue.

Bold = Detected chemicals

U = The analyte was not detected at or above the reported result.

UJ = The analyte was not detected at or above the reported estimated result.

NJ = There is evidence that the analyte is present. The associated numerical result is an estimate.

J = The analyte was positively identified. The associated numerical result is an estimate.

Table 9. Comparison of Historical and Current Chlorinated Pesticide and PCB Concentrations (ug/Kg, wet weight) in Fish Tissue from the Skagit River.

Year:	1984 ¹		2004 ²		1984 ¹		2004 ²		National Toxics Rule Criteria*
Species:	Bridgelip Sucker	Largescale Sucker		Mountain Whitefish					
Length (mm)	403	450	404	225	242	247			
Weight (g)	805	931	645	139	81	125			
% Lipids	0.6	1.32	1.05	2.6	0.25	1.82			
4,4'-DDT	47	0.3	0.3	19	0.4	0.9		32	
4,4'-DDE	33	1.4	1.6	28	3.2	4.6		32	
4,4'-DDD	31	0.3	nd (0.5)	5	nd (0.5)	0.6		45	
Total DDT	111	2.0	1.9	52	3.6	6.1			
Alpha-BHC	4	nd (0.5)	nd (0.5)	4	nd (0.5)	nd (0.5)		1.7	
PCB-1254	--	2.6	nd (0.5)	--	4	12			
PCB-1260	36	4	nd (0.5)	28	6.3	6.3			
Total PCBs	36	6.6	nd (0.5)	28	10.3	18.3		5.3	

¹ = Data (Hopkins et al., 1985); Values are from one composite sample. Length and weight data are the average for the composite sample.

² = Current Study; Values are from one composite sample. Length and weight data are the average for the composite sample.

* Based on EPA bioconcentration factors and water column criteria established under the National Toxics Rule (40 CFR Part 131).

Bolded values exceed NTR criteria

-- = not analyzed for

nd = Not detected

Table 10. Chemical Concentrations (ug/Kg ww) for Pend Oreille River Fish, Skin-On Fillets (collected August - October 2004)

Sample ID:	N Pend LSS-1	N Pend LSS-2	N Pend NPM-1	N Pend YP-1	S Pend LSS-3	S Pend LSS-1	S Pend LSS-2	S Pend NPM-1	S Pend NPM-2	NTR Human Health Criteria
Sample Lab No:	04458105	04458106	04458107	04458108	4458109*	04458111	04458112	04458113	04458114	
Species:	Largescale Sucker		N Pike Minnow	Yellow Perch	Largescale Sucker			N Pike Minnow		
Mean fish age (yrs)	10.4	11.8	3.6	4	12.4	16.2	18	5.6	18.6	
Lipids (%)	1.06	1.08	1.17	0.47	2.65	1.34	1.05	0.77	2.15	
Alpha-BHC	0.47 U	0.44 U	0.49 U	0.5 U	0.46 U	0.47 UJ	0.48 U	0.5 U	0.49 U	1.7
Aldrin	0.47 UJ	0.44 UJ	0.49 UJ	0.5 UJ	0.46 UJ	0.47 UJ	0.48 UJ	0.5 UJ	0.49 UJ	0.65
Dieldrin	0.47 U	0.44 U	0.49 U	0.5 U	0.46 U	0.47 U	0.48 U	0.5 U	0.49 U	0.65
Endrin	0.47 U	1.2 UJ	0.49 U	0.84 UJ	0.46 U	0.47 U	0.48 U	0.5 U	0.49 U	3216
Heptachlor	0.47 UJ	0.44 UJ	0.49 UJ	0.5 UJ	0.46 UJ	0.47 UJ	0.48 UJ	0.5 UJ	0.49 UJ	2.4
Heptachlor epoxide	0.47 U	0.44 U	0.49 U	0.5 U	0.46 U	0.47 U	0.48 U	0.5 U	0.49 U	1.2
4,4'-DDE	1.5	0.93	0.74	0.5 U	3.3	3.50 J	2.6	1.7 J	5.4	32
4,4'-DDD	0.47 U	0.19 NJ	0.49 U	0.5 U	0.61	0.31 J	0.39 J	0.5 U	0.31 J	45
4,4'-DDT	0.21 NJ	0.27 NJ	0.49 U	0.5 U	0.5 NJ	0.44 J	0.4 NJ	0.5 U	0.49 U	32
Total DDT	1.7 J	1.4 NJ	0.74	0.5 U	4.4 J	4.3 J	3.4 J	1.7 J	5.7 J	
PCB-1016	4.7 U	4.4 U	4.9 U	5 U	4.6 U	4.7 UJ	4.8 U	5 U	4.9 U	
PCB-1221	4.7 U	4.4 U	4.9 U	5 U	4.6 U	4.7 UJ	4.8 U	5 U	4.9 U	
PCB-1232	4.7 U	4.4 U	4.9 U	5 U	4.6 U	4.7 UJ	4.8 U	5 U	4.9 U	
PCB-1242	4.7 U	4.4 U	4.9 U	5 U	4.6 U	4.7 UJ	4.8 U	5 U	4.9 U	
PCB-1248	4.7 U	4.4 U	4.9 U	5 U	4.6 U	4.7 UJ	4.8 U	5 U	4.9 U	
PCB-1254	6.8 NJ	5.2 NJ	3.2 NJ	5 U	11	11 NJ	14 NJ	4 NJ	13 NJ	
PCB-1260	10	9.3 NJ	4.2 NJ	5 U	7.1 NJ	14	9.5 UJ	3.5 J	13	
PCB-1262	4.7 U	4.4 U	4.9 U	5 U	4.6 U	4.7 U	4.8 U	5 U	4.9 U	
PCB-1268	4.7 U	4.4 U	4.9 U	5 U	4.6 U	4.7 U	4.8 U	5 U	4.9 U	
Total PCBs	16.8 J	14.5 NJ	7.4 NJ	5 U	18.1 J	25 J	14 NJ	7.5 J	26 J	5.3

* = Results are the mean of laboratory duplicate analysis

□ = Boxed values exceed National Toxics Rule (NTR) human health criteria for edible fish tissue

Bold = Detected chemicals

U = The analyte was not detected at or above the reported result.

NJ = There is evidence that the analyte is present. The associated numerical result is an estimate.

UJ = The analyte was not detected at or above the reported estimated result.

J = The analyte was positively identified. The associated numerical result is an estimate.

Based on the exceedances of NTR human health criteria, the Pend Oreille River should be placed on the Category 5 303(d) list for total PCBs in fish tissue. The historical fish tissue listing for aldrin should be moved to Category 1 (Meets Tested Standards). Recommendations for both the Category 5 fish tissue and Category 2 water column 303(d) listings are provided in the *Conclusions and Recommendations* section of this report.

Comparisons between historical and current study data for the Pend Oreille River were not made due to differences in species and types of fish tissue analyzed.

Comparison to Statewide Data for PCBs and DDT

To give more perspective on the current PCB and DDT concentrations in Skagit River and Pend Oreille River fish, data from the present study were compared to statewide concentrations and are shown in Figures 4 & 5. Each figure is a cumulative frequency plot that displays the distribution of values in the data set as percentiles. The data are plotted on a logarithmic scale.

Data for the figures were compiled from the following Ecology and EPA fish tissue studies: Davis and Johnson, 1994; Davis et al., 1995; Davis and Serdar, 1996; Davis et al., 1998; Ecology, 1995; EPA, 1992; EPA 2002a; EPA 2002b; Hopkins et al., 1985; Hopkins, 1991; Jack and Roose, 2002; Johnson and Norton, 1990; Johnson, 1997; Johnson, 2000; Johnson et al., 2004; Rogowski, 2000; Seiders and Kinney, 2004; Seiders, 1995; Serdar, Johnson, and Davis, 1994; Serdar, Yake, and Cabbage, 1994; Serdar, 1998; Serdar and Davis, 1999; Serdar, 1999; and Serdar 2003.

PCBs

As shown in Figure 4, all results for total PCBs from the Skagit and Pend Oreille rivers fell below the 30th percentile when compared to other statewide values. All but one result (from the Skagit River) still exceeds the NTR human health criterion of 5.3 ug/Kg ww.

DDT

Figure 5 illustrates that results for total DDT from the Skagit and Pend Oreille rivers fell below the 16th percentile, far below the NTR human health criteria of 31.6 and 45 ug/Kg ww for DDT analogs.

Addressing PCBs in a Statewide Context

The Federal Clean Water Act requires the development of a TMDL for Category 5-listed waters. Results from the current study indicate that the Skagit and Pend Oreille rivers should be listed for total PCBs in fish tissue. Total PCB concentrations, however, do not seem high enough to warrant a TMDL study for the Skagit and Pend Oreille rivers. Total PCB concentrations in the Skagit and Pend Oreille rivers are relatively low compared to other areas of Washington State.

An alternative to a river-specific TMDL for the Skagit and Pend Oreille rivers would be to address PCBs by a statewide approach such as a statewide TMDL. Background levels would first need to be established for PCBs. Waterbodies with 303(d) listings for PCBs could then be prioritized statewide.

Results from the Washington State Toxics Monitoring Program show that PCBs were found in 63% of fish tissue samples analyzed, and that more than half of those samples exceeded the NTR human health criteria. The results were from 80 fish tissue samples collected from nearly 50 sites between 2001 and 2004 (Keith Seiders, personal communication).

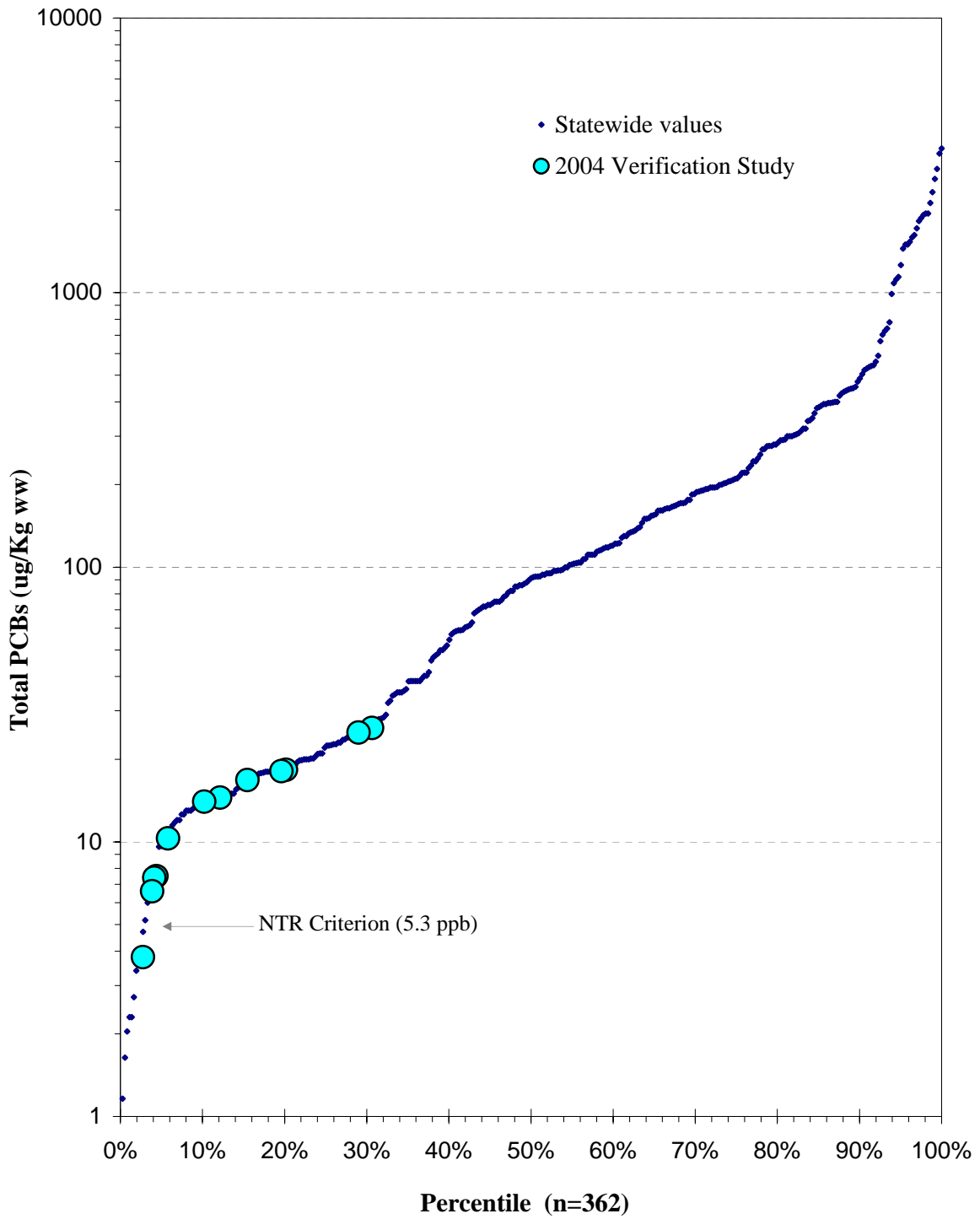


Figure 4. Cumulative Frequency Distribution of Total PCBs in Edible Fish Tissue.

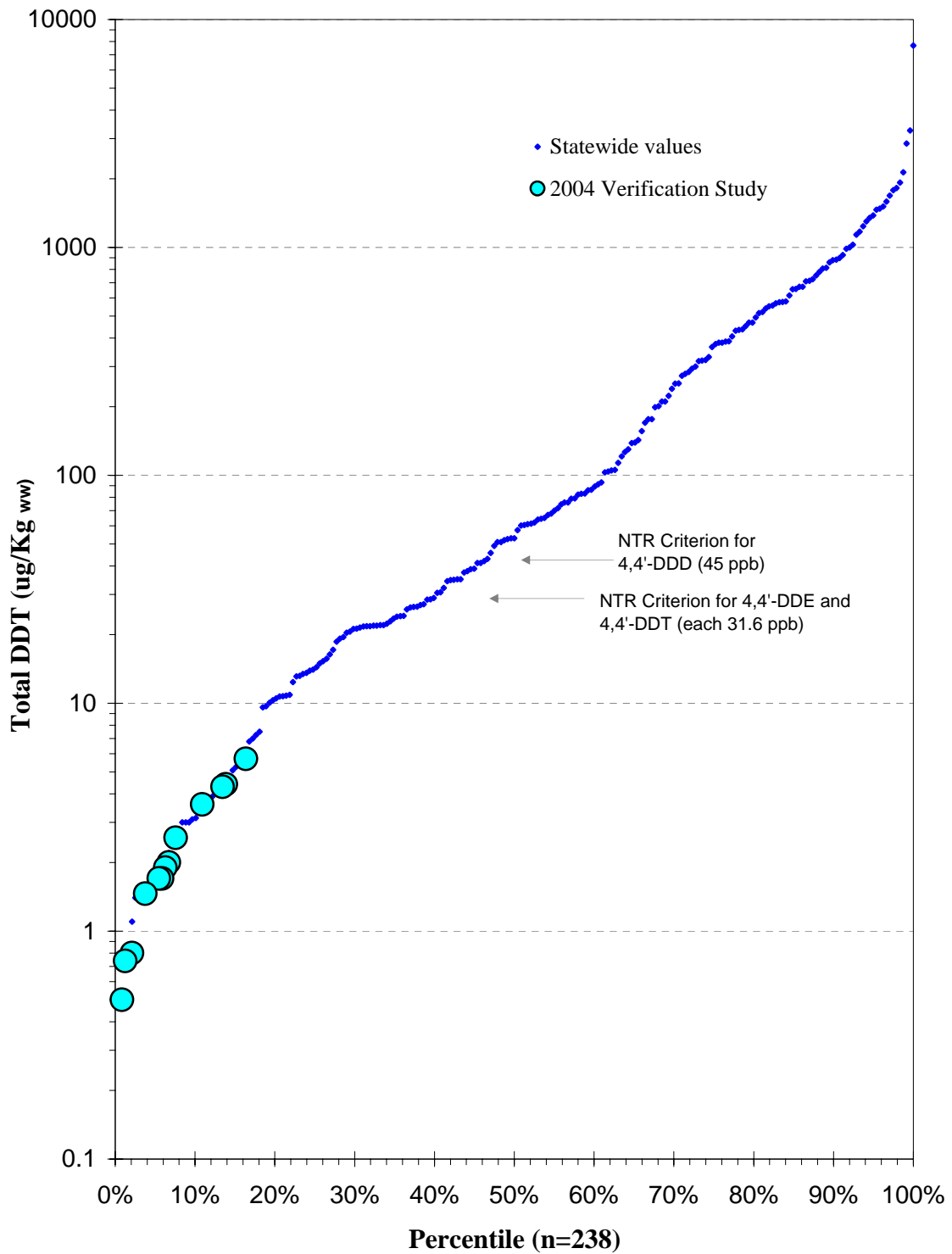


Figure 5. Cumulative Frequency Distribution of Total DDT in Edible Fish Tissue.

Conclusions and Recommendations

Skagit River

The only chemical to exceed the NTR human health criteria in Skagit River fish was total PCBs. Over half of the analyzed contaminants were not detected at or above detection limits in any of the fish tissue composite samples. Those that were detected were present at low concentrations. Contaminant levels in the Skagit River fish appear to be decreasing overall. Recommendations for 303(d) listing for the Skagit River are shown in Table 11.

Table 11. Recommended Listing Status for each of the Current 303(d) Listings for Fish Tissue in the Skagit River (Waterbody ID 5V53RP).

River Segment	Listing ID No.	303(d)-Listed Parameter	Matrix	Proposed Listing Category	Recommended Listing Category
North	14032	4,4'-DDT	Fish Tissue	5	1
"	14034	4,4'-DDE	Fish Tissue	5	1
"	14035	Alpha BHC	Fish Tissue	5	1
"	14036	Total PCBs	Fish Tissue	5	5
South	35541	4,4'-DDE	Fish Tissue	2	1
"	35550	Dieldrin	Fish Tissue	2	1
"	35548	Bis(2-ethylhexyl)phthalate	Fish Tissue	2	1
"	35570	Total PCBs	Fish Tissue	2	5

Bold = Category 5 listings

The Category 5 listings on the 2002/2004 303(d) list for the Skagit River include 4,4'-DDE, 4,4'-DDT, alpha BHC, and total PCBs in fish tissue. Results from the current fish tissue verification study indicate that, with the exception of total PCBs, these contaminants no longer exceed the NTR human health criteria. Alpha BHC, 4,4'-DDE, and 4,4'-DDT should therefore be moved to Category 1 for meeting tested standards. The total PCB listing should be retained in Category 5.

The Category 2 fish tissue listings for 4,4'-DDE, dieldrin, and bis(2-ethylhexyl)phthalate should be moved to Category 1. The Category 2 total PCB listing should be moved to Category 5.

Recommendations for the next steps in addressing PCBs in Skagit River fish include:

1. Fish tissue should be monitored again in five years.
2. Total PCBs should be addressed by a statewide approach such as statewide TMDL.

Pend Oreille River

The only chemical to exceed the NTR human health criteria in Pend Oreille River fish was total PCBs. Over half of the analyzed contaminants were not detected at or above detection limits in any of the fish tissue composite samples. Those that were detected were present at low concentrations. Recommendations for 303(d) listing for the Pend Oreille River are shown in Table 12.

Table 12. Recommended Listing Status for each of the Current 303(d) Listings for Fish Tissue and for the Water Column in the Pend Oreille River (Waterbody ID DS54SI).

River Segment	Listing ID No.	303(d)-Listed Parameter	Matrix	Proposed Listing Category	Recommended Listing Category
North	9077	4,4'-DDT	Water	2	1
"	9078	4,4'-DDE	Water	2	1
"	9079	4,4'-DDD	Water	2	1
"	9072	Endrin	Water	2	1
"	9073	Aldrin	Water	2	1
"	9074	Dieldrin	Water	2	1
"	9075	Heptachlor	Water	2	1
"	9076	Heptachlor Epoxide	Water	2	1
"	NL	Total PCBs	Fish Tissue	NL	5*
South	9080	Aldrin	Fish Tissue	5	1
"	NL	Total PCBs	Fish Tissue	NL	5*

*New listing for the 2002/2004 303(d) list

NL = not currently 303(d) listed

Bold = Category 5 listings

The Category 5 listing on the 2002/2004 303(d) list for the Pend Oreille River is for aldrin in fish tissue. Results from the current fish tissue verification study indicate that the chemical aldrin no longer exceeds the NTR human health criteria and therefore should be moved to Category 1 for meeting tested standards. Results also indicate that total PCBs exceeded NTR criteria in a majority of samples from the north and south river segments. Therefore, total PCBs should be added as Category 5 listings for fish tissue. These will be new listings.

The Category 2 water column listings were addressed through the fish tissue results. By way of the process of biomagnification, it was assumed that contaminants present in the water column would show up in the fish tissue results. The Category 2 water column contaminants are recommended to be moved to Category 1 of the 303(d) list.

Recommendations for the next steps in addressing PCBs in Pend Oreille River fish include:

1. Fish tissue should be monitored again in five years.
2. Total PCBs should be addressed by a statewide approach such as statewide TMDL.

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Appendices

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Appendix A

Water Quality Assessment Categories for the 303(d) List (Ecology, 2002)

Category 1. Meets Tested Standards Category 2. Water of Concern Category 3. No Data	Not impaired, or not known to be impaired	EPA approval and TMDL not required
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	
Category 5. The 303(d) list		EPA approval and TMDL required

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Appendix B

Descriptions of 303(d) Listings

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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.

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Appendix C

Background Information on Skagit River and Pend Oreille River 303(d)-Listed Pesticides and PCBs¹

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 were 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 – 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 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, this broad-spectrum pesticide that was first used in the U.S. in 1951. Its use was gradually phased out through restrictions until 1984, when its 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.

¹ Summarized from information in EPA (1992) and the Agency for Toxic Substances and Disease Registry (ATSDR) Website

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Appendix D

Historical Water Column Data for the Pend Oreille River

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STORET LDC - Detailed Data Report

Organization Code: **1119C050** Organization Name: **USEPA REGION 10**
Station ID: **543012** Station Alias:
Station Name: **PEND OREILLE R AT METALINE FALLS**
PACIFIC NORTHWEST
CLARK FORK-PEND OREILLE RIVER BASIN
State: **Washington** County: **Pend Oreille**
Latitude: **48deg. 51min. 56sec. N** Longitude: **117deg. 22min. 19sec. W**
Hydrologic Unit Code (HUC): **17010216**
Station Type Indicator Description: **Surface Water**
Legacy STORET Station Type: **/TYP/AMBNT/LAKE**

Start Date: **08-19-1969** Start Time: **1530**
End Date: End Time: **0**
Sample Depth: **feet** Effluent Monitoring Code:
UMK: Replicate Number:
Composite Method Code: Pipe ID:
Composite/Grab Number:
Primary/Secondary Activity Category:

Parameter Code	Parameter Long Name	Result Value	Remark Code	Composite Statistic Code
39330	ALDRIN IN WHOLE WATER SAMPLE (UG/L)	0.001	K	A
39340	GAMMA-BHC(LINDANE),WHOLE WATER,UG/L	0.005		A
39360	DDD IN WHOLE WATER SAMPLE (UG/L)	0.001	K	A
39365	DDE IN WHOLE WATER SAMPLE (UG/L)	0.001		A
39370	DDT IN WHOLE WATER SAMPLE (UG/L)	0.003		A
39380	DIELDRIN IN WHOLE WATER SAMPLE (UG/L)	0.001		A
39390	ENDRIN IN WHOLE WATER SAMPLE (UG/L)	0.003	K	A
39410	HEPTACHLOR IN WHOLE WATER SAMPLE (UG/L)	0.001	K	A
39420	HEPTACHLOR EPOXIDE IN WHOLE WATER SAMPLE (UC	0.001	K	A

STORET LDC - Detailed Data Report

Organization Code: **1119C050** Organization Name: **USEPA REGION 10**
Station ID: **543012** Station Alias:
Station Name: **PEND OREILLE R AT METALINE FALLS**
PACIFIC NORTHWEST
CLARK FORK-PEND OREILLE RIVER BASIN
State: **Washington** County: **Pend Oreille**
Latitude: **48deg. 51min. 56sec. N** Longitude: **117deg. 22min. 19sec. W**
Hydrologic Unit Code (HUC): **17010216**
Station Type Indicator Description: **Surface Water**
Legacy STORET Station Type: **/TYP/AMBNT/LAKE**

Start Date: **12-10-1969** Start Time: **1100**
End Date: End Time: **0**
Sample Depth: **feet** Effluent Monitoring Code:
UMK: Replicate Number:
Composite Method Code: Pipe ID:
Composite/Grab Number:
Primary/Secondary Activity Category:

Parameter Code	Parameter Long Name	Result Value	Remark Code	Composite Statistic Code
39330	ALDRIN IN WHOLE WATER SAMPLE (UG/L)	0.001	K	A
39340	GAMMA-BHC(LINDANE),WHOLE WATER,UG/L	0.001		A
39360	DDD IN WHOLE WATER SAMPLE (UG/L)	0.001	K	A
39365	DDE IN WHOLE WATER SAMPLE (UG/L)	0.001	K	A
39370	DDT IN WHOLE WATER SAMPLE (UG/L)	0.003	K	A
39380	DIELDRIN IN WHOLE WATER SAMPLE (UG/L)	0.001	K	A
39390	ENDRIN IN WHOLE WATER SAMPLE (UG/L)	0.003	K	A
39410	HEPTACHLOR IN WHOLE WATER SAMPLE (UG/L)	0.001	K	A
39420	HEPTACHLOR EPOXIDE IN WHOLE WATER SAMPLE (UC	0.001	K	A

STORET LDC - Detailed Data Report

Organization Code: **1119C050** Organization Name: **USEPA REGION 10**
Station ID: **543012** Station Alias:
Station Name: **PEND OREILLE R AT METALINE FALLS**
PACIFIC NORTHWEST
CLARK FORK-PEND OREILLE RIVER BASIN

State: **Washington** County: **Pend Oreille**
Latitude: **48deg. 51min. 56sec. N** Longitude: **117deg. 22min. 19sec. W**
Hydrologic Unit Code (HUC): **17010216**

Station Type Indicator Description: **Surface Water**
Legacy STORET Station Type: **/TYP/AMBNT/LAKE**

Start Date: **02-11-1970** Start Time: **930**
End Date: End Time: **0**
Sample Depth: **feet** Effluent Monitoring Code:
UMK: Replicate Number:
Composite Method Code: Pipe ID:
Composite/Grab Number:
Primary/Secondary Activity Category:

Parameter Code	Parameter Long Name	Result Value	Remark Code	Composite Statistic Code
39330	ALDRIN IN WHOLE WATER SAMPLE (UG/L)	0.001	K	A
39340	GAMMA-BHC(LINDANE),WHOLE WATER,UG/L	0.001	K	A
39360	DDD IN WHOLE WATER SAMPLE (UG/L)	0.001	K	A
39365	DDE IN WHOLE WATER SAMPLE (UG/L)	0.009		A
39370	DDT IN WHOLE WATER SAMPLE (UG/L)	0.003	K	A
39380	DIELDRIN IN WHOLE WATER SAMPLE (UG/L)	0.01		A
39390	ENDRIN IN WHOLE WATER SAMPLE (UG/L)	0.003		A
39410	HEPTACHLOR IN WHOLE WATER SAMPLE (UG/L)	0.001		A
39420	HEPTACHLOR EPOXIDE IN WHOLE WATER SAMPLE (UC	0.001		A

STORET LDC - Detailed Data Report

Organization Code: **1119C050** Organization Name: **USEPA REGION 10**
Station ID: **543012** Station Alias:
Station Name: **PEND OREILLE R AT METALINE FALLS**
PACIFIC NORTHWEST
CLARK FORK-PEND OREILLE RIVER BASIN
State: **Washington** County: **Pend Oreille**
Latitude: **48deg. 51min. 56sec. N** Longitude: **117deg. 22min. 19sec. W**
Hydrologic Unit Code (HUC): **17010216**
Station Type Indicator Description: **Surface Water**
Legacy STORET Station Type: **/TYP/AMBNT/LAKE**

Start Date: **08-26-1970** Start Time: **815**
End Date: End Time: **0**
Sample Depth: **feet** Effluent Monitoring Code:
UMK: Replicate Number:
Composite Method Code: Pipe ID:
Composite/Grab Number:
Primary/Secondary Activity Category:

Parameter Code	Parameter Long Name	Result Value	Remark Code	Composite Statistic Code
39330	ALDRIN IN WHOLE WATER SAMPLE (UG/L)	0.001	K	A
39340	GAMMA-BHC(LINDANE),WHOLE WATER,UG/L	0.001	K	A
39360	DDD IN WHOLE WATER SAMPLE (UG/L)	0.001	K	A
39365	DDE IN WHOLE WATER SAMPLE (UG/L)	0.001	K	A
39370	DDT IN WHOLE WATER SAMPLE (UG/L)	0.001	K	A
39380	DIELDRIN IN WHOLE WATER SAMPLE (UG/L)	0.001	K	A
39410	HEPTACHLOR IN WHOLE WATER SAMPLE (UG/L)	0.001	K	A
39420	HEPTACHLOR EPOXIDE IN WHOLE WATER SAMPLE (UC	0.001	K	A

STORET LDC - Detailed Data Report

Organization Code: **1119C050** Organization Name: **USEPA REGION 10**
Station ID: **543012** Station Alias:
Station Name: **PEND OREILLE R AT METALINE FALLS**
PACIFIC NORTHWEST
CLARK FORK-PEND OREILLE RIVER BASIN

State: **Washington** County: **Pend Oreille**
Latitude: **48deg. 51min. 56sec. N** Longitude: **117deg. 22min. 19sec. W**
Hydrologic Unit Code (HUC): **17010216**

Station Type Indicator Description: **Surface Water**
Legacy STORET Station Type: **/TYP/AMBNT/LAKE**

Start Date: **03-10-1971** Start Time: **1800**
End Date: End Time: **0**
Sample Depth: **0 feet** Effluent Monitoring Code:
UMK: Replicate Number:
Composite Method Code: Pipe ID:
Composite/Grab Number:
Primary/Secondary Activity Category:

Parameter Code	Parameter Long Name	Result Value	Remark Code	Composite Statistic Code
39330	ALDRIN IN WHOLE WATER SAMPLE (UG/L)	0.001		A
39340	GAMMA-BHC(LINDANE),WHOLE WATER,UG/L	0.001		A
39360	DDD IN WHOLE WATER SAMPLE (UG/L)	0.003		A
39365	DDE IN WHOLE WATER SAMPLE (UG/L)	0.002		A
39370	DDT IN WHOLE WATER SAMPLE (UG/L)	0.02		A
39380	DIELDRIN IN WHOLE WATER SAMPLE (UG/L)	0.001		A
39390	ENDRIN IN WHOLE WATER SAMPLE (UG/L)	0.001		A
39410	HEPTACHLOR IN WHOLE WATER SAMPLE (UG/L)	0.001		A
39420	HEPTACHLOR EPOXIDE IN WHOLE WATER SAMPLE (UC	0.001		A

STORET LDC - Detailed Data Report

Organization Code: **1119C050** Organization Name: **USEPA REGION 10**
Station ID: **543012** Station Alias:
Station Name: **PEND OREILLE R AT METALINE FALLS**
PACIFIC NORTHWEST
CLARK FORK-PEND OREILLE RIVER BASIN

State: **Washington** County: **Pend Oreille**
Latitude: **48deg. 51min. 56sec. N** Longitude: **117deg. 22min. 19sec. W**
Hydrologic Unit Code (HUC): **17010216**

Station Type Indicator Description: **Surface Water**
Legacy STORET Station Type: **/TYP/AMBNT/LAKE**

Start Date: **05-05-1971** Start Time:
End Date: End Time:
Sample Depth: **0 feet** Effluent Monitoring Code:
UMK: Replicate Number:
Composite Method Code: Pipe ID:
Composite/Grab Number:
Primary/Secondary Activity Category:

Parameter Code	Parameter Long Name	Result Value	Remark Code	Composite Statistic Code
39330	ALDRIN IN WHOLE WATER SAMPLE (UG/L)	0.001		A
39340	GAMMA-BHC(LINDANE),WHOLE WATER,UG/L	0.001		A
39360	DDD IN WHOLE WATER SAMPLE (UG/L)	0.002		A
39365	DDE IN WHOLE WATER SAMPLE (UG/L)	0.001		A
39370	DDT IN WHOLE WATER SAMPLE (UG/L)	0.003		A
39380	DIELDRIN IN WHOLE WATER SAMPLE (UG/L)	0.003		A
39390	ENDRIN IN WHOLE WATER SAMPLE (UG/L)	0.001		A
39410	HEPTACHLOR IN WHOLE WATER SAMPLE (UG/L)	0.001		A
39420	HEPTACHLOR EPOXIDE IN WHOLE WATER SAMPLE (UC	0.001		A

Appendix E

Location and Sample Information

Appendix E-1. Fish Tissue Sampling Location Descriptions.

Location Name	Dates of Collection	Latitude North	Longitude West	Location Description
<i>Pend Oreille River - Southern End near Cusick</i>				
Upstream extent		48° 16.41'	117° 15.06'	north of Dalkena
Downstream extent	10/19/04	48° 31.19'	117° 17.35'	south of Ruby
Centroid position		48° 23.18'	117° 17.25'	center of 20 river mile sampling transect
<i>Pend Oreille River - Northern End near Metaline</i>				
Upstream extent		48° 51.05'	117° 23.23'	boat launch at Metaline
Downstream extent	8/17 - 10/19/04	48° 51.97'	117° 22.27'	near Pend Oreille Village
Centroid position		48° 51.56'	117° 22.73'	center of 1.5 river mile sampling transect
<i>Lower Skagit River</i>				
Upstream extent		48° 28.72'	122° 15.87'	left bank fork at Hart Island
Downstream extent	10/4 - 10/5/04	48° 25.03'	122° 20.50'	boat launch at Edgewater Park
Centroid position		48° 26.83'	122° 19.28'	center of 10 river mile sampling transect

Bolded coordinates were used for study locations in Ecology's Environmental Information System (EIM)
Datum = NAD83

Table E-2. Fish Tissue Sample Biological Information.

Sample ID	Sample No.	Collection Date	Species	Total Length (mm)	Weight (g)	Sex	Age (yrs)
SK LSS-1	04458100	10/4/04	LSS	428	919	M	7
		10/4/04	LSS	435	853	F	8
		10/4/04	LSS	444	951	M	10
		10/4/04	LSS	470	960	F	7
		10/4/04	LSS	475	970	F	9
			Mean	450	931	n/a	8.2
SK LSS-2	04458101	10/4/04	LSS	378	551	F	7
		10/4/04	LSS	387	592	M	5
		10/5/04	LSS	392	637	M	8
		10/5/04	LSS	430	737	F	10
		10/5/04	LSS	433	710	M	12
			Mean	404	645	n/a	8.4
SK MWF-1	04458102	10/5/04	MWF	235	85	U	3
		10/5/04	MWF	237	76	U	3
		10/4/04	MWF	242	75	U	2
		10/5/04	MWF	243	77	U	2
		10/4/04	MWF	255	93	U	2
			Mean	242	81	n/a	2.4
SK MWF-2	04458103	10/5/04	MWF	242	98	U	2
		10/4/04	MWF	246	116	M	2
		10/5/04	MWF	248	143	M	3
		10/5/04	MWF	249	110	F	3
		10/5/04	MWF	252	160	F	3
			Mean	247	125	n/a	2.6
SK PEA-1	04458104	10/5/04	PEA	221	101	F	5
		10/5/04	PEA	240	132	F	5
		10/5/04	PEA	244	141	M	7
		10/5/04	PEA	261	145	F	7
		10/5/04	PEA	285	235	F	7
			Mean	250	151	n/a	6.2
N Pend LSS-1	04458105	10/19/04	LSS	391	585	U	16
		10/19/04	LSS	397	573	M	9
		10/19/04	LSS	425	659	U	9
		10/19/04	LSS	427	629	F	10
		10/19/04	LSS	433	711	F	8
N Pend LSS-2	04458106	10/19/04	LSS	434	707	F	10
		10/19/04	LSS	436	759	F	9
		10/19/04	LSS	437	713	F	10
		10/19/04	LSS	453	735	M	14
		10/19/04	LSS	472	975	F	16
			Mean	254	139	n/a	11.8

Sample ID	Sample No.	Collection Date	Species	Total Length (mm)	Weight (g)	Sex	Age (yrs)
N Pend NPM-1	04458107	10/19/04	NPM	216	79	F?	3
		10/19/04	NPM	237	104	F?	3
		10/19/04	NPM	240	107	M	4
		10/19/04	NPM	250	119	U	4
		10/19/04	NPM	255	130	U	4
			Mean	240	108	n/a	3.6
N Pend YP-1	04458108	10/19/04	YP	187	81	F	3
		10/19/04	YP	206	95	F	4
		10/19/04	YP	210	119	F	3
		10/19/04	YP	214	109	F	4
		10/19/04	YP	280	234	F	6
			Mean	219	128	n/a	4
N Pend LSS-3	04458109	8/18/04	LSS	440	828	F	7
		8/18/04	LSS	451	998	F	12
		8/18/04	LSS	455	868	U	10
		8/18/04	LSS	456	1065	M	14
		8/17/04	LSS	495	1204	F	19
			Mean	459	993	n/a	12.4
S Pend LSS-1	04458111	8/17/04	LSS	424	675	U	17
		10/18/04	LSS	434	708	U	21
		10/18/04	LSS	436	1167	F	12
		10/19/04	LSS	465	893	F	9
		10/19/04	LSS	492	1091	F	22
			Mean	450	907	n/a	16.2
S Pend LSS-2	04458112	10/18/04	LSS	500	1215	F	23
		10/18/04	LSS	502	1173	F	19
		10/19/04	LSS	504	1383	F	11
		10/19/04	LSS	512	1130	F	20
		10/18/04	LSS	518	1252	F	17
			Mean	507	1231	n/a	18
S Pend NPM-1	04458113	10/18/04	NPM	251	112	M	4
		8/18/04	NPM	267	153	F	5
		10/18/04	NPM	269	141	M?	6
		8/18/04	NPM	278	185	F	7
		10/18/04	NPM	295	181	M?	6
			Mean	272	154	n/a	5.6
S Pend NPM-2	04458114	8/18/04	NPM	462	1042	M	16
		8/18/04	NPM	476	1232	M	16
		8/18/04	NPM	514	1316	M	18
		10/18/04	NPM	544	1684	F	23
		10/18/04	NPM	556	1535	F	20
			Mean	510	1362	n/a	18.6

n/a = not applicable

U = Sex not determined

MWF = Mountain Whitefish, *Prosopium williamsoni*

LSS = Largescale Sucker, *Catostomus macrocheilus*

YP = Yellow Perch, *Perca flavescens*

PEA = Peamouth Chub, *Mylocheilus caurinus*

NPM = Northern Pike Minnow, *Ptychocheilus oregonensis*

Appendix F

Case Narratives from Manchester Environmental Laboratory

Data Qualifier Codes

- U - The analyte was not detected at or above the reported result.
- J - The analyte was positively identified. The associated numerical result is an estimate.
- UJ - The analyte was not detected at or above the reported estimated result.
- REJ - The data are unusable for all purposes.
- NAF - Not analyzed for.
- N - For organic analytes there is evidence the analyte is present in this sample.
- NJ - There is evidence that the analyte is present. The associated numerical result is an estimate.
- NC - Not calculated
- E - The concentration exceeds the known calibration range.
- bold** - The analyte was present in the sample. (Visual Aid to locate detected compounds on report sheet.)

Manchester Environmental Laboratory
7411 Beach Dr E, Port Orchard, Washington 98366

Case Narrative

December 22, 2004

Subject: 303 (d) Verification 2004 – Fish Tissue

Sample(s): 04-458100 to -458104

Officer(s): Brandee Era-Miller

By: Dickey Huntamer

Semivolatiles

Bis(2-ethylhexyl)phthalate

Analytical Method(s)

The semivolatile tissue samples were Soxhlet extracted with hexane-methylene chloride (50:50) following the Manchester modification of the EPA SW 846 8270 with capillary GC/MS analysis of the sample extracts. No cleanup was used on the extracts. The extracts were concentrated to 5.0 ml for analysis.

Holding Times

Samples were stored frozen until extraction. All samples were prepared and analyzed within the recommended method holding times.

Instrument Tuning

Calibration against DFTPP is acceptable for the initial calibration, continuing calibration and all associated sample analyses.

Calibration

The average relative response factors for target analytes were above the minimums and % Relative Standard Deviations were within the maximum of 15% for bis(2ethylhexyl)-phthalate in the initial calibration. The continuing calibration on December 20th was above the minimums and % Relative Standard Deviations was within the maximum of 20%.

Blanks

The target compound bis(2ethylhexyl)phthalate was not detected in the laboratory blanks.

Surrogates

The surrogate recoveries were reasonable, acceptable, and within QC limits of 25% to 121% for 2-fluorophenol, 24% to 113% for d5-phenol, 20% to 130% d4-1, 2-dichlorobenzene, 23% to 120% for d5-nitrobenzene, 18% to 137% for d14-terphenyl, 50% to 150% for d10-pyrene and 30% to 115% for 2-fluorobiphenyl.

As a check isotopically labeled bis(2ethylhexyl)phthalate-d4 was added to all samples and compared to a spike dilution at the same concentration to determine recoveries. Results are listed below.

OCT4350A1 (Blank)	98.2%
OCT4350A2 (Blank)	105.6%
04-458100	92.7%
04-458100 (LDP1)	93.9%
04-458101	83.4%
04-458101 (LMX1)	83.0%
04-458101 (LMX2)	85.5%
04-458102	87.2%
04-458103	93.1%
04-458104	85.0%
OCT4350A1 (Fortified Blank)	98.2%

Matrix Spikes

Samples -458101 was used for the tissue matrix spikes. Bis(2ethylhexyl)phthalate recoveries were within acceptable limits at 107% for LMX1 and 112% for LMX2. The Relative Percent Differences (RPD) was less than 40%.

Duplicates

One sample was analyzed in duplicate, sample -458100 (LDP1). The target compound, bis(2ethylhexyl)phthalate was not detected in either sample.

Laboratory Control Samples

Recovery for the laboratory fortified blank, OCT4350A1 (117%) was reasonable, acceptable, and within QC limits of 50% to 150%.

Comments

No significant problems were encountered in the analysis. No certified tissue reference material for bis(2ethylhexyl)phthalate was available.

The target compound bis(2ethylhexyl)phthalate was not detected in any of the samples analyzed at the reporting limit of 100 ug/Kg wet weight.

The data is acceptable as qualified.

Manchester Environmental Laboratory
7411 Beach Dr E, Port Orchard, Washington 98366

Case Narrative

December 20, 2004

Subject: Organic Chemistry 303 (d) Verification 2004 – Fish Tissue

Project No: 193504

Officer: Brandee Era-Miller

By: Dean Momohara

Summary

The samples were analyzed by the following method: SOP700009 for lipids.

The analysis requested was evaluated by established regulatory quality assurance guidelines.

Sample Information

Samples were received by Manchester Environmental Laboratory on 11/29/04. All coolers were received frozen. All samples were received in good condition. Fourteen (14) samples were received and assigned laboratory identification numbers 458100 – 458109 and 458111 – 458114.

Holding Times

The analysis was performed within established EPA holding times.

Calibration

Balances are professionally calibrated yearly and calibrated in-house daily.

Method Blanks

No analytically significant levels of analyte were detected in the method blanks associated with these samples.

Matrix Spikes

NA

Replicates

The duplicate relative percent differences of samples were within the acceptance range of 0% - 20%.

Laboratory Control Samples

NA

Other Quality Assurance Measures and Issues

U - The analyte was not detected at or above the reported result.

bold - The analyte was present in the sample. (Visual Aid to locate detected compounds on report sheet.)

Please call Dean Momohara at (360) 871-8808 to further discuss this project.

cc: Project File

Manchester Environmental Laboratory
7411 Beach Dr E, Port Orchard, Washington 98366

Case Narrative

January 7, 2005

Subject: 303(3) Verification Fish Tissue

Samples: 04458100 -04458109, 044581011 - 044581014

Officer: Brandee Era-Miller

By: M. Mandjikov

Pesticides and PCB Analysis

Analytical Method(s)

The tissue samples were extracted into methylene chloride and hexane (50/50 v/v) using a Soxhlet apparatus. Following the extraction, 10% of each extract, with the exceptions of the matrix spiked samples and the laboratory control sample, was reserved for lipid analysis. The remaining 90% of each extract was then solvent exchanged into hexane. The hexane extract was eluted through a macro Florisil[®] column first with 100% hexane (0% diethyl ether Florisil[®] fraction) and then with a 50/50% v/v mixture of a hexane/preserved diethyl ether solution (50% diethyl ether Florisil[®] fraction). The 0% fraction was solvent exchanged to iso-octane, concentrated to 1 mL, and treated with concentrated sulfuric acid prior to analysis.

The 50% fraction of each extract was adjusted to 10 mL. Five mL of each extract were back extracted with acetonitrile to remove lipids. The remaining extract volume was archived. After the back extraction, the extracts were eluted again with the 50% hexane/ether solution. After this final Florisil[®] column cleanup, the sample extracts were solvent exchanged to iso-octane, concentrated to 1 mL, and analyzed.

All the extracts were analyzed using dual column GC-ECD. These methods are modifications of EPA SW- 846 methods 3540, 3620, 3665, and 8081/8082.

Holding Times

All samples were prepared and analyzed within the method holding times.

Calibration

All the results are reported from calibration curves, initial calibration verification standards, and continuing calibration verification (CCV) standards that are acceptable and within the established QC limits.

Degradation Check

DDT and Endrin degradation are acceptable and within the established QC limits for most of the samples. Several extracts of the 50% Florisil fraction that did not undergo clean up with acid prior to analysis were bracketed by degradation check standards with unacceptable DDT degradation. However, only Heptachlor Epoxide, Dieldrin and Endrin are reported from the non-treated extracts. The degradation properties of these analytes are simulated by the behavior of Endrin and are not affected by the properties that degrade DDT. Therefore, no results are affected by this degradation and no action is taken.

Blanks

There are no target analytes detected in the method blanks.

Surrogates

100 ng of Tetrachloro-m-xylene (TMX), 4, 4-Dibromooctafluorobiphenyl (DBOB), Dibutylchlorodate (DBC) and Decachlorobiphenyl (DCB) were added to each sample and quality control (QC) sample prior to extraction. All the surrogate recoveries are within the established QC limits with the exception of:

The 0% Florisil fraction of sample 04458100 has low recoveries for TMX and DCB due to a loss of ~ 40% of the extract during the extraction procedure. All the results reported from the 0% Florisil fraction of this extract are qualified as estimates, "J" and should be considered to be biased low. The results affected are for Heptachlor, Aldrin, 4, 4' DDE, and Aroclors 1248, 1254, 1260, 1262, and 1268.

Sample 04458111 has a low recovery for DBC in the acid treated 50% fraction only. All the analyte results affected are qualified either at an estimated reporting limit, "UJ" or as an estimated result, "J" or "NJ". The results affected are for alpha-BHC, 4,4' DDE, 4,4' DDD, 4,4' DDT and Aroclors 1016, 1221, 1232, 1242, 1248 and 1254 and may be biased low.

Some of the Aroclors and 4,4'DDE did not completely elute within the 0% Florisil fraction of the extract as desired. Because these analytes have the potential to be found in both the 0% and the 50% acid treated extracts, they are qualified when either fraction is qualified due to low surrogate recoveries.

Duplicate Samples

Samples 04458100 and 04458109 were prepared in duplicate to assess the precision of this procedure. The relative percent difference (RPD) between all analyte results reported above the reporting limits (RL) is within the established QC limits.

Matrix Spiked Samples

Sample 04458114 was prepared in triplicate. Two of the replicates were spiked with 100 ng of chlorinated pesticides and 500 ng of PCB Aroclors 1016 and 1260. All analytes have recoveries within the established QC limits with the following exceptions:

One of the matrix spiked samples had no recovery for Heptachlor and the other recovered at 21%. Both spiked samples had low Aldrin recoveries. The RPD between the Heptachlor results exceeds the established QC limits whereas the Aldrin RPD is acceptable.

Neither of these analytes has been detected in any sample. All the reporting limits for these samples should be considered to be estimates and are qualified, "UJ".

Laboratory Control Samples (LCS)

A laboratory control sample (LCS) was prepared by spiking analytically clean Ottawa sand with 100 ng of Chlorinated pesticides and 500 ng of PCB Aroclors 1016 and 1260. All analytes have recoveries within the established QC limits with the exception of Heptachlor and Aldrin. All results for these analytes are qualified "UJ", at an estimated reporting limit.

Comments

Results reported between the reporting limit and method detection limit are associated with a higher degree of error. Results reported below the reporting limits are qualified as estimates, "J".

When results are reported because there is evidence that the analyte is indeed present in the sample but the RPD between the concentrations found on each column exceeds 40%, the result is considered to be non-confirmed by the second chromatographic column. This commonly occurs when another compound is interfering with the analyte of interest on one of the columns. In this situation, the result is qualified, "NJ" and the analyte should be considered to be tentatively identified (TIC).

On the occasion that an analyte peak is obscured on both columns by known or unknown compounds and it can not be quantified, the reporting limit of that analyte is raised to the level of the interference and qualified as an estimated reporting limit, "UJ".