WASHINGTON STATE
DEPARTMENT OF
ECOLOGY

Washington State Pesticide Monitoring Program

1995 Fish Tissue Sampling Report

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WASHINGTON STATE DEPARTMENT OF ECOLOGY

Washington State Pesticide Monitoring Program

1995 Fish Tissue Sampling Report

by
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Environmental Investigations and Laboratory Services Program
Olympia, Washington 98504-7710

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Abstract

Twenty-four fish tissue samples were collected from eight sites in September 1995 for the Washington State Pesticide Monitoring Program (WSPMP). Samples were analyzed for 44 target pesticides and breakdown products, and five polychlorinated biphenyls (PCBs). Total lipid content was also determined for each tissue sample.

A total of 24 pesticides and breakdown products, as well as two PCBs (Aroclor equivalents), were detected in the fish. DDT or its breakdown products, DDD and DDE, were found in all 24 samples. Hexachlorobenzene was detected in 21 samples, dieldrin in 16, and chlordane or its breakdown products in 15 samples. Four of the detected compounds – chlorpyrifos (Lorsban), DCPA (Dacthal), endosulfan (Thiodan), and trifluralin (Treflan) – are currently registered for use in Washington. Pentachlorophenol is also still used, and its breakdown product, pentachloroanisole, was found in six samples.

Results were compared to USEPA human health screening values and proposed wildlife criteria. Screening values were exceeded for one or more compounds at seven of the eight sites. Total DDT, dieldrin, and PCBs were above screening values in samples from six, five, and four of the sites, respectively. Total DDT and aldrin/dieldrin levels exceeded proposed wildlife criteria at three of the four sites where samples were analyzed as whole fish.

Four sites were sampled in the Mid-Columbia area to assess the distribution of pesticide contamination in this popular sport-fishing destination. Results from whole-fish analyses support conclusions reached by the U.S. Geological Survey that concentrations of some pesticides may pose a threat to fish and wildlife. Concentrations of total DDT and dieldrin in bass fillets were consistently high in waterbodies receiving irrigation return water. Levels of most pesticides were substantially lower in smaller fish, indicating that fishermen can reduce their risk by not eating large fish. Pesticide concentrations are also considerably lower in fish from seep lakes that do not receive direct inputs from irrigation return water.

Recommendations include (1) an intensive survey of sport fish from waterbodies within the Columbia Basin Irrigation Project area to satisfy data requirements for a human health risk assessment, and (2) addition of Cowiche Creek to the lower Yakima River "area of concern" as defined by a Washington State Department of Health recommendation to eat fewer bottom fish contaminated with DDT from the river.

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Summary

The Washington State Pesticide Monitoring Program (WSPMP) was initiated in 1991 by the Department of Ecology to monitor surface water and ground water for pesticide residues. This report describes fish tissue monitoring results for samples collected in 1995. Tissue and sediment samples were also collected in 1992 (Davis and Johnson, 1994) and 1994 (Davis and Serdar, 1996), and fish were sampled in 1993 (Davis *et al.*, 1995).

Fish were collected from Canal, Redrock, and Royal Lakes, Scooteney Reservoir, Clear and Cowiche Creeks, and the Cowlitz and Yakima Rivers. Whole samples of bottom feeding fish were collected to assess wildlife impacts, and fillets from sport fish were collected to evaluate potential risk to human health. Largescale suckers and carp were chosen as bottom feeding species. Fillets were collected from rainbow and cutthroat trout, mountain whitefish, smallmouth and largemouth bass, yellow perch, and carp. A composite of five individuals was collected for each sample. Samples were analyzed for 44 pesticides and breakdown products, and five PCB mixtures (as Aroclor equivalents).

A total of 24 pesticides and breakdown products, as well as two PCBs, were detected in the fish. DDT or its breakdown products, DDD and DDE, were found in all 24 samples. Hexachlorobenzene was detected in 21 samples, dieldrin in 16, and chlordane or its breakdown products in 15 samples. Four of the detected compounds – chlorpyrifos (Lorsban), DCPA (Dacthal), endosulfan (Thiodan), and trifluralin (Treflan) – are currently registered for use in Washington. Pentachlorophenol is also still used, and its breakdown product, pentachloroanisole, was found in six samples. A total DDT concentration of 822 μg/kg in rainbow trout fillets from Cowiche Creek is the highest level ever recorded in Washington State for this species. Aldrin was found in whole carp from Scooteney Reservoir, which is the first detection of this compound in WSPMP tissue samples.

Results were compared to USEPA human health screening values and proposed wildlife criteria. Screening values were exceeded for one or more compounds at seven of the eight sites. Only fish from Canal Lake did not contain contaminants exceeding screening values. Total DDT, dieldrin, and PCBs were above screening values in samples from six, five, and four of the sites, respectively. Total chlordane was also above the screening value in carp fillets from the Yakima River. Total DDT and aldrin/dieldrin levels exceeded proposed wildlife criteria at three of the four sites where samples were analyzed as whole fish.

Four sites were sampled in the Mid-Columbia area to assess the distribution of pesticide contamination in this popular sport-fishing destination. Results from whole-fish analyses support conclusions reached by the U.S. Geological Survey that concentrations of some pesticides may pose a threat to fish and wildlife. Concentrations of total DDT and dieldrin in bass fillets were consistently high in waterbodies receiving irrigation return water. Levels of most pesticides were substantially lower in smaller fish, indicating that fishermen can reduce their risk by not eating large fish. Pesticide concentrations are also considerably lower in fish from seep lakes that do not receive direct inputs from irrigation return water.

Recommendations

Mid-Columbia Basin

Many of the lakes and reservoirs within the Columbia Basin Irrigation Project area are popular sport fishing destinations. Available data indicate that fish in these waterbodies are contaminated with high concentrations of total DDT and dieldrin. An intensive survey of pesticide contamination in sport fish from these waterbodies should be developed to satisfy the data requirements of a human health risk assessment.

Cowiche Creek

Fish from Cowiche Creek have not been sampled prior to this study, and the creek was not included in the "area of concern" as defined by the Washington State Department of Health in their recommendation to eat fewer bottom fish from the Yakima River and its tributaries. No bottom fish were collected from Cowiche Creek, but the level of total DDT in rainbow trout from the creek was exceptionally high. Cowiche Creek should be included in the "area of concern" so that consumers of fish from this creek are aware of the risks.

Introduction

The Washington State Pesticide Monitoring Program (WSPMP) was initiated in 1991 by the Department of Ecology (Ecology) to monitor ground water and surface water, including bed sediments and associated biota such as fish, shellfish, and waterfowl for pesticide residues. The goal and objectives of the WSPMP are as follows:

Goal

To characterize pesticide residues geographically and over time in ground water and surface water (including sediments and biota) throughout Washington.

Objectives

- Identify and prioritize aquifers, lakes, and streams with known or potential pesticide contamination.
- Quantify pesticide concentrations in high priority areas.
- Document temporal trends in pesticide types and concentrations at selected sites.
- Provide data to the State Department of Health for assessment of potential adverse effects on human health.
- Assess the potential for adverse effects of pesticides on aquatic biota and other wildlife.
- Construct and maintain a pesticide database for ground water and surface water in Washington.
- Provide information for the improvement of pesticide management in Washington.

In a guidance document for assessing chemical contaminants in fish tissue, the U.S. Environmental Protection Agency (USEPA, 1995) describes two types of surveys. Initially, screening surveys are designed to identify potential problem areas by collecting one or two composite samples from a number of waterbodies. These data are used to determine where more intensive surveys should be implemented to thoroughly investigate the extent and severity of the problem. The surface water portion of the WSPMP is essentially an ongoing screening survey.

The first set of fish tissue and sediment samples was collected in 1992 (Davis and Johnson, 1994). Fish tissue was collected in 1993, but not sediments (Davis *et al.*, 1995). Both fish and sediment were sampled again in 1994 (Davis and Serdar, 1996). This report addresses fish tissue sampling for 1995; no sediment samples were collected. As a separate study for the WSPMP, marine mussels were collected from five sites in Puget Sound and one at the mouth of the Columbia River in May 1995 (Johnson and Davis, 1996). Surface water samples were also collected in April, June, August, and October of 1995, and results have been summarized in a report by Davis *et al.*, (1997).

Methods

Sampling Design

Fish tissue samples were collected for the WSPMP at eight sites (Figure 1) in September 1995. Table 1 lists sample sites, their location, and the number and type of samples collected. Latitude, longitude, and state-plane coordinates are listed for each site in Appendix A.

Tissue samples were analyzed for 44 pesticides and breakdown products, and five polychlorinated biphenyl mixtures (PCBs) (Appendix B). Samples were also analyzed for percent total lipids. The length and weight of each fish was recorded in the field and is summarized in Appendix C. Scientific names for each species collected are also listed in Appendix C.

The timing for sample collection was intended to allow spring spawners to rebuild lipid reserves, which concentrate bioaccumulative pesticides, and take place before fall spawning occurred. In addition, stream flows are lower in late summer, allowing easier and safer access.

For a screening survey, the USEPA recommends collecting one composite for each of two species at each sample site (USEPA, 1995). One species should be a bottom feeder and the other a sport fish. The USEPA document was written for use in developing advisories regarding human consumption of fish fillets. The WSPMP is designed as a screening survey, but is also to provide data to evaluate the effects of pesticides in the environment. Thus, samples of whole bottom feeding fish were collected to assess wildlife impacts, and sport fish fillets were collected to evaluate potential risk to human health.

Target species were selected based on the following criteria:

- wide geographic distribution (statewide is desirable),
- potential to bioaccumulate high concentrations of pesticides (have a high lipid content),
- popular resident sport fish,
- easily identified,
- abundant, easy to capture, and large enough to provide an adequate sample size.

Largescale suckers were chosen as the bottom feeding species because they possess nearly all of the desired attributes of a target species. No largescale suckers were found at two sites, so carp were collected and analyzed whole. No single species of sport fish is widely distributed throughout Washington, so the species of opportunity was collected: rainbow and cutthroat trout, mountain whitefish, largemouth and smallmouth bass, yellow perch, and carp.

Washington State Pesticide Monitoring Program

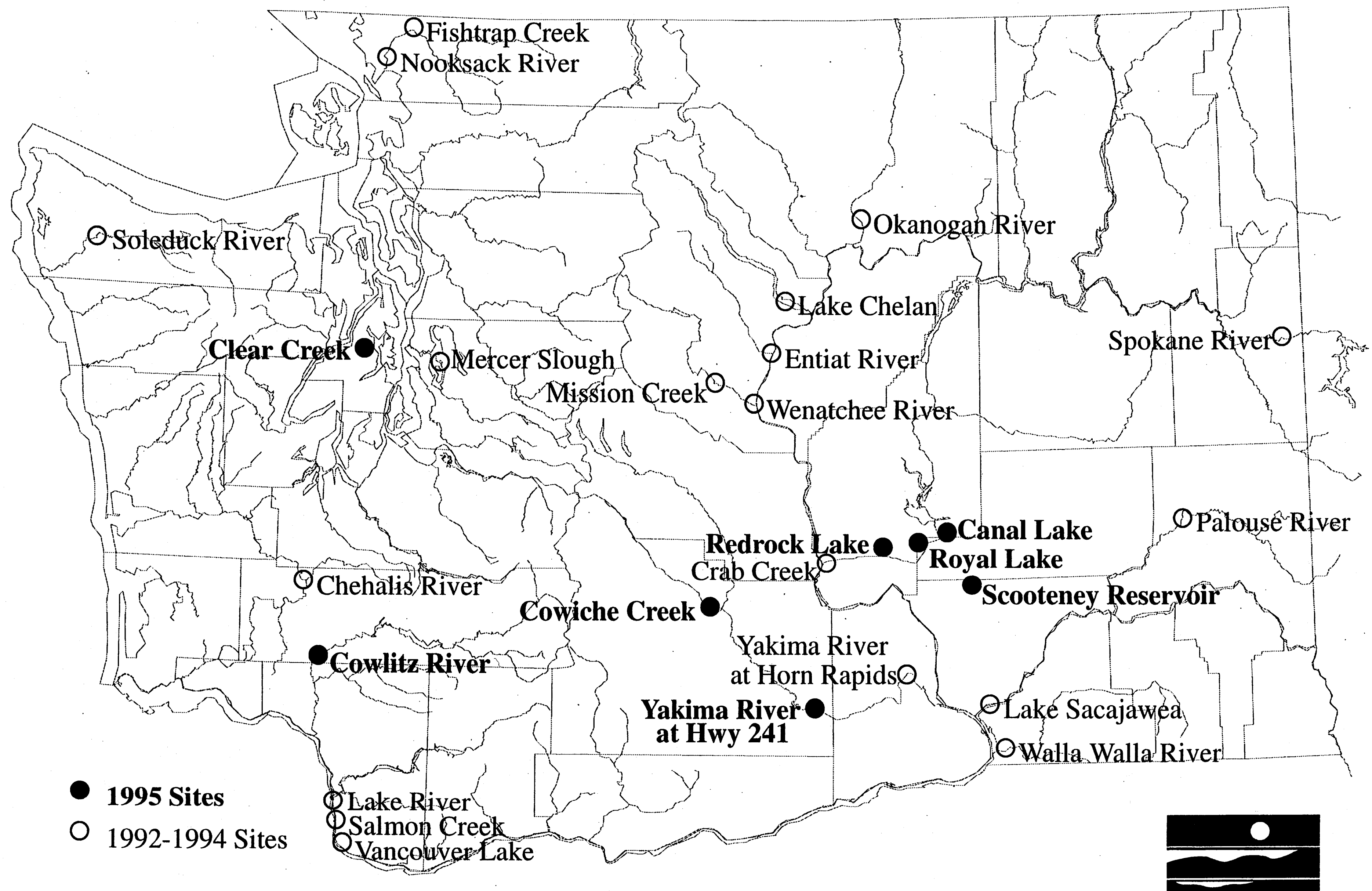

Figure 1. 1992-1995 WSPMP Fish Tissue Sampling Sites

Table 1. List of 1995 WSPMP Sampling Sites, Locations, and Number and Type of Samples

Sample Site	Location	Number of Composite Samples Whole Fish	Fillet
Mid-Columbia Watershed			
Canal Lake	North of Othello Grant County		2
Redrock Lake	Southeast of Royal City Grant County		2
Royal Lake	Northwest of Othello Adams County	1	2
Esquatzel Watershed			
Scooteney Reservoir	Southeast of Othello Franklin County	2	4
Upper Yakima Watershed			
Cowiche Creek	Northwest of Yakima Yakima County		1
Lower Yakima Watershed			
Yakima River	Southwest of Grandview Yakima County	3	2
Kitsap Watershed			
Clear Creek	At Silverdale Kitsap County		1
Lower Columbia Watershed			
Cowlitz River	Northeast of Vader Lewis County	2	3

Composite samples were analyzed rather than individual fish because composites are the most cost-effective method of estimating average contaminant concentrations (USEPA, 1995). For assessment of waterbodies to be added to Ecology's water quality limited list (section 303(d) of the federal Clean Water Act), fish composites must be composed of at least five individuals. Composite samples for the WSPMP included material from five fish. Fish for replicate tissue composites were collected at three sites – the Cowlitz and Yakima Rivers and Scooteney Reservoir – to evaluate variability between composites.

When possible, all fish collected within a composite were similar in size. Larger (older) individuals were selected when there was a choice, because they have had more time to accumulate contaminants and generally should represent a "worst-case" sample. At three sites – the Cowlitz River, Redrock Lake, and Scooteney Reservoir – fish of the same species were divided into two separate composites based on size to evaluate differences in accumulated contaminants between age classes.

Sampling Site Selection

Sampling for the WSPMP is integrated into the five-year cycle developed for the Ecology watershed approach to water quality management. Pesticide sampling for each watershed is implemented one year prior to the needs assessment scheduled for that watershed. Results from the WSPMP are used to identify areas with potential pesticide-related problems. These results are presented during needs assessments for the watersheds so potential problems can be evaluated more effectively using recent, pertinent data.

The sampling emphasis for the 1995 WSPMP was within the Kitsap, Mid and Lower Columbia, and Upper and Lower Yakima watersheds. Clear Creek is in the Kitsap watershed, and was judged the most likely stream in this watershed to be contaminated with pesticides. There was also an attempt to collect resident fish from the Union River, but migrating salmon were the only fish seen while electroshocking (none were collected). The Cowlitz River is in the Lower Columbia watershed, and was sampled upstream from the confluence with the Toutle River near Vader. This stretch of the river receives water from numerous streams that run through or near Christmas tree farms, which are probably the heaviest users of pesticides in the area.

Cowiche Creek is in the Upper Yakima watershed, and receives runoff from orchards in the Naches Heights area northwest of Yakima before discharging into the Naches River. The Yakima River was sampled near Grandview, which is in the Lower Yakima watershed. Most of the major irrigation returns in the Yakima Valley flow into the Yakima River upstream of the area sampled. The river was sampled in conjunction with Ecology's Yakima River total maximum daily load (TMDL) study (Joy and Patterson, 1997).

Canal, Redrock, and Royal Lakes are in the Mid-Columbia watershed. Scooteney Reservoir is in the Esquatzel watershed, but all of these waterbodies are part of or a result of the Columbia Basin Irrigation Project (CBIP). Many of the lakes and reservoirs in the CBIP were not present prior to the start of irrigation, or they have been significantly enhanced by irrigation water. These lakes are all heavily fished, and were selected for sampling to represent numerous other lakes and reservoirs in the CBIP that are impacted by agricultural pesticide use.

Target Analytes

Appendix B lists the fish tissue target pesticides analyzed for the WSPMP in 1995. An initial list of target compounds for fish tissue analysis was compiled from other studies or guidelines on analyzing tissue samples for bioaccumulative pesticides (USEPA, 1995; Schmitt *et al.*, 1990; Rasmussen and Blethrow, 1991; Crawford and Luoma, 1993). Pesticides recommended for monitoring in tissues in the Puget Sound Basin (Tetra Tech, 1988) were also included on the list. Endrin aldehyde and endrin ketone were added to the list because they were detected in fish samples from the Yakima River (Johnson *et al.*, 1986).

A discussion of the characteristics and interrelationships of target compounds, including current registration status, can be found in Appendix D.

Sampling Procedures, Analytical Methods, QA/QC, and Data Review

Details of sampling procedures are outlined in Appendix E. A brief discussion of analytical methods, quality assurance/quality control, and the data review is in Appendix F.

Results and Discussion

Pesticides Detected

For this report, total DDT refers to the sum of 4,4'- and 2,4'- isomers of DDT, DDD, and DDE. Total chlordane is the sum of cis- and trans-chlordane, cis- and trans-nonachlor, and oxychlordane. Total PCBs is the sum of all Aroclors. These compounds are used in this report for various comparisons because these were the most frequently detected compounds, they comprise a majority of the compounds detected, and in their summed form they simplify comparisons.

A total of 24 pesticides and breakdown products were detected in 24 fish tissue samples collected in 1995 (Table 2). In addition, two PCB mixtures (as Aroclor equivalents) were identified. A maximum of 21 target analytes were detected per sample; these were in largescale suckers from the Yakima River. The average detection rate per sample was 10 analytes: eight in fillet samples and 16 in whole-fish samples. DDT or its breakdown products, DDD and DDE, were found in all 24 samples. Hexachlorobenzene was detected in 21 samples, dieldrin in 16, and chlordane or its breakdown products in 15 samples. Four of the detected compounds – chlorpyrifos (Lorsban), DCPA (Dacthal), endosulfan (and its breakdown product, endosulfan sulfate), and trifluralin – are currently registered for use in Washington. In addition, pentachlorophenol is still in use, and its breakdown product, pentachloroanisole, was found in six samples. For reference, pesticides detected in tissue samples collected for the 1992, 1993, and 1994 WSPMP are included in Appendix G.

Comparisons with Applicable Criteria

Human Health Screening Values

As discussed earlier, the WSPMP should be interpreted as a screening survey. Standing alone, data from screening surveys are not adequate for making decisions regarding fish consumption by humans, but the USEPA recommends evaluating detected chemical contaminants with screening values to prioritize problem areas. Sites with concentrations exceeding screening values will be evaluated based on a variety of parameters. These parameters include, but are not limited to, the level of exceedance, local fish consumption patterns, and toxicity of the contaminant. If necessary, an intensive survey would be recommended to determine if consumption recommendations or advisories are warranted. The following summarizes factors that were used to calculate screening values as outlined by the USEPA (1995).

Calculation of Screening Values

Screening values for carcinogenic compounds are calculated using a risk level. A risk level is a value that predicts the increased number of cancer cases caused by a specific or multiple contaminant(s). A risk level of $1x10^{-6}$ is the probability that one person in a million will contract cancer as a result of long-term exposure to the contaminant(s) through consumption of fish tissue.

Table 2. Pesticides and PCBs Detected in 1995 WSPMP Fish Tissue Samples (μg/kg (ppb) wet weight)

	Canal Lake		Redrock Lake		Royal Lake		Scooteney Reservoir					
	LM Bass	Perch	Largemouth Bass		SM Bass	Carp	Smallmouth Bass		Largemouth Bass		Carp	
Tissue Type	Fillet	Fillet	Fillet		Fillet[1]	Whole	Fillet		Fillet		Whole	
Size Group or Replicate			Small	Large[1]			Small	Large	Small	Large[1]	Rep-1	Rep-2
Percent Total Lipid	1.07	0.56	0.85	1.75	1.99	10.62	0.71	1.73	0.68	1.88	8.29	7.09
2,4'-DDE				0.7 J		8.4		0.9 J			1.9 J	1.3 J
4,4'-DDE	5.2	3.4 J	59	130	68	2000	16	120	24	63	370	250
2,4'-DDD				1.6 J		17		1.3 J			4.6	3.2 J
4,4'-DDD			4.9	15	4.2 J	120	3.2 J	13	3.3	6.7 J	46	28
2,4'-DDT				0.7 J		17		1.7 J			1.8 J	1.7 J
4,4'-DDT			2.0 J	6.4	3.8 J	13	3.4 J	23	1.4 J	4.5 J	4.1	3.4 J
total DDT	5.2	3.4	66	154	75	2175	23	160	29	74	428	288
DDMU			1.3 J	5.3 J	1.2 J	61					10	7.1 J
cis-chlordane						6.2 J					2.4 J	1.4 J
trans-chlordane						1.6 J					1.5 J	0.8 NJ
oxychlordane											0.9 NJ	
cis-nonachlor								1.0 J			2.1 J	1.5 J
trans-nonachlor				0.7 J		11 J		1.8 J		0.7 J	4.6	3.1 J
total chlordane				0.7		19		2.8		0.7	11.5	6.8
aldrin											0.7 NJ	0.6 NJ
dieldrin			4.1	8.6	8.2 J	42	2.9 J	13	3.2	9.9	28	19
chlorpyrifos			1 NJ	3.9 J	3.8 J	28	2.4 J	5.1 J	3.8 J	3.8 J	20	18
DCPA (Dacthal)	2.7 NJ		1.7 J	4.8	5.0 J	150	2.9 J	8.2	3.6	8.3	32	26
endosulfan I						4.0 NJ						
endosulfan sulfate						11						
heptachlor epoxide						1.2 NJ						
hexachlorobenzene			0.4 NJ	0.7 J	1.1 J	6.1	0.6 J	1.5	0.6 J	1.3 J	5.7	3.3
toxaphene											120 NJ	140 NJ
trifluralin			0.5 NJ	1.2 J	1.4 J	12 J		1.5 J		1.3 J	9.3 J	7.1 J

[1] - Values are means of duplicate analyses

A blank indicates that the target analyte was not detected.

J - The analyte was positively identified. The associated numerical value in an estimate.

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

Table 2 (cont.). Pesticides and PCBs Detected in 1995 WSPMP Fish Tissue Samples (µg/kg (ppb) wet weight)

	Clear Creek	Cowlitz River					Yakima River					Cowiche Creek
	Cutthroat	Cutthroat	Mountain Whitefish		Largescale Sucker		SM Bass	Carp	Largescale Sucker			RB Trout
Tissue Type	Fillet	Fillet	Fillet		Whole		Fillet	Fillet[1]	Whole			Fillet
Size Group or Replicate			small	large	Rep-1	Rep-2			Rep-1[1]	Rep-2	Rep-3	
Percent Total Lipid	1.63	3.00	5.99	5.78	2.53	2.75	0.40	0.82	3.79	2.48	2.47	2.02
2,4'-DDE							3.0 J	5.1	20	15	11	
4,4'-DDE	13	43	13	10	73	59	180	845	3150	3000	1900	740
2,4'-DDD							1.9 J	6.4	32	26	21	1.4 J
4,4'-DDD		5.7			10	7.6	8.2	46	200	150	140	20
2,4'-DDT							2.8 J	4.7	42	36	24	7.9
4,4'-DDT		3.8			7.6	4.5	17	10.2	285	250	180	53
total DDT	13	53	13	10	91	71	213	917	3728	3477	2276	822
DDMU								17	57	51	42	15
cis-chlordane								5.0	9.1 J	7.6 J	9.4 J	0.94 NJ
trans-chlordane								1.0 J	2.9 J	2.2 J	1.5 NJ	
oxychlordane								0.35 J	2.0 J	2.1 J	2.0 J	0.75 NJ
cis-nonachlor								2.7 J	4.3 J	4.0 J	5.0 J	
trans-nonachlor	2.5 J	2.3 J	1.9 NJ			2.4 NJ		6.1	18	14	12	3.1 J
total chlordane	2.5	2.3	1.9			2.4		15.0	36	30	30	4.8
dieldrin							4.7	8.8	44	38	35	1.5 J
heptachlor epoxide								0.61 NJ	1.0 NJ	0.84 NJ	0.71 NJ	
hexachlorobenzene	0.72 J	1.4 J	1.1 J	1.1 J	1.1 J	1.2 J		0.49 J	1.7	1.5	1.4 J	0.79 J
dicofol (Kelthane)									51 J	51 J	55 J	
pentachloroanisole	0.72 NJ					0.6 NJ			1.0 J	1.0 J	1.0 J	0.83 NJ
toxaphene									240 J	230 J	200 J	
trifluralin							1.4 J	3.6 J	14 J	11 J	9.8 J	
PCB-1254	22 J	64	29 J	30 J	37 J	66		29 J	89	77 J	77 J	71
PCB-1260	24 J	20 J	18 J	30 J	47	42	25 J	106	225	220	150	25 J
total PCBs	46	84	47	60	84	108	25	135	314	297	227	96

[1] - Values are means of duplicate analyses

A blank indicates that the target analyte was not detected.

J - The analyte was positively identified. The associated numerical value in an estimate.

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

Washington State has adopted $1x10^{-6}$ as its risk level under the State Water Quality Standards (173-201A-040 WAC) and the Model Toxics Control Act (173-340-730 WAC).

Exposure assumptions used to calculate screening values include a body weight of 70 kg and a fish tissue consumption rate of 6.5 grams per day. These values represent the mean body weight for all adults and the average consumption rate for the general U.S. population (USEPA, 1995).

Screening values for non-carcinogens were calculated using a reference dose that is derived from No Observed Adverse Effects Levels (NOAELs) or Lowest Observed Adverse Effects Levels (LOAELS).

Pesticides detected in 1995 WSPMP fish tissue samples are compared to screening values in Table 3. Screening values calculated with a risk level of $1x10^{-6}$ were exceeded for one or more compounds at seven of the eight sites. Only fish from Canal Lake did not contain contaminants exceeding screening values. Total DDT, dieldrin, and PCBs were above screening values in samples from six, five, and four of the sites, respectively. Total chlordane was also above the screening value in carp fillets from the Yakima River.

Water Quality Limited List

When calculated with a risk level of $1x10^{-6}$, screening levels for the carcinogenic compounds listed in Table 3 have the same numerical value as National Toxics Rule (NTR) criteria (40 CFR part 131) that are used to assess sites for possible addition to the water quality limited list (section 303(d) of the federal Clean Water Act). The 303(d) list contains state waterbodies that do not meet water quality standards, and is used to help set priorities for addressing water pollution from a variety of sources. WSPMP sites will be added to the list if there is one or more NTR criterion exceeded for a five fish composite of edible tissue (Washington State Water Quality Policy 1-11, 1993).

Fillet samples from Redrock and Royal Lakes, Scooteney Reservoir, Clear and Cowiche Creeks, and the Cowlitz and Yakima Rivers contained one or more compounds in concentrations above NTR criteria. All of these sites qualify for addition to the water quality limited list.

Wildlife Criteria

There are no Washington State or national pesticide or PCB criteria that have been adopted for protection of wildlife. The WSPMP whole fish results are compared to criteria proposed by the state of New York for protection of fish-eating wildlife (Newell *et al.*, 1987) in Table 4. For more information regarding wildlife criteria, see the discussion in the "Ecological Assessment" section starting on page 19.

Total DDT and aldrin/dieldrin levels exceeded criteria proposed by Newell *et al.* (1987) at three of the four sites where whole fish were collected. Levels of total DDT in Royal Lake carp and Yakima River suckers were an order of magnitude higher than proposed criteria. Yakima River suckers also contained high concentrations of total PCBs.

Table 3. Comparison of Pesticides Detected in 1995 WSPMP Fish Fillet Samples to Human Health Screening Values (µg/kg (ppb) - wet weight)

Sample Site	Canal Lake		Redrock Lake		Royal Lake	Scooteney Reservoir				Screening
Fish Species	LM Bass	Perch	Largemouth Bass		SM Bass[1]	Smallmouth Bass		Largemouth Bass		Values
Size Group			Small	Large[1]		Small	Large	Small	Large[1]	$1x10^{-6}$ *
Carcinogens										
total DDT	5.2	3.4	**66**	**154**	**75**	23	**160**	29	**74**	32
total chlordane				0.7			2.8		0.7	8.3
dieldrin			**4.1**	**8.6**	**8.2**	**2.9**	**13**	**3.2**	**9.9**	0.7
hexachlorobenzene			0.4	0.7	1.1	0.6	1.5	0.6	1.3	6.7
Non-Carcinogens										
chlorpyrifos			1	3.9	3.8	2.4	5.1	3.8	3.8	32,300
DCPA (Dacthal)	2.7		1.7	4.8	5.0	2.9	8.2	3.6	8.3	5,400

[1] - Values are the means of duplicate analyses

* - $1x10^{-6}$ is the risk level adopted by Washington State

Shaded values exceed screening values calculated using a risk level of $1x10^{-6}$

A blank indicates that the target analyte was not detected

Table 3 (cont.). Comparison of Pesticides Detected in 1995 WSPMP Fish Fillet Samples to Human Health Screening Values (µg/kg (ppb) - wet weight)

Sample Site	Clear Creek	Cowlitz River			Yakima River		Cowiche Creek	Screening
Fish Species	Cutthroat	Cutthroat	Mountain Whitefish		SM Bass	Carp	RB Trout	Values
Size Group			Small	Large				$1x10^{-6}$ *
Carcinogens								
total DDT	13	**53**	13	10	**213**	**917**	**822**	32
total chlordane	2.5	2.3	1.9			**15.0**	4.8	8.3
dieldrin					**4.7**	**8.8**	**1.5**	0.7
heptachlor epoxide						0.61		1.2
hexachlorobenzene	0.72	1.4	1.1	1.1		0.49	0.79	6.7
total PCBs	**46**	**84**	**47**	**60**	**25**	**135**	**96**	1.4

[1] - Values are the means of duplicate analyses

* - $1x10^{-6}$ is the risk level adopted by Washington State

Shaded values exceed screening values calculated using a risk level of $1x10^{-6}$

A blank indicates that the target analyte was not detected

Table 4. Comparison of Pesticides Detected in 1995 WSPMP Whole Fish Samples to Recommended Wildlife Criteria (µg/kg (ppb) - wet weight)

Sample Site	Royal Lake	Scooteney Reservoir		Cowlitz River		Yakima River			Criteria Newell *et al*., 1987	
Fish Species	Carp	Carp		Largescale Sucker		Largescale Sucker			as Non-	as
Replicate		Rep-1	Rep-2	Rep-1	Rep-2	Rep-1[1]	Rep-2	Rep-3	Carcinogens	Carcinogens
DDE	**2008**	**372**	**251**	73	59	**3170**	**3015**	**1911**	200	270
DDD	137	51	31	10	8	**232**	176	161	200	
DDT	30	6	5	8	5	**327**	**286**	**204**	200	
total DDT	**2175**	**428**	**288**	91	71	**3728**	**3477**	**2276**	200	
total chlordane	19	12	7		2	36	30	30	500	370
aldrin + dieldrin	**42**	**29**	20			**44**	**38**	**35**	120	22
heptachlor epoxide	1					1	1	1	200	210
hexachlorobenzene	6	6	3	1	1	2	2	1	330	200
pentachloroanisole					1	1	1	1	2000[2]	
total PCBs				84	**108**	**314**	**297**	**227**	100	110

[1] - Values are the means of duplicate analyses

[2] - Criterion is for pentachlorophenol, which is the parent compound for pentachloroanisole

Shaded values exceed recommended criteria

A blank indicates that the target analyte was not detected

Site Evaluations

Canal Lake

Canal Lake is one of the largest waterbodies in the Seep Lakes Wildlife Area between Potholes Reservoir and Othello. This is a true seep lake that does not have a supply or discharge, and the water level is maintained primarily by seepage from Potholes Reservoir and the Potholes East Canal. When water levels are high enough, Canal Lake is continuous with Windmill Lake. For the purpose of this study these lakes were both sampled as a single waterbody.

Few fish were encountered in either lake while electroshocking, but enough perch and largemouth bass were collected to make up two composite samples. Each species was collected from both lakes, but more were found in Windmill Lake. Gill nets were set overnight near the bottom of Canal Lake at the north end in a deep pool in an effort to collect rainbow trout, but none were found.

Fish from these lakes had the lowest number of pesticides, and the two that were found – 4,4'-DDE and DCPA (Dacthal) – were in low concentrations. Neither of these pesticides exceeded screening values or wildlife criteria.

Redrock Lake

Redrock Lake is long and narrow, and is formed by a dam across the west end of Redrock Coulee. This lake is also called Natural Corral, and is located about three miles southeast of Royal City. Irrigation return water from various crops along the Royal Slope flows into the east end of the lake by way of a spectacular waterfall, and discharges from the west end into Lower Crab Creek.

Much of the lake is shallow and inundated with weeds, making access by boat difficult. The south side of the lake is apparently deeper, and has areas clear of weeds. Carp were plentiful, but none were collected. The only other species encountered was largemouth bass, which was also fairly plentiful. Two or three hours of electroshocking along the south shore produced enough fish for two composites.

Nine pesticides were found in the composite of small specimens, and 13 in the large specimens. Concentrations of detected compounds were consistently higher in the composite of larger specimens. Total DDT and dieldrin in both samples exceeded human health screening values, and the level of dieldrin in the larger fish was an order of magnitude higher than the screening value. No whole fish samples were analyzed for this site.

Royal Lake

Royal Lake is located about 10 miles northwest of Othello on the east end of the Royal Slope, and receives the majority of irrigation return water from this area by way of the Crab Creek Lateral. Water from this irrigation return flows into the northeast end of the lake, and then discharges from the west end into Lower Crab Creek. Royal Lake is in the Columbia National Wildlife Refuge, and collection of fish required a special use permit (No. 74236) from the U.S. Fish and Wildlife Service. Water was collected from the Crab Creek Lateral for the 1995 WSPMP, and results are summarized in the surface water sampling report (Davis *et al.*, 1997).

Royal Lake is similar to Redrock Lake in several ways. The source of water is essentially the same, the lake is shallow and inundated with weeds except for a small strip down the middle, and the fish have similar pesticide contaminants. In addition, carp and bass were the only species of fish encountered. Smallmouth bass were collected instead of largemouth, but the fish had similar levels of pesticides. One composite of carp was analyzed as whole-fish tissue.

Total DDT and dieldrin in bass fillets were above screening values, and the dieldrin was an order of magnitude higher. These same pesticides were very high in the whole carp sample, and both exceeded wildlife criteria.

Scooteney Reservoir

Scooteney Reservoir is about 12 miles southeast of Othello, and receives water from the Potholes Canal and irrigation return water from the Paradise Flats area east of Othello. This is a relatively large and deep waterbody compared to lakes in this area, and much of it is free of weeds. These attributes make it attractive to the general public for recreation, and apparently make it good habitat for bass, which were plentiful and large. Easy access, a nice park on the east shore, and excellent bass fishing contribute to making this site a popular recreation destination.

Both smallmouth and largemouth bass were present in the reservoir, and enough individuals of each species were collected to make up two fillet composites: one of small and one of large individuals. Two replicate samples of whole carp were also collected. The bass were collected by electroshocking along the west shore, and the carp were collected at the north end where the water is shallow and mostly filled with reeds.

The types and concentrations of pesticides detected in the bass fillets were similar to those found in bass from Redrock and Royal Lakes. Total DDT levels in the composites of small individuals were slightly below the screening value, but concentrations in the large individuals were higher than the screening value. The levels of dieldrin in all four samples exceeded the screening value. The concentrations of total DDT in whole carp from Scooteney Reservoir were substantially lower than in the carp from Royal Lake, but levels of other detected pesticides were similar. Aldrin + dieldrin in one replicate and total DDT levels in both replicates were above wildlife criteria.

Although the concentrations were low, the detection of aldrin in carp from Scooteney Reservoir is significant because it is unusual to find aldrin. Most uses for aldrin have been canceled, and it readily breaks down to form dieldrin, so detection of aldrin may indicate recent use.

Summary of Data from the Mid-Columbia Area

From the data presented here, it appears that lakes and reservoirs in the Mid-Columbia area that receive water directly from irrigation returns have fish with high levels of total DDT and dieldrin. Several other pesticides were also consistently detected at concentrations below human health screening values or wildlife criteria, but the combination of these chemicals, in addition to DDT and its breakdown products and dieldrin, may be more toxic than the compounds considered individually. Seep lakes, such as Canal Lake, that do not receive water directly from irrigation returns appear to have fish with fewer pesticides, and chemicals that are found in the fish are at much lower concentrations.

Fish and other biota from nine lakes, reservoirs, and wasteways in the CBIP area were sampled and analyzed for pesticides as a part of a reconnaissance investigation in 1991-92 by the USGS (Embrey and Block, 1995). They found elevated levels of total DDT and dieldrin in most of their samples. Scooteney Reservoir was the only site common to this study; total DDT, dieldrin, and other pesticide concentrations in whole carp were consistently higher in the USGS samples.

Embrey and Block (1995) compared total DDT concentrations in carp and perch to historical data collected over the past 15 years from the CBIP area, and concluded that there has been little change. In fact, some of the highest levels of total DDT were in carp collected in 1992.

Additional data were obtained by the USGS as a part of their Columbia Basin National Water Quality Assessment (NAWQA) Program (Munn and Gruber, 1997). They collected fish and sediment from seven waterbodies associated with the CBIP. The objective of their study was to assess the relationship between land use and organochlorine compounds in fish and sediment. They found that there was a significant linear relationship between the percentage of gravity (furrow) irrigation and total DDT concentrations in fish and sediments. Gravity irrigation results in more erosion, which transports soil particles contaminated with DDT into the irrigation returns. A site fed by ground water (seeps), and another by sprinkler irrigation, had the lowest levels of chlorinated pesticides.

The only lake sampled by the USGS was Royal Lake; other sites were creeks, wasteways, and irrigation returns. Carp were collected from all of the sites except one, and all samples were analyzed as whole fish. These data were presented in a USGS fact sheet to assess potential impacts to fish and wildlife (Gruber and Munn, 1996). The type and concentrations of chemicals found in Royal Lake carp were very similar to those detected in the WSPMP sample. The USGS concluded that concentrations of some detected compounds may pose a threat to fish and wildlife.

Most of the lakes and reservoirs in the Mid-Columbia Basin are easily accessable to fishermen. Boat ramps are provided on waterbodies where boating is appropriate, and others are open to anglers on foot.

Many of the lakes in this area are stocked yearly with rainbow and brown trout for recreational fishermen (Foster, 1995). Apparently the trout either die or are quickly removed by fishermen because none were found in any of the lakes sampled. None of these lakes offer suitable habitat for the trout to reproduce; the water gets too warm. Planted trout are probably not present long enough to accumulate contaminants to levels that may adversely impact human health or wildlife, although a few individuals may survive from year to year and accumulate pesticide concentrations similar to resident bass and carp.

Bass appear to be the most common resident sportfish in these lakes, and are probably favored by fishermen. Total DDT concentrations in bass were moderately high; five out of eight samples exceeded the human health screening value. Dieldrin levels were consistently high in bass from lakes receiving irrigation return water; all samples were above the screening value, and some were an order of magnitude higher. In the three pairs of samples with composites of small and large individuals, most pesticides were substantially lower in the sample of smaller fish. This indicates that regular consumers of bass from this area can reduce their risk by avoiding large fish. Pesticide concentrations were higher in carp and they are abundant throughout the CBIP, but few people eat them.

Clear Creek

Clear Creek drains the Clear Creek Valley north of Silverdale in Kitsap County. Land use in this valley is almost exclusively suburban/rural residential. There are a few hobby farms with pasture for a few horses or cattle, but no large farms or ranches. Historically, this valley probably supported some agriculture. Through the town of Silverdale, a wide riparian zone has been left along both sides of the creek, and stormwater runoff from surface streets is collected in a drainage system that discharges directly into Dyes Inlet to protect the creek. Water was collected near the mouth of Clear Creek for the 1995 WSPMP, and results are presented in the surface water sampling report (Davis *et al.*, 1997).

Few fish were encountered in Clear Creek. A single composite of cutthroat trout was collected after about two hours of electroshocking. A few sculpin were also seen, but a sample was not collected. The cutthroat were collected from a reach less than a mile long starting at the mouth, working upstream. Access was difficult due to heavy vegetation along both banks.

Low levels of four pesticides and two PCBs were detected in cutthroat fillets. The pesticides were below screening values, but total PCBs exceeded the screening value. No whole fish were analyzed for comparison to wildlife criteria.

Cowlitz River

The headwaters of the Cowlitz River are in the Goat Rocks Wilderness area near White Pass. Highway 12 follows the river from the west side of the pass to Riffe and Mayfield Lakes. From the lakes, the Cowlitz River flows southwest through the Cowlitz Prairie, and then south to the Columbia River at Longview. Many of the residents along the river are employed by the logging industry, especially in towns above the lakes like Morton, Randle, and Packwood. Fertile land along the river valley has attracted some agriculture, but the dominant crop grown in this area is Christmas trees.

Fish were collected upstream from the confluence with the Toutle River in a reach just below the point where the Cowlitz River passes under Interstate-5 near Vader. Fish were plentiful and one composite of cutthroat trout, two of mountain whitefish, and two of largescale suckers were easily collected by electroshocking. The cutthroat and whitefish were analyzed as fillets, and the suckers were analyzed whole.

All of the samples contained low or moderate levels of total DDT, hexachlorobenzene, and total PCBs. Three samples also had low concentrations of trans-nonachlor, and pentachloroanisole was tentatively identified in one sucker sample. The whitefish had the lowest levels of pesticides and PCBs, which is unusual because their lipid content was double the other fish. Total DDT in cutthroat fillets and total PCBs in cutthroat and whitefish exceeded the human health screening values. PCBs in one of the largescale sucker samples were above the wildlife criterion.

Yakima River

The Yakima River flows southeast from the east side of the Cascade Mountains near Snoqualmie Pass to Ellensburg where it flows south to Yakima. Just south of Yakima at Union Gap the direction of the river changes back to the southeast, and stays on this course most of the way through the Yakima Valley to the Columbia River at Richland. Along its course, much of the water is diverted for crop irrigation throughout the Yakima Valley (Joy and Patterson, 1997). Irrigation drainage ditches return the water to the river, but some carry heavy loads of pesticides.

Samples were collected from the reach just upstream of the bridge on Euclid Road in the Sunnyside Wildlife Area. Throughout this reach, the river widens out and slows down, allowing suspended sediments to settle out. Of the tissue samples collected from the river in 1989-90 for the USGS Yakima River NAWQA, fish from their site near the Euclid Road bridge contained the highest concentrations of pesticides (Rinella *et al.*, 1992). The higher levels of pesticides in fish from this site are probably the result of close contact with contaminated sediment.

Smallmouth bass, carp, and largescale suckers were the only species encountered in this part of the river. Trout and whitefish that were collected by the USGS at other sites are restricted to riffles where the bottom is rocky. Single composites of bass and carp were analyzed as fillets, and three replicate composites of suckers were analyzed whole.

All of the samples had high concentrations of pesticides. Levels of total DDT, dieldrin, and total PCBs in the bass and carp fillet samples, as well as total chlordane in the carp, exceeded screening values. Concentrations of total DDT, dieldrin, and total PCBs in whole suckers were higher than wildlife criteria. Levels of total DDT are an order of magnitude higher than the screening value or wildlife criterion.

Total DDT data from whole fish and fillet samples collected from the lower Yakima River since 1970, including data from this study, were summarized by Joy and Patterson (1997). Concentrations were variable depending on the site and species sampled, ranging from 18 to 4940 μg/kg. The average level of total DDT in whole largescale suckers sampled by the USGS from the river near Euclid Road bridge in 1989-90 was 3340 μg/kg (Rinella *et al.*, 1992), which is essentially the same as the average of 3160 μg/kg for the three replicate sucker samples collected for this study in 1995. Similar levels have been found in fish at other sites from Granger to Kiona since 1970, indicating that concentrations have stayed the same for the last 25 years.

In 1993, the Washington State Department of Health promulgated a recommendation to eat fewer bottom fish taken from the lower Yakima River. That recommendation was based on the high levels of total DDT in these fish. The data presented here suggest that this recommendation should remain in effect.

Cowiche Creek

Cowiche Creek drains a large area northwest of Yakima, and flows into the Naches River about three miles upstream from its confluence with the Yakima River. The north fork of Cowiche Creek runs through an agricultural area consisting primarily of orchards, and land along the south fork is sparsely populated until it approaches the confluence with the north fork.

Rainbow trout were collected near the creek mouth at the bridge on Powerhouse Road; no other species were seen. A single composite was processed and analyzed as fillets.

This sample contained 14 different chemicals, and the concentration of total DDT was 822 µg/kg. Both are high for rainbow trout fillets. Levels of total DDT, dieldrin, and total PCBs exceeded human health screening values, and total DDT was over an order of magnitude higher than the screening value. The concentration of total DDT in this sample is the highest level ever recorded for rainbow trout in Washington State.

Ecological Assessment

A detailed assessment of the effects of detected pesticides on fish and fish-eating wildlife was presented in the 1993 and 1994 WSPMP fish tissue sampling reports (Davis *et al.*, 1995; Davis and Serdar, 1996). Most of that information is applicable to this report, but will not be repeated here. Instead, specific information from the 1993 and 1994 reports will be used to evaluate possible toxic effects on fish and wildlife from compounds detected in 1995.

The wildlife criteria previously presented in Table 4 are fish flesh criteria developed by Newell *et al.* (1987) for contaminants found in Niagara River fish to protect piscivorous (fish-eating) wildlife. The methodology used by Newell *et al.* to calculate criteria has been selected to develop Canadian tissue residue guidelines for protecting wildlife (Environment Canada, 1994-draft).

DDE, DDD, DDT, aldrin/dieldrin, and total PCBs were detected in 1995 fish tissue samples at concentrations exceeding wildlife criteria. All of these were found in largescale suckers from the Yakima River. Detected pesticides that were not addressed by Newell *et al.* (1987) include chlorpyrifos, DCPA, endosulfan, dicofol, toxaphene, and trifluralin.

Piscivorous wildlife in Washington that may be sensitive to high concentrations of total DDT includes white pelicans, osprey, and bald eagles. White pelicans are frequently seen in many parts of eastern Washington and are known to nest within the McNary National Wildlife Refuge (Linehan, 1995). Osprey are large, exclusively piscivorous birds that are commonly seen nesting along rivers and lakes throughout Washington. Bald eagle diets consist of up to 90% fish, but they also eat other birds and small mammals, including carrion.

Concentrations of total DDT over 300 µg/Kg in fish eaten by osprey can accumulate in the bird's tissues resulting in total DDT levels in their eggs over 3000 µg/Kg and significant eggshell thinning (Steidl *et al.*, 1991). Similar concentrations of DDE in fish consumed by bald eagles have been linked to eggshell thinning and low reproductive success (Wiemeyer *et al.*, 1972, 1984).

All of the whole-fish samples collected east of the Cascade Mountains in 1995 contained total DDT concentrations above the wildlife criterion. DDE and total DDT concentrations in whole carp and suckers from Royal Lake and the Yakima River were substantially higher than the criteria, and have the potential to cause adverse effects in sensitive species. White pelicans, osprey, and bald eagles are likely to experience significant eggshell thinning and reduced hatching success if a high proportion of their diet consists of fish from these two waterbodies.

Fish collected from the Wenatchee River for the 1993 WSPMP (Davis *et al.*, 1995), and from Lake Chelan, and the Entiat and Okanogan Rivers for the 1994 WSPMP (Davis and Serdar, 1996) had elevated levels of total DDT. Data from this report and recent USGS reports (Embrey and Brock, 1995; Munn and Gruber, 1997) show that fish from the Yakima River, Cowiche Creek, and many waterbodies in the CBIP area also have elevated levels of DDT. These data suggest that a high proportion of the fish consumed by piscivorous birds inhabiting the Columbia Basin are contaminated with high concentrations of total DDT.

Many of the fish tested had several other chemical contaminants in their tissues, and some of these compounds may cause problems similar to DDT. Dieldrin has also been linked to eggshell thinning and reproductive failure (Lehner and Egbert, 1969; Ratcliffe, 1970). PCBs can cause reproductive problems, particularly in mammals (Platonow and Karstad, 1973). Some of the pesticides detected in fish have unknown consequences for piscivores.

There is a study currently in progress to evaluate the condition of the osprey population in the Mid-Columbia Basin (Henny, 1997). The study includes analyzing eggs for contaminant concentrations and assessing reproductive success. Results from the osprey study should give a good indication of impacts from pesticide contamination in fish to piscivorous wildlife.

The discussions above generally address only one or two compounds at a time. Very little is known about the effects due to combinations of chemical contaminants, but they may very well be additive or synergistic, resulting in more environmental damage than expected. These chemicals may also cause sublethal effects, such as endocrine disruption, that can result in subtle adverse changes in the population without increased mortality (Colborn *et al.*, 1996). In addition, there may be other toxic chemicals present that were not analyzed for the WSPMP. Therefore, fish and piscivorous wildlife at the sites investigated may be experiencing problems that would not be anticipated from the information available.

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Appendices

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Appendix A. Fish Tissue Sampling Site Positions for the 1995 WSPMP

Site Name	Latitude			Longitude			State Plane	
	deg	min	sec	deg	min	sec	X	Y
Canal Lake in the Seep Lakes Wildlife Area north of Othello	46	55	49	119	10	51	2329568	585109
Redrock (Corral) Lake southeast of Royal City	46	57	45	119	17	44	2300730	596401
Royal Lake northwest of Othello	46	51	58	119	21	18	2286399	561030
Scooteney Reservoir southeast of Othello	46	40	10	119	01	53	2368671	490662
Clear Creek at Silverdale	47	39	15	122	41	08	1461398	853698
Cowlitz River northeast of Vader	46	24	50	122	53	27	1397053	403175
Yakima River upstream from Euclid Road Bridge	46	13	54	120	00	02	2126395	327987
Cowiche Creek near Hwy. 12	46	37	38	120	34	53	1979524	471788

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Appendix B. Target Pesticides List for 1995 WSPMP Tissue Analyses

Analyte	Quantitation Limit[1] (µg/kg, ppb wet)	Analyte	Quantitation Limit (µg/kg, ppb wet)
2,4'-DDD	3.6	endosulfan I	5
4,4'-DDD	3.6	endosulfan II	3.6
2,4'-DDE	3.6	endosulfan sulfate	3.6
4,4'-DDE	3.6	endrin	3.6
4,4'-DDMU	3.6	endrin aldehyde	3.6
2,4'-DDT	3.6	endrin ketone	3.6
4,4'-DDT	3.6	ethion	14
aldrin	3.6	heptachlor	3.6
alpha-BHC	3.6	heptachlor epoxide	3.6
beta-BHC	3.6	hexachlorobenzene	1.8
delta-BHC	3.6	methoxychlor	3.6
gamma-BHC (Lindane)	3.6	mirex	3.6
cis-chlordane	3.6	oxadiazon	3.6
trans-chlordane	3.6	ethyl-parathion	7.1
oxychlordane	3.6	methyl-parathion	7.1
cis-nonachlor	3.6	pentachloroanisole	1.8
trans-nonachlor	3.6	tetradifon	14
alpha-chlordene	3.6	toxaphene	110
gamma-chlordene	3.6	trifluralin	3.6
chlorpyrifos	7.1	PCB-1232	36
DCPA (Dacthal)	3.6	PCB-1242	36
diazinon	36	PCB-1248	36
dichlorobenzophenone	14	PCB-1254	36
dicofol (Kelthane)	14	PCB-1260	36
dieldrin	3.6		

[1] - Quantitation limits are approximate and are often different for each sample; these values are representative of a typical sample

Appendix B (cont.). Distribution of Compounds Eluted from Florisil Columns for Tissue Analyses

Fraction 1 (0%)	Fraction 2 (6%)	Fraction 3 (15%)	Fraction 4 (50%)
alpha BHC*	alpha BHC*	DCPA (Dacthal)	endosulfan II
aldrin	beta BHC	diazinon	endosulfan sulfate
alpha chlordene	gamma BHC	dichlorobenzophenone	endrin aldehyde
gamma chlordene	delta BHC	dieldrin	endrin ketone
2,4'-DDE	cis-chlordane	endosulfan I	
4,4'-DDE	trans-chlordane	endrin	
4,4'-DDMU*	chlorpyrifos	oxadiazon	
2,4'-DDT	2,4'-DDD	ethyl parathion	
4,4'-DDT*	4,4'-DDD	methyl parathion	
heptachlor	4,4'-DDMU*	tetradifon	
hexachlorobenzene	4,4'-DDT*		
mirex	dicofol (kelthane)		
trans-nonachlor	ethion		
PCB-1232	heptachlor epoxide		
PCB-1242	methoxychlor		
PCB-1248	cis-nonachlor		
PCB-1254	oxychlordane		
PCB-1260	pentachloroanisole		
	toxaphene		
	trifluralin		

* - Found in both 0% and 6% fractions

Appendix C. 1995 WSPMP Fish Length (Total) and Weight Data

Location and Species	Date and Sample Number	Mean Length (mm)	Length Range (mm)	Mean Weight (grams)	Weight Range (grams)	Tissue Type
<u>Canal Lake</u>	9/14					
largemouth bass	378237	248	230-260	246	207-276	fillet
yellow perch	378238	236	230-260	142	115-185	fillet
<u>Redrock Lake</u>	9/13					
largemouth bass (small)	378239	289	240-345	423	245-697	fillet
largemouth bass (large)	378240	396	354-455	1069	785-1474	fillet
<u>Royal Lake</u>	9/13					
smallmouth bass	378235	237	215-270	207	149-307	fillet
carp	378236	652	630-680	4656	3888-5277	whole
<u>Scooteney Reservoir</u>	9/14					
smallmouth bass (small)	378246	291	280-305	337	266-399	fillet
smallmouth bass (large)	378243	391	335-482	1022	544-1832	fillet
largemouth bass (small)	378245	218	215-230	154	142-170	fillet
largemouth bass (large)	378244	404	365-430	1239	886-1456	fillet
carp (Rep-1)	378241	573	518-605	2758	2215-3355	whole
carp (Rep-2)	378242	558	536-577	2691	2086-3518	whole
<u>Clear Creek</u>	9/21					
cutthroat trout	388035	232	219-251	122	105-149	fillet

Appendix C (cont.). 1995 WSPMP Fish Length (Total) and Weight Data

Location and Species	Date and Sample Number	Mean Length (mm)	Length Range (mm)	Mean Weight (grams)	Weight Range (grams)	Tissue Type
<u>Cowlitz River</u>	9/20					
cutthroat trout	388034	312	270-380	315	175-579	fillet
mountain whitefish (small)	388033	350	310-400	403	298-602	fillet
mountain whitefish (large)	388032	382	330-505	611	322-1448	fillet
largescale sucker (Rep-1)	388030	434	398-450	868	645-950	whole
largescale sucker (Rep-2)	388031	467	405-525	1036	643-1368	whole
<u>Yakima River</u>	9/12-13					
smallmouth bass	378231	333	260-375	535	204-707	fillet
carp	378230	452	410-485	1288	987-1563	fillet
largescale sucker (Rep-1)	378232	456	425-480	851	661-1036	whole
largescale sucker (Rep-2)	378233	443	395-480	807	526-1153	whole
largescale sucker (Rep-3)	378234	429	410-445	718	588-805	whole
<u>Cowiche Creek</u>	9/25					
rainbow trout	388036	316	230-404	411	118-865	fillet

<u>Common Name</u>	<u>Scientific Name</u>
largemouth bass	*Micropterus salmoides*
smallmouth bass	*Micropterus dolomieui*
yellow perch	*Perca flavescens*
carp	*Cyprinus carpio*
cutthroat trout	*Salmo clarki*
rainbow trout	*Oncorhynchus mykiss*
mountain whitefish	*Prosopium williamsoni*
largescale sucker	*Catostomus macrocheilus*

Appendix D. Characteristics and Interrelationships of Pesticides/PCBs Detected in 1995

The tissue target analytes for the WSPMP were selected because they tend to accumulate in animal tissues. These compounds are lipophilic, i.e. they are chemically attracted to lipids (fats). Once accumulated, these chemicals are either metabolized or excreted by the animal, but the rate can be very different for each compound depending on a variety of factors.

Chlorinated Hydrocarbon Insecticides

All chlorinated hydrocarbon insecticides have a cyclic structure and their molecular weight ranges from about 285 to 545 (Smith, 1991). Most can be divided into five groups: DDT and its analogs, cyclodienes, benzene hexachloride (BHC), toxaphene, and mirex. Thirty-three of the 43 target pesticides are in one of these five groups. These compounds are grouped by structural similarity, but even small differences in structure can result in dramatic differences in toxicity and persistence.

Dicofol (kelthane), endosulfan, and methoxychlor are currently registered for use in Washington. Dicofol and endosulfan are used primarily as acaricides to control aphids and mites on a variety crops and ornamental plants. Methoxychlor is used on a variety of crops, and is used to control flies and mosquitoes in areas of human habitation (USEPA, 1992).

DDD, DDE, and DDMU are metabolites of DDT. DDD was also marketed as a pesticide (Rhothane). Dicofol and methoxychlor can occur as contaminants in commercial formulations of DDT. Both are much less toxic and persistent than DDT (Smith, 1991). Dicofol products are often contaminated with DDT and/or PCBs. Dichlorobenzophenone is a metabolite in animals exposed to DDT (Matsumura, 1985).

Cyclodiene pesticides include chlordane, heptachlor, aldrin, dieldrin, endrin, and endosulfan. The structure of this group is characterized by an endomethylene bridge (FCH, 1991). Chlordane has two main isomers, cis- and trans-. Oxychlordane is the major breakdown product of chlordane. The two isomers of nonachlor are found as contaminants of technical chlordane. Heptachlor is more likely to be found as its major breakdown product, heptachlor epoxide. Dieldrin is the metabolite of aldrin, but was also marketed as an insecticide. Endrin is the stereoisomer of dieldrin. Dieldrin is less toxic than endrin, but much more persistent (Smith, 1991). Endosulfan is a mixture of two stereoisomers, I and II. Endosulfan breaks down fairly quickly to endosulfan sulfate, which is more persistent than the parent chemical (Seyler *et al.*, 1994).

Limited use of chlordane is allowed with the requirement that all applications must be done by a licensed applicator (USEPA, 1992). All uses of dieldrin have been voluntarily cancelled by industry. Registration of all endrin products was cancelled in 1984. All uses of heptachlor, except ground insertion for termite control and dipping of roots or tops of nonfood plants, were banned by 1983.

Appendix D. Characteristics and Interrelationships of Pesticides/PCBs Detected in 1995

Toxaphene is a complex mixture of as many as 177 compounds; however, only three of the compounds account for most of the toxicity (Smith, 1991). Toxaphene has been classified as a probable human carcinogen, but it is easily metabolized and is not stored to any great extent in tissues (USEPA, 1995). Toxaphene's registration was canceled for most uses in 1982.

Organophosphorous Insecticides

Organophosphorous (OP) insecticides are acetylcholinesterase (an enzyme that regulates nerve transmissions) inhibitors, and are generally more toxic than chlorinated insecticides, but usually breakdown much more quickly. Animals that do not receive a fatal dose can usually metabolize OP pesticides and recover completely. Some OPs have the potential to accumulate in tissue, but not to the extent of most chlorinated insecticides.

Five OP pesticides, chlorpyrifos, diazinon, ethion, ethyl parathion, and methyl parathion, are included on the WSPMP target analyte list. All of these compounds are presently registered for use in Washington. Chlorpyrifos (Dursban, Lorsban) is the only OP insecticide detected in 1995 WSPMP tissue samples. Chlorpyrifos was also detected in fish tissue collected in 1992 (Davis and Johnson, 1994). Ethion was detected in tissue samples analyzed for the WSPMP in 1993 (Davis *et al.*, 1995) and 1994 (Davis and Serdar, 1996).

Miscellaneous Pesticides

DCPA (Dacthal) is a pre-emergence herbicide that has low toxicity to most animals, but is relatively persistent and accumulates in some tissues (Rasmussen and Blethrow, 1991). DCPA is currently used throughout Washington and has been detected numerous times in tissue, surface water, and ground water samples (Davis and Serdar, 1996; Davis, 1996; Davis and Johnson, 1994; Larson, 1993; Larson and Erickson, 1993).

Hexachlorobenzene (HCB) is a fungicide that was widely used as a seed protectant until it was banned in 1985 (USEPA, 1995). HCB has low toxicity, but can bioaccumulate to high concentrations and is a known animal carcinogen.

Pentachlorophenol (PCP) has been used on a restricted basis since 1986, primarily as a wood preservative with insecticidal, fungicidal, herbicidal, molluscicidal, and anti-microbial actions (Newell *et al.*, 1987). PCP is extremely toxic to most animals and plants, but is usually not found at concentrations that are lethal (Seyler *et al.*, 1994). PCP is quickly metabolized by animals, but the primary metabolite, pentachloroanisole, is persistent and has a high potential for bioaccumulation (USEPA, 1992). Information on the toxicity of pentachloroanisole is lacking (Newell *et al.*, 1987). Pentachloroanisole was identified in several tissue samples from the 1992 and 1995 WSPMP, but was not found in 1993 or 1994.

Appendix D. Characteristics and Interrelationships of Pesticides/PCBs Detected in 1995

Trifluralin is a preemergence herbicide used to control many annual grasses and broadleaf weeds in a large variety of crops. This compound was not included as a target analyte for tissue until 1995 when it was detected in over half of the samples. Trifluralin is toxic to fish and other aquatic organisms, but it strongly adsorbs to soils which limits its availability to aquatic animals (Seyler *et al.*, 1994). The highest tissue concentrations were found in fish from waterbodies receiving irrigation returns carrying heavy sediment loads. The toxicity of accumulated trifluralin is unknown. The LC_{50} in water for rainbow trout is 41 µg/L (Johnson and Finley, 1980).

Polychlorinated Biphenyls

Polychlorinated biphenyls (PCBs) are not pesticides, but are typically analyzed using the same method as chlorinated pesticides and so are usually included in studies monitoring for pesticides. In addition, PCBs are similar to chlorinated pesticides in their chemical and physical properties, and toxicity (USEPA, 1995). There are 209 different PCB compounds (congeners). In the United States, mixtures of these congeners were formulated for commercial use under the trade name Aroclor. Different Aroclors were named based on their chlorine content; for example, Aroclor 1260 (PCB-1260) has an average chlorine content of 60%. The first two digits indicate the number of carbon atoms in the parent molecule (except PCB-1016, which was named by the manufacturer).

PCBs were used in a number of applications, primarily as thermal stabilizers in lubricants, hydraulic fluids, and insulating fluids for transformers and capacitors. Although all uses of PCBs in the United States were banned in 1979, there are still many transformers and capacitors in use that contain PCBs.

PCBs are extremely persistent and readily bioaccumulate in fatty tissues. PCBs produce a variety of adverse biological effects in animals including hepatotoxicity, developmental toxicity, immunotoxicity, neurotoxicity, and carcinogenicity (USEPA, 1995). The toxicity of PCBs to humans is poorly understood and is still being investigated.

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Appendix E. Sample Collection and Processing Procedures

Fish Collection

Electroshocking equipment was used to collect fish. When possible, each fish for a composite was taken from a different location within the specified sampling area (space-bulking). This should result in a sample that is more representative of a water body than a sample of fish all taken from the same location. The position of each sampling site was recorded using a global positioning system (GPS). When possible, whole fish samples were a mixture of males and females. As fish were collected, they were placed in clean stainless steel buckets until they could be processed.

Field Processing of Fish Tissue Samples

After collection, all fish samples were rinsed with native water, measured for total length, and weighed. A portable battery powered scale was used to weigh the fish to the nearest gram. Scale samples were taken from each sport fish for age determinations, but these samples have not been analyzed.

Fish samples were preserved on ice and transported to the laboratory whole. Each fish was wrapped in aluminum foil with the dull side in contact with the specimen. All specimens forming a composite were placed in a separate polyethylene bag. At the laboratory, all fish were kept frozen until processed.

Laboratory Processing of Fish Tissue Samples

Fish collected for whole body analysis were cut into chunks small enough to be put through a Hobart® commercial meat grinder. The sex of each fish was recorded during processing. Subsamples of the homogenized whole fish samples were transferred to appropriate containers and refrozen. Fillet samples were homogenized with a grinding attachment for a Kitchen-Aid® food mixer. All fish were processed frozen.

Fish collected for fillets (skin on) were processed in the laboratory using the following procedure:

- Clean and remove scales (skin of catfish and carp was removed).
- Remove entire fillet on left side of fish from the gill arch to the caudal peduncle.
- Place fillet in an appropriate container.
- Open body cavity to determine sex.
- Repeat process for all specimens to be included in the composite.
- Homogenize fillets and refreeze to -20° C.

Appendix E (cont.). Sample Collection and Processing Procedures

Decontamination Procedures

Fish tissue samples destined for pesticide analysis were contained in glass jars with teflon-lined lids. These containers were precleaned by Eagle-Picher Environmental Services Miami, Oklahoma using the following process:

- washed in laboratory grade detergent,
- rinsed three times with distilled water,
- rinsed with 1:1 nitric acid,
- rinsed three times with organic-free water,
- oven-dried for one hour,
- rinsed with hexane,
- and oven-dried again for one hour.

Sampling equipment, homogenization equipment and utensils, and filleting instruments were stainless steel or glass and were precleaned using the following procedure:

- rinsed with tap water,
- rinsed with deionized water,
- washed with laboratory grade detergent (Alconox®),
- rinsed with pesticide grade acetone, and
- allowed to air dry.

Appendix F-1. Analytical Methods - QA/QC - Data Review

Analytical Methods

Fish tissue samples were analyzed by Ecology's Manchester Environmental Laboratory (extraction SOP 7300722, version1.0 and 730073, version 1.0; cleanup SOP 730018, version 1.0) incorporating the acetonitrile back-extraction clean-up portion of a method developed by the California Department of Fish and Game, Water Pollution Control Laboratory. A detailed explanation of the analytical procedure can be found in Rasmussen and Blethrow (1991). Briefly, the tissue is extracted with acetonitrile and the extract is partitioned with petroleum ether and water. The petroleum ether extract is then eluted through a Florisil column in four fractions; fraction one is eluted with petroleum ether, fraction two is eluted with 6% ethyl ether, fraction three is eluted with 15% ethyl ether, and the fourth fraction is eluted with 50% ethyl ether.

Each fraction was analyzed separately with a gas chromatograph using an electron capture detector (USEPA Method 8080). A five meter J&W DB5 fused silica pre-column was connected to the injector, and the effluent from the pre-column was split into 60 meter J&W DB5 and 60 meter J&W DB17 columns. Pesticide detections in the sample extracts were confirmed with a gas chromatograph/mass spectrometer (GC/MS) using an ion trap detector.

Percent lipid in tissue samples is determined using the method described in the USEPA document "Manual of Analytical Methods for the Analyses of Pesticides in Humans and Environmental Samples", EPA-600 18-80-038, June 1980 (Manchester Laboratory SOP 730009, version 1.0).

Quality Assurance/Quality Control

Field Quality Control Procedures

Field replicate samples were taken to estimate overall precision and to assess environmental variability. Replicate largescale sucker samples were collected from the Cowlitz and Yakima Rivers, and replicate carp samples were collected from Scooteney Reservoir.

Duplicate tissue samples (splits) were submitted to evaluate analytical precision. Duplicate samples were analyzed from Redrock and Royal Lakes, Scooteney Reservoir, and the Yakima River. Fish tissue quality control check material was submitted in duplicate to estimate analytical accuracy and precision.

Laboratory Quality Control Procedures

A portion of the largescale sucker sample collected from the Cowlitz River was used for matrix spike and matrix spike duplicate analyses to detect bias due to interferences from the sample matrix. Surrogate standards were added to each sample prior to extraction to evaluate the efficiency of the extractions. Matrix and surrogate spikes performed by the laboratory also provide estimates of accuracy and precision.

Data Review

Fish tissue analysis data packages and quality control results were reviewed and assessed by Karin Feddersen of Ecology's Manchester Environmental Laboratory. No significant problems were encountered. Minor difficulties are discussed in the attached data validation report.

Detection Limits

The values in Appendix B are quantitation limits, which are often different for each sample. Detection limits were not calculated separately, but were generally substantially lower than quantitation limits. A quantitation limit is the smallest concentration of a compound that the laboratory can quantify with a specified degree of confidence. When compounds are detected below the quantitation limit, these chemicals can often be positively identified, but the degree of confidence for the concentration of these compounds is lower than for those above the quantitation limit, and reported concentrations are qualified as estimates. In most instances, the level of detection was sufficiently low to compare with even the lowest criteria. However, comparison of qualified results to criteria should be made with caution.

While there is some uncertainty associated with the concentration of compounds detected below the quantitation limit, the probability of a false positive is still low in most cases. In a screening survey, such as the WSPMP, the consequences of a false positive are generally not serious. Detected compounds of interest would simply require additional sampling to verify their presence. False negatives would be more serious, indicating that there is no problem when one may be present.

Quality Control Samples

No accuracy or precision criteria have been established for any of the analytical methods used, but duplicate samples and matrix and surrogate spike analyses provide estimates of accuracy and precision. Recoveries near 100% indicate good accuracy and low relative percent difference (RPD) values indicate high precision between duplicate analyses. Evaluation of matrix and surrogate spike results is included in the attached data validation report. The laboratory has set the range for recommended matrix and surrogate spike recoveries in tissue samples at 50% to 150%. Data associated with recoveries above or below this range are "J" qualified. RPDs below 75% are considered acceptable.

Fish tissue quality control check material samples were submitted to the laboratory in duplicate. The check material was composed of frozen lake trout from Lake Michigan, obtained from the U.S. Fish and Wildlife Service in Ann Arbor, Michigan. This is not certified reference material, but the USFWS has been analyzing it since 1985 for their studies and have compiled considerable data to establish the expected values.

Appendix F-1 (cont.). Analytical Methods - QA/QC - Data Review

Appendix F-2 compares check material results to expected values. RPD values between the means of the duplicate analyses and the expected values were 50% or lower for all compounds except oxychlordane and heptachlor epoxide, which were 78% and 57% respectively. The average RPD was 30%. These results suggest good analytical accuracy.

Results from duplicate analyses (splits) are presented in Appendix F-3. Five sets of duplicate samples were analyzed, in addition to the quality control check sample that was analyzed in duplicate. RPDs ranged from 0-78% with an overall average of 17%. These results indicate good precision.

Replicate samples were collected to evaluate environmental variability between samples from the same site. Differences between replicate samples were generally small (Appendix F-4), and with an average RPD of 32% for the Scooteney Reservoir and Cowlitz River samples, were about double the differences between duplicate analyses. Coefficients of variation for the Yakima River samples were also low, averaging only 16%. Since some of the disparity between replicates can be attributed to analytical variability, differences between replicates due to environmental variability is probably low.

Appendix F-2. 1995 Fish Tissue Quality Control Check Material Results (μg/kg (ppb) wet weight)

Analyte	Mean Concentration (± ½ duplicate range)		Expected Value	RPD[1]
4,4'-DDD	60	± 21	65	9
4,4'-DDE	550	± 30	495	11
4,4'-DDT	52	± 4	31	50
cis-chlordane	92	± 3	82	11
trans-chlordane	45	± 1	45	0
cis-nonachlor	54	± 0	45	18
trans-nonachlor	125	± 5	94	28
oxychlordane	12	± 5	28	78
dieldrin	93	± 8	152	49
heptachlor epoxide	21	± 7	37	57
total PCBs	1675	± 75	1333	23

[1] - RPD = Relative Percent Difference, (difference/mean) x 100

Appendix F-3. 1995 Fish Tissue Duplicate Analysis Results (µg/kg (ppb) wet weight)

Analyte	Sample 1	Sample 2	RPD[1]
Redrock Lake largemouth bass (large)			
2,4'-DDD	1.3	1.8	32
2,4'-DDE	0.7	0.6	15
2,4'-DDT	0.7	0.6	15
4,4'-DDD	15	15	0
4,4'-DDE	130	130	0
4,4'-DDT	6.3	6.4	2
DDMU	5.3	5.3	0
chlorpyrifos	3.8	4	5
DCPA (dacthal)	4.9	4.7	4
dieldrin	8.5	8.7	2
hexachlorobenzene	0.7	0.6	15
trans-nonachlor	0.7	0.7	0
trifluralin	1.2	1.2	0
Royal Lake smallmouth bass			
4,4'-DDD	4.5	3.8	17
4,4'-DDE	68	67	1
4,4'-DDT	3.8	3.8	0
DDMU	7.9 U[2]	1.2	NC[3]
chlorpyrifos	3.9	3.7	5
DCPA (dacthal)	5.4	4.5	18
dieldrin	8.3	8.0	4
hexachlorobenzene	1.2	0.9	29
trifluralin	1.4	1.3	7
Scooteney Reservoir largemouth bass			
4,4'-DDD	7.5	5.9	24
4,4'-DDE	57	68	18
4,4'-DDT	5.7	3.2	56
chlorpyrifos	3.3	4.3	26
DCPA (dacthal)	7.8	8.7	11
dieldrin	8.7	11	23
hexachlorobenzene	1.2	1.3	8
trans-nonachlor	3.9 U	0.7	NC
trifluralin	1.2	1.4	15

[1] - RPD = Relative Percent Difference, (difference/mean) x 100

[2] - U = Undetected at or above the reported value.

[3] - NC = Not Calculated.

Appendix F-3 (cont.). 1995 Fish Tissue Duplicate Analysis Results (µg/kg (ppb) wet weight)

Analyte	Sample 1	Sample 2	RPD[1]
Yakima River carp			
2,4'-DDD	7.0	5.7	20
2,4'-DDE	5.4	4.7	14
2,4'-DDT	5.2	4.1	24
4,4'-DDD	51	41	22
4,4'-DDE	940	750	22
4,4'-DDT	12	8.4	35
DDMU	19	15	24
cis-chlordane	5.1	4.8	6
trans-chlordane	1.1	0.89	21
cis-nonachlor	2.9	2.4	19
trans-nonachlor	6.6	5.6	16
oxychlordane	0.42	0.27	43
dieldrin	9.6	8.0	18
heptachlor epoxide	0.68	0.54	23
hexachlorobenzene	0.53	0.45	16
trifluralin	3.8	3.3	14
PCB-1254	30	28	7
PCB-1260	120	91	27
Yakima River largescale sucker (Rep-1)			
2,4'-DDD	32	31	3
2,4'-DDE	20	20	0
2,4'-DDT	37	46	22
4,4'-DDD	210	190	10
4,4'-DDE	2900	3400	16
4,4'-DDT	250	320	25
DDMU	56	57	2
4,4'-dichlorobenzophenone	8.7	10	14
cis-chlordane	8.8	9.3	6
trans-chlordane	2.7	3.1	14
cis-nonachlor	4.0	4.6	14
trans-nonachlor	17	18	6
oxychlordane	1.9	2.1	10
dieldrin	42	45	7
heptachlor epoxide	0.92	1.1	18
hexachlorobenzene	1.6	1.8	12
kelthane	44	58	27
pentachloroanisole	0.96	1.0	4
toxaphene	250	230	8
trifluralin	12	15	22
PCB-1254	81	97	18
PCB-1260	220	230	4

[1] - RPD = Relative Percent Difference, (difference/mean) x 100

Appendix F-3 (cont.). 1995 Fish Tissue Duplicate Analysis Results (µg/kg (ppb) wet weight)

Analyte	Sample 1	Sample 2	RPD[1]
QC Check Material			
2,4'-DDD	4.8	11	78
4,4'-DDD	39	80	69
4,4'-DDE	580	520	11
4,4'-DDT	55	48	14
BHC-alpha	12	11	9
cis-chlordane	95	89	7
trans-chlordane	44	46	4
cis-nonachlor	54	54	0
trans-nonachlor	130	120	8
oxychlordane	7.5	17	78
DCPA (dacthal)	6.7	12	57
dieldrin	85	100	16
endrin	6	7.1	17
heptachlor epoxide	14	27	63
hexachlorobenzene	12	11	9
toxaphene	300	500	50
PCB-1254	1100	1000	10
PCB-1260	650	600	8

[1] - RPD = Relative Percent Difference, (difference/mean) x 100

Appendix F-4. 1995 WSPMP Fish Tissue Replicate Analysis Results (µg/kg (ppb) wet weight)

Analyte	Replicate 1	Replicate 2	RPD[1]
Scooteney Reservoir carp			
aldrin	0.7	0.6	15
trans-chlordane	1.5	0.8	61
dieldrin	28	19	38
4,4'-DDE	370	250	39
4,4'-DDD	46	28	49
4,4'-DDT	4.1	3.4	19
oxychlordane	0.9	3.9 U[2]	NC[3]
DDMU	10	7.1	34
cis-chlordane	2.4	1.4	53
cis-nonachlor	2.1	1.5	33
2,4'-DDE	1.9	1.3	38
trans-nonachlor	4.6	3.1	39
2,4'-DDD	4.6	3.2	36
2,4'-DDT	1.8	1.7	6
toxaphene	120	140	15
hexachlorobenzene	5.7	3.3	53
DCPA (dacthal)	32	26	21
chlorpyrifos	20	18	11
trifluralin	9.3	7.1	27
Cowlitz River largescale sucker			
4,4'-DDE	73	59	21
4,4'-DDD	10	7.6	27
4,4'-DDT	7.6	4.5	51
trans-nonachlor	3.7 U	2.4	NC
hexachlorobenzene	1.1	1.2	9
pentachloroanisole	1.9 U	0.6	NC
PCB-1254	37	66	56
PCB-1260	47	42	11

[1] - RPD = Relative Percent Difference, (difference/mean) x 100

[2] - U = Undetected at or above the reported value.

[3] - NC = Not Calculated.

Appendix F-4. 1995 WSPMP Fish Tissue Replicate Analysis Results (µg/kg (ppb) wet weight)

Analyte	Replicate 1[1]	Replicate 2	Replicate 3	COV[2]
Yakima River largescale sucker				
2,4'-DDD	32	26	21	21
2,4'-DDE	20	15	11	29
2,4'-DDT	42	36	24	27
4,4'-DDD	200	150	140	20
4,4'-DDE	3150	3000	1900	25
4,4'-DDT	285	250	180	22
DDMU	57	51	42	15
4,4'-dichlorobenzophenone	9.4	6.4	7.1	21
cis-chlordane	9.1	7.6	9.4	11
trans-chlordane	2.9	2.2	1.5	32
cis-nonachlor	4.3	4.0	5.0	12
trans-nonachlor	18	14	12	21
dieldrin	44	38	35	12
heptachlor epoxide	1.0	0.84	0.71	17
hexachlorobenzene	1.7	1.5	1.4	10
kelthane	51	51	55	4
oxychlordane	2.0	2.1	2.0	3
pentachloroanisole	1.0	1.0	1.0	0
toxaphene	240	230	200	9
trifluralin	14	11	9.8	19
PCB-1254	89	77	77	9
PCB-1260	225	220	150	21

[1] - Values are means of duplicate analyses

[2] - COV = Coefficient of Variation (%), (standard deviation/mean)x100

Appendix G-1. Pesticides Detected in Fish Tissue for the 1992 WSPMP (µg/Kg (ppb) wet weight)

Sample Site	Lake Chelan				Crab Creek			Walla Walla River		
					Rep1	Rep2				
Fish Species	LSS	RBT	KOK	KOK	LSS	LSS	MWF	LSS	LSS	WCR
Tissue Type	WF	FIL	FIL	EGG	WF	WF	FIL	WF	EGG	FIL
% Total Lipid	1.79	0.13	0.54	2.59	2.54	2.19	2.47	1.94	0.89	0.15
% Non-Polar Lipid	0.43	0.06	0.27	2.53	1.38	1.60	1.78	1.65	0.58	0.10
Analyte										
4,4'-DDE	133	53	398	1370	218	162	105	425	57	17
4,4'-DDD	29	2.2 J	17	59	63	26	20	51	7.2 J	1.7 J
4,4'-DDT	5.1 J	1.8 J	19	82	14	7 J	5.2 J	26	3.6 J	
2,4'-DDE	1.7 J		2 J	11				2.9 J		
2,4'-DDD	3 J		2.1 J	12 J	6.5 J	2 J	1.3 J	7.0		
2,4'-DDT			5.1 J	24						
total DDT	172	57	443	1558	302	197	132	512	68	19
DDMU	17 J	6.9 J	14 J	41	18 J	10 J	11 J	16 J	1.9 J	
alpha-BHC	0.5 J			1.5 J				0.5 J		
gamma-BHC (lindane)				0.5 J				7.9	2.3 J	1.3 J
heptachlor epoxide								8.3	2.1 J	3.7 J
dieldrin						3 NJ		5 J		
endrin			2.8 J	13 J						
kelthane			9 NJ	50 NJ						
methoxychlor					1.7 NJ	1.0 NJ				
alpha-chlordene				0.3 NJ						
gamma-chlordene				0.5 NJ						
cis-chlordane (alpha)	1.3 J		0.5 J	2.1 J		0.8 J		4.6 J	0.8 J	0.7 J
trans-chlordane (gamma)	1.3 J		0.6 J	0.8 J				4.9 J	0.7 J	0.7 J
cis-nonachlor			0.1 J	3.8 J				1.9 J		
trans-nonachlor			1.5 J	7.3 J				10 J		
oxychlordane	0.4 J		1.0 J	4.0 J				2.0 J		
total chlordane	3.0		3.7	18.0		0.8		23	1.5	1.4
hexachlorobenzene				2.1 J				6.9 J	2.7 J	2.1 J
pentachloroanisole				0.3 J						
PCB-1242				10 NJ						
PCB-1254	17 J	15 J	12 J	14 J	26 J	19 J	14 J	48 J	10 J	
PCB-1260				16 J	25 J	24 J	16 J	90	22 J	
total PCBs	17	15	12	40	51	43	30	138	32	

Fish species key
LSS=Largescale Sucker
RBT=Rainbow Trout
KOK=Kokanee (land-locked Sockeye Salmon)
MWF=Mountain Whitefish
WCR=White Crappie

Tissue type key
WF=Whole Fish
FIL=Fillet (muscle only)
EGG=Eggs

Data qualifier codes
J = The analyte was positively identified, but the value is an estimate.
NJ = There is evidence that the analyte is present. The value is an estimate.

Appendix G-1 (cont.). Pesticides Detected in Fish Tissue for the 1992 WSPMP (µg/Kg (ppb) wet weight)

	Yakima River						Mercer Slough		Lake River
	Rep1	Rep2	Rep1	Rep2	Rep1	Rep2			
Fish Species	LSS	LSS	LSS	LSS	SMB	SMB	LSS	RBT	LSS
Tissue Type	WF	WF	EGG	EGG	FIL	FIL	WF	FIL	WF
% Total Lipid	5.90	4.47	1.25	1.11	0.06	0.05	3.66	0.05	4.47
% Non-Polar Lipid	5.08	3.32	0.88	0.85	0.00	0.00	3.36	NAF[1]	3.66
Analyte									
4,4'-DDE	1420	532	107	252	45	43	144	15	157
4,4'-DDD	151 J	76	15	21	3.2 J	2.0 J	75	4.4 J	39
4,4'-DDT	94	45	8.6 J	15	1.3 J	1.0 J	18	3.5 J	17
2,4'-DDE	13	7.1	0.8 J	1.7 J					
2,4'-DDD	26	11	1.6 J	2.4 J			12		5.1 J
2,4'-DDT	13	6.4							2.2 J
total DDT	1717	678	133	292	50	46	249	23	220
DDMU	55	23	6.0 J	13	2.6 J	1.3 J	26 NJ	3 J	13 J
alpha-BHC									0.3 NJ
gamma-BHC (lindane)							1.1 J		0.4 NJ
dieldrin	42	31	11	12	3.3 J	3.3 J			
kelthane								1.4 NJ	
alpha-chlordene	1.7 J	0.7 J					1.7 J		
gamma-chlordene	5.6 J	2.0 J					2.7 J		
cis-chlordane (alpha)	21	7.4 J	1.4 J	2.0 J	0.4 NJ	0.3 NJ	24	2.1 J	3.0 J
trans-chlordane (gamma)	15	6.0 J	0.9 J	1.3 J	0.4 NJ	0.3 NJ	10 J	1.3 J	2.4 J
cis-nonachlor	8.1	3.6 J		0.4 J		0.5 J	17	0.7 J	1.2 J
trans-nonachlor	32	15	2.2 J	4.6 J	0.7 J	0.4 J	43	3.8 J	6.1 J
oxychlordane	7.5	2.9 J	0.8 J	1.5 J	0.5 J	0.5 J	2.3 J	0.4 J	1.0 J
total chlordane	84	35	5.3	9.8	2.0	2.0	96	8.3	13.7
hexachlorobenzene	1.7 J						2.9 J		1.7 J
pentachloroanisole	1.1 NJ	0.5 J		0.2 J			6.2 J	0.6 J	6.1 J
chlorpyrifos	3.37 J								
PCB-1242									21 NJ
PCB-1254	68 J	27 J	6 J	13 J	7 J		104 J	20 J	95
PCB-1260	164	49 J	13 J	27 J	9 J	8 J	275	31 J	83
total PCBs	232	76	19	40	16	8	379	51	199

[1] NAF = Not Analyzed For (insufficient sample volume for analysis)

Fish species key	LSS=Largescale Sucker	Tissue type key	WF=Whole Fish
	RBT=Rainbow Trout		FIL=Fillet (muscle only)
	SMB=Smallmouth Bass		EGG=Eggs

Data qualifier codes

J = The analyte was positively identified, but the value is an estimate.

NJ = There is evidence that the analyte is present. The value is an estimate.

Appendix G-2. Pesticides Detected in Fish Tissue for the 1993 WSPMP (µg/Kg (ppb) wet weight)

Sample Site	Walla Walla River					Nooksack River	Fishtrap Creek	Spokane River	
Fish Species	Suckers[1]	Suckers	Suckers	Carp[1]	Steelhead[1]	Suckers	RB trout	Suckers	RB trout
Tissue Type	Whole-Fish Replicate 1	Whole-Fish Replicate 2	Whole-Fish Replicate 3	Fillets	Fillets	Whole-Fish	Fillets	Whole-Fish	Fillets
% total lipid	4.67	6.41	3.87	3.91	7.01	4.56	3.89	4.73	2.69
2,4'-DDE		1							
4,4'-DDE	305	390	320	600	15	19	7	39	18
2,4'-DDD		8	5	9.8	1				
4,4'-DDD	43	63	40	97	15		2		
2,4'-DDT		1			1				
4,4'-DDT	13	14	17		4	6	2		
total DDT	**361**	**477**	**382**	**707**	**36**	**25**	**11**	**39**	**18**
4,4'-DDMU		10	6	15					
cis-chlordane	3	4	2	8	2	1	1		
trans-chlordane	2	4	2	8.5	1	1	1		
oxychlordane	0.3	1	1	1	1				
cis-nonachlor	2	3	2	5	1		1		
trans-nonachlor	5	7.8		13	3	3	1		1
total chlordane	**12**	**20**	**7**	**36**	**8**	**5**	**4**		**1**
DCPA (dacthal)					5				
dieldrin	4	5.6	4	10	4				
ethion		3		2					
gamma-BHC (lindane)	1	1		1	1	1			
heptachlor epoxide		4	3	8.2	4		1		
hexachlorobenzene	6.2	12	8.3	20	4.8				
PCB-1248								130	150
PCB-1254								950	430
PCB-1260	113		130	300				150	140
total PCBs	**113**		**130**	**300**				**1230**	**720**

[1] - Values are means of duplicate analyses

Appendix G-2 (cont.). Pesticides Detected in Fish Tissue for the 1993 WSPMP (µg/Kg (ppb) wet weight)

Sample Site	Wenatchee River		Mission Creek	Chehalis River			Salmon Creek	Vancouver Lake	
Fish Species	Suckers[1]	Suckers	RB Trout	Suckers	Suckers	M Whitefish	LM Bass	LM Bass	Carp
Tissue Type	Whole-Fish Replicate 1	Whole-Fish Replicate 2	Fillets	Whole-Fish Replicate 1	Whole-Fish Replicate 2	Fillets	Fillets	Fillets	Whole-Fish
% total lipid	4.98	5.18	1.84	4.66	3.30	5.72	2.82	3.85	7.80
2,4'-DDE	2								
4,4'-DDE	380	270	270	14	27	23	11	47	95
2,4'-DDD	8								
4,4'-DDD	68	47	51		3		4	17	45
2,4'-DDT	4					1			
4,4'-DDT	32	26	42						
total DDT	494	343	363	14	30	24	15	64	140
4,4'-DDMU	14	12						5	
cis-chlordane				2	3	2	1	1	5
trans-chlordane				2	2	3	1		
oxychlordane		1			1	1			1
cis-nonachlor					1				2
trans-nonachlor				3	5	5.6		3	
total chlordane		1		7	12	12	2	4	8
dieldrin									4
endosulfan sulfate			8						
heptachlor						0.5			
hexachlorobenzene				0.5	0.7	1			
PCB-1248	170								
PCB-1254	250	55		45	55	75		50	120
PCB-1260	48	49		35	34	68		60	160
total PCBs	468	104		80	89	143		110	280

[1] - Values are means of duplicate analyses

Appendix G-3. Pesticides and PCBs Detected in 1994 Fish Tissue Samples (µg/Kg (ppb) wet weight)

Sample Site	Lake Sacajawea		Palouse River		Okanogan River		
Fish Species	LS Sucker	Catfish	LS Sucker	Squawfish	LS Sucker	LS Sucker	Carp
Tissue Type	Whole-fish	Fillet	Whole-fish	Fillet	Whole-fish Replicate 1	Whole-fish Replicate 2	Fillet[1]
% Total Lipid	26.8	4.9	2.6	1.5	8.4	6.1	9.1
2,4'-DDE	0.91 J	1.4 J			1.4 J	2.2 J	12
4,4'-DDE	270	360	170	73	760	1100	1650
2,4'-DDD	5.6 J	2.9 J	2.8 J		13 J	18	135
4,4'-DDD	43	44	18		120	180	1050
2,4'-DDT						1.2 J	
4,4'-DDT	15	21	12 J		21	39	5.6 J
total DDT	335	429	203	73	915	1340	2853
4,4'-DDMU	7.0 J	5.8 J	2.7 J		19	28	125
cis-chlordane	2.9 J	4.0 J	5.7	1.2 J			
trans-chlordane	3.0 J	5.8	14				
oxychlordane	1.4 J		7.2	2.4 J			
cis-nonachlor	2.3 J	3.2 J	1.7 J	0.75 J	0.38 NJ	0.42 J	
trans-nonachlor	5.1 J	6.5 J	4.7 J	2.1 J		0.81 NJ	
total chlordane	14.7	19.5	33	6.5	0.38	1.23	
DCPA (Dacthal)	25	9.5					
dieldrin	7.5	4.0 J	13	7 NJ			
ethion	4.5 J	5.4 J					
alpha-BHC							
gamma-BHC (Lindane)			0.27 NJ	0.44 J			
heptachlor epoxide	1.0 J	0.45 J	14	6.3			
hexachlorobenzene	6.9	4.7	10	3.6	0.66 J	0.78 NJ	0.89 J
methoxychlor	1.2 J					0.70 NJ	
PCB-1254	29 NJ	26 NJ	13 J		22 J	24 NJ	25 J
PCB-1260	33	35 J	18 J	11 J	34 J	48 J	20 NJ
total PCBs	62	61	31	11	56	72	45

[1] - Values are means of duplicate analyses

J - The analyte was positively identified. The associated numerical value is an estimate.

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

Appendix G-3 (cont.). Pesticides and PCBs Detected in 1994 Fish Tissue Samples (µg/Kg (ppb) wet weight)

Sample Site	Lake Chelan				Entiat River		Soleduck River
Fish Species	LS Sucker	Kokanee	RB Trout	SM Bass	LS Sucker	LS Sucker	M Whitefish
Tissue Type	Whole-fish	Fillet	Fillet	Fillet	Whole-fish Replicate 1	Whole-fish Replicate 2	Fillet
% Total Lipid	4.7	1.6	1.5	3.9	4.9	4.5	6.5
2,4'-DDE	1.6 J				4.0 J	1.6 J	
4,4'-DDE	800	140	56	330	1700	1100	
2,4'-DDD	9.7 NJ	2.5 J		4.8 J	28	11 J	
4,4'-DDD	93	12 NJ		34	130	120	
2,4'-DDT					2.0 J		
4,4'-DDT	53	12 J		28	160	130	
total DDT	957	167	56	397	2024	1363	
4,4'-DDMU	34	3.7 J	5.0	12	47	26	
cis-chlordane				0.5 J			
trans-chlordane	0.45 NJ						
oxychlordane		0.47 J		0.48 NJ			
cis-nonachlor	0.95 J	1.1 NJ	0.29 J	0.61 NJ	0.28 NJ		
trans-nonachlor	2.3 J	1.2 NJ	0.31 NJ	0.86 J	2.2 NJ	1.0 NJ	
total chlordane	3.7	2.8	0.60	2.4	2.5	1.0	
DCPA (Dacthal)							
dieldrin							
ethion							
alpha-BHC	0.38 J			0.38 J			
gamma-BHC (Lindane)							
heptachlor epoxide							
hexachlorobenzene					0.63 J		
methoxychlor							
PCB-1254	34 J	84	65	16 J	28 NJ	20 NJ	
PCB-1260	35 J	15 J	15 NJ		36 J	40 J	
total PCBs	69	99	80	16	64	60	

J - The analyte was positively identified. The associated numerical value is an estimate.

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

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Data Validation Report

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Manchester Environmental Laboratory

7411 Beach Dr E
Port Orchard Washington 98366
July 31, 1996

Project: **WSPMP Fish Tissue**

Samples: 95378230 through 95378254, 95388030 through 95388036

By: Karin Feddersen KF

These samples were analyzed for Pesticides and PCB's, employing the dual column confirmation technique, and for % Lipids and % Solids.

Holding Times:

These samples were extracted and analyzed within the method-specified holding times.

Method Blanks:

No analytes of interest were detected in the method blanks.

Initial Calibration:

The % Relative Standard Deviations were within the maximum of 20%, or the coefficient was greater than 0.995, for all target analytes with several exceptions which did not affect the results.

Continuing Calibrations:

The percent difference between the initial and continuing calibration standards were within the maximum of 25%, with several exceptions which did not affect the results.

Matrix Spikes (MS and MSD):

Sample 95388230 was analyzed as MS and MSD. Matrix spike recoveries are within QC limits of 50% to 150% with two exceptions. Kelthane recovery was 0% in both spikes. It apparently degraded completely to 4'4-Dichlorobenzophenone (DCBP). DCBP recoveries were 210% and 280%. Subtraction of the calculated Kelthane contribution yields recoveries of 79% and 147%.

Positive results for DCBP have been qualified with "NJ" to indicate that some or all of the DCBP present in the samples may be due to the degradation of Kelthane. Positive results for Kelthane have been qualified "J". Also, since there is little information available regarding other possible breakdown products, all non-detects for these compounds have been qualified "UJ".

The MS/MSD recoveries for Captan and Captafol are relatively low. This was expected since the stability these compounds is somewhat less then the other targets. They both have the tendency to degrade the dicarboximide base structure, losing the chlorinated portions of their respective structures. However, because the precision between recovery results is good, no qualifiers were applied.

P'p-DDE recoveries are high due to the high native concentration. The concentration of p'p-native to the sample was much higher than the amount spiked. Thus accurate quantitation of this analyte in the MS and MSD is not possible. No qualification of the results is necessary.

Duplicate:

Sample 95378254 was analyzed in duplicate. Precision data between the two analyses is acceptable for all analytes except Heptachlor Epoxide. There was an unclear baseline in the original analysis. The duplicate result is likely to be more accurate.

Sample 95378230 was also analyzed in duplicate. All target compounds in the duplicate analysis are approximately 15 to 20% below the values reported for the original analysis. Since the surrogate recoveries are similarly low, the differences are most likely due to sample loss during preparation.

Surrogates:

Four surrogates are reported for each sample. The recommended range for surrogate recovery in tissue is between 50% and 150%.

Dibromooctafluorobiphenyl (DBOB) recoveries are slightly below 50% in samples 953. However, recoveries for Decachlorobiphenyl (DCB) and Tetrachloro-m-xylene (TCMX) were acceptable. DCB and TCMX are better indicators of analyte recovery. No qualification of the data is warranted in these instances.

Dibutyl Chlorendate (DBC) recoveries were slightly below 50% in samples 95378246, 95378253 and the duplicate analysis of 95378254. DBC is recovered in the 15% florisil fraction. Results for all analytes typically found in this fraction have been qualified for these samples: Detected analytes have been qualified with "J"; non-detects with "UJ".

Surrogate recoveries were acceptable in all other samples and in the blanks.

Sample Results:

All fish tissue results are reported on an "as received" (wet weight) basis.
The Mass Spectrometer was used to confirm compounds in some instances.

All positive Trifluralin results are estimated from a single concentration standard and are qualified with "J". Non-detects are qualified "UJ".

Toxaphene was confirmed present by GC/MS. Toxaphene patterns in samples 95378232, 95378233, and 95378234 were inconsistent, most likely due to weathering. This makes accurate quantitation difficult. All positive results ion these samples have therefore been qualified with "J".

Evidence for the presence of Toxaphene in samples 95378242, 953782354, and 953782354 duplicate is less conclusive, and it was not confirmed present by GC/MS. Toxaphene results for these samples have been therefore qualified "NJ".

This data is acceptable for use with the qualifications mentioned.

DATA QUALIFIER CODES:

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

J - The analyte was positively identified. The associated reported value is an estimate.

UJ - The analyte was not detected at or above the reported value. The reported value is an estimate.

NJ - There is evidence that the analyte is present. The associated reported value is an estimate.