CHRISTINE O. GREGOIRE
Director

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STATE OF WASHINGTON
DEPARTMENT OF ECOLOGY
7171 Cleanwater Lane, Building 8, LH-14 • Olympia, Washington 98504-6814

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TO: Carl Nuechterlein

FROM: Art Johnson

SUBJECT: Results of Screen for EPA Xenobiotics in Sediment and Bottom Fish from Lake Roosevelt (Columbia River).

In June 1990, as part of Ecology's investigation of contaminants in Lake Roosevelt, a series of sediment and bottom fish samples were collected from Lake Roosevelt and vicinity for analysis of polychlorinated dibenzo-*p*-dioxins (PCDDs) and -dibenzofurans (PCDFs). The impetus for this survey was the need to better understand the spatial distribution of these compounds as a result of their discharge by the Celgar bleached kraft pulp mill in Castlegar, B.C., approximately 30 river miles above the international border. The results of these and other PCDD/PCDF analyses on Lake Roosevelt samples have been reported elsewhere (Johnson *et al.*, 1991a,b,c).

The sediment and bottom fish samples were subsequently analyzed for an additional 44 compounds that are the subject of the present report. These 44 compounds, along with 15 PCDDs and PCDFs, compose the list of target analytes* for EPA's National Bioaccumulation Study - a survey of bioaccumulative pollutants in U.S. fish (Tetra Tech, Inc., 1991 - draft). This list of chemicals, termed xenobiotics (xeno meaning foreign), was developed through a detailed screening process that considered bioaccumulation potential, human toxicity, exposure potential, persistence in aquatic environments, biochemical fate in fish, and analytical feasibility. The analytical protocol developed to detect these compounds constitutes one of the most thorough single analyses of bioaccumulative organic chemicals currently available. Analytical support for the present survey was provided by EPA Region 10.

* Mercury is also on this list, but has been analyzed in previous Ecology surveys of Lake Roosevelt (Johnson *et al.*, 1988; Johnson, 1989) so was not analyzed here.

METHODS

Sampling

Figure 1 shows where sediment and fish were collected. Six sites were sampled in Lake Roosevelt between the international border and Grand Coulee Dam, and one site each in the Spokane River (the major tributary to Lake Roosevelt) and Rufus Woods Lake (the Columbia River reservoir below Lake Roosevelt formed by Chief Joseph Dam). The Spokane samples were collected behind Long Lake Dam. One sample each of sediments and fish were collected at each location. Appendix A has detailed descriptions of the sampling sites. Field work was conducted June 26-28, 1990.

Each sediment sample was a composite of three grabs taken with a stainless steel 0.1 m^2 van Veen grab sampler. Only the top 2-cm surface layer was taken for analysis. Sediments were transferred to stainless steel beakers and homogenized by stirring with stainless steel spoons. Spoons and beakers had been washed with Liqui-Nox® detergent followed by rinsing with Milli-Q® water, pesticide-grade acetone, and pesticide-grade hexane. Sample containers were 8 oz. amber glass with teflon lid-liners, specially cleaned for low-level organics analysis (I-CHEM, Hayward, California, series 300). The samples were stored on ice in the field.

Largescale suckers (*Catostomus macrocheilus*) were collected for analysis. This species is a bottom dweller that feeds on a variety of organisms such as crustaceans, insect larvae, snails, and detritus (Wydoski and Whitney 1979). Apart from their benthic habit, largescale suckers were selected because they are available throughout the study area and because results from an earlier Ecology survey (Johnson *et al.*, 1988) suggested they do not move extensively through the lake.

The fish were collected by electro-shocking. After recording the length and weight of each specimen, they were individually wrapped in aluminum foil, placed in polyethylene bags, and stored on ice. The fish were analyzed as composites of five whole fish per sample.

Analysis

Table 1 shows the list of target analytes. Ancillary analyses included percent total organic carbon (TOC) and grain size in sediment samples, and percent lipid in fish samples.

Xenobiotics analysis and lipid determination were done by the EPA Environmental Research Laboratory in Duluth, Minnesota. Sample preparation and analysis followed methods described in Call *et al.*, (1991). TOC and grain size were analyzed at Analytical Resources Inc. and Hart Crowser, respectively, both of Seattle, Washington. Analytical methods for TOC and grain size are described in Tetra Tech, Inc. (1986).

Quality Assurance (QA) procedures for these analyses were established by the EPA Duluth laboratory (EPA *et al.*, 1990). All data included in the present report passed EPA's quality assurance criteria.

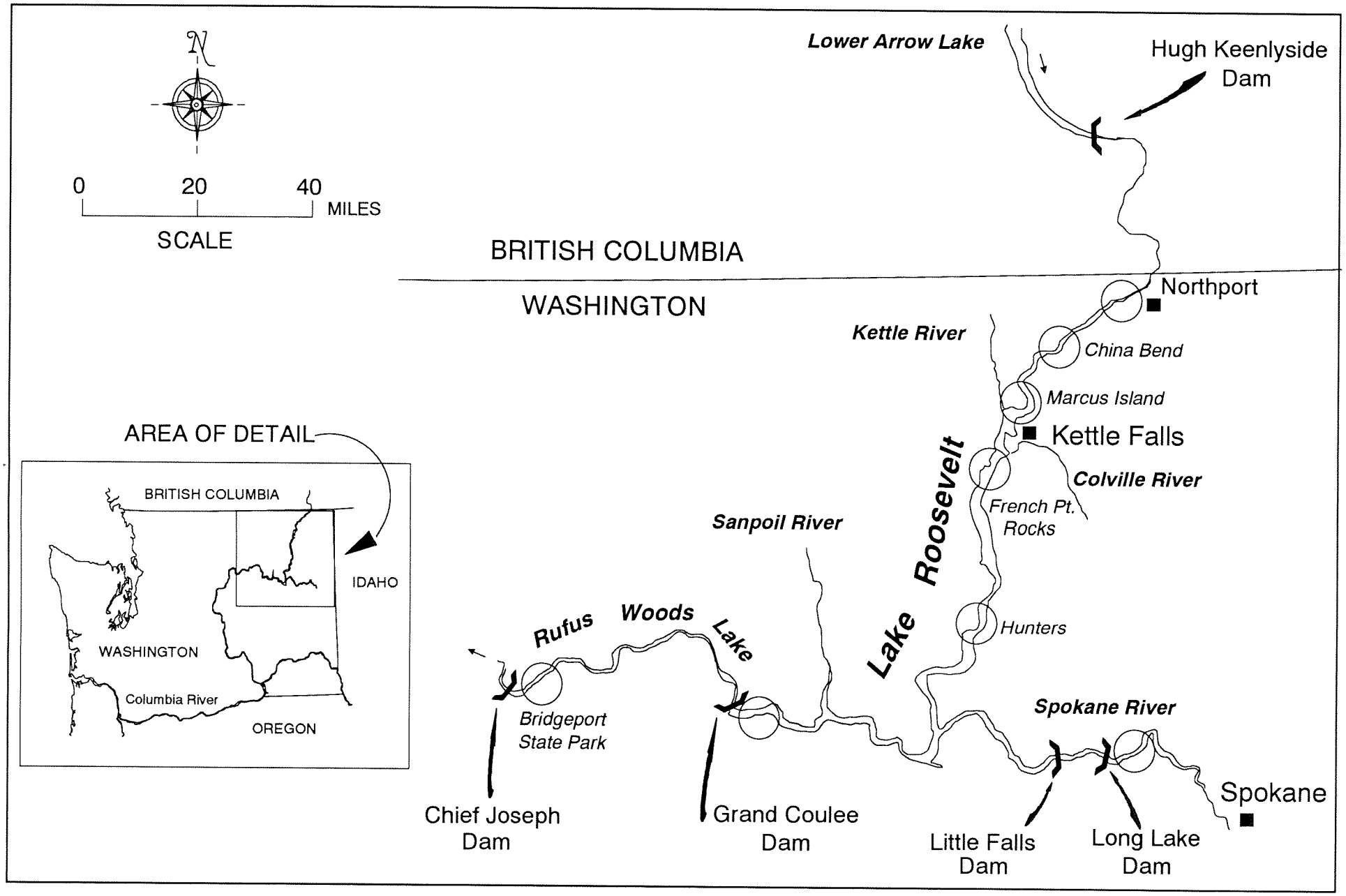

Figure 1. Location of Sediment and Bottom Fish Samples Collected by Ecology, June 1990

Table 1. Compounds Analyzed in Sediment and Bottom Fish Samples.

Compound Name	Chemical Abstracts Registry No.
1,3,5- Trichlorobenzene	108-70-3
1,2,4- Trichlorobenzene	120-82-1
1,2,3- Trichlorobenzene	87-61-6
Hexachlorobutadiene	87-68-3
1,2,4,5- Tetrachlorobenzene	95-94-3
1,2,3,5- Tetrachlorobenzene	634-90-2
Biphenyl	92-52-4
1,2,3,4- Tetrachlorobenzene	634-66-2
Pentachlorobenzene	608-93-5
Trifluralin	1582-09-8
Alpha-BHC	319-84-6
Hexachlorobenzene	118-74-1
Pentachloroanisole	1825-21-4
Gamma-BHC (Lindane)	58-89-9
Pentachloronitrobenzene	82-68-8
Diphenyl Disulfide	882-33-7
Heptachlor	76-44-8
Chlorpyrifos	2921-88-2
Isopropalin	33820-53-0
Octachlorostyrene	29082-74-4
Heptachlor epoxide	1024-57-3
Oxychlordane	27304-13-8
Chlordane, trans	5103-74-2
Chlordane, cis	5103-73-1
Nonachlor, trans	39765-80-5
p,p'- DDE	72-55-9
Dieldrin	60-57-1
Nitrofen	1836-75-5
Endrin	72-20-8
Perthane	72-56-0
Nonachlor, cis	3734-49-4
Methoxychlor	72-43-5
Dicofol (Kelthane)	115-32-2
Mirex	2385-85-5
Total Monochlorobiphenyl	27323-18-8
Total Dichlorobiphenyl	25512-42-9
Total Trichlorobiphenyl	25323-68-6
Total Tetrachlorobiphenyl	26914-33-0
Total Pentachlorobiphenyl	25429-29-2
Total Hexachlorobiphenyl	26601-64-4
Total Heptachlorobiphenyl	28655-71-2
Total Octachlorobiphenyl	31472-83-0
Total Nonachlorobiphenyl	53742-07-7
Total Decachlorobiphenyl	2051-24-3
Total Polychlorinated Biphenyls	1336-36-3

Limited information is available to assess the precision of the data reported here. Based on results from one pair each of laboratory duplicates, the relative percent difference between duplicate analyses of total pentachlorobiphenyls (PCBs) in sediment and dichlorodiphenyl-dichloroethylene (DDE) in fish tissue was 40% and 26%, respectively.

The following limits of quantitation were achieved during analysis of the sediment and fish tissue samples:

PCBs: mono-trichlorobiphenyls	- 1.25 ng/g (parts per billion)
tetra-hexachlorobiphenyls	- 2.50 ng/g
hepta-octachlorobiphenyls	- 3.75 ng/g
nona-decachlorobiphenyls	- 6.25 ng/g
Pesticides and other compounds:	2.5 ng/g

RESULTS AND DISCUSSION

Detection of target compounds was almost exclusively limited to PCBs and DDE, a metabolite of DDT. Additional compounds detected in sediment samples, but below limits of quantitation, were 1,2,3- and 1,2,4-trichlorobenzene at Lake Roosevelt near Grand Coulee, and biphenyl in Lake Roosevelt off French Point Rocks. No additional compounds were detected in any fish samples. In light of their single detection and low concentration, the trichlorobenzene and biphenyl data are not discussed further.

The concentrations of PCBs and DDE measured in sediment and fish samples are in Tables 2 and 3, respectively. PCBs were detected in sediment and/or fish from six of the eight sampling sites. Tetra-, penta-, and hexachlorobiphenyls were the predominant PCB congener groups in both media. DDE was primarily detected in fish samples - six of eight sites - with only one site having DDE detected in sediment.

The highest level of PCBs was found in the Spokane River samples. Total PCB concentrations were 20.8 ng/g (dry weight) in sediment, and 337 ng/g (wet weight) in fish. PCB concentrations in samples from Rufus Woods Lake were among the lowest found in the survey. Total PCBs in sediment were 4.9 ng/g; no PCBs were detected in the fish.

Results for Lake Roosevelt showed PCB levels generally intermediate between those in the Spokane River and Rufus Woods Lake. Sediment collected near Grand Coulee, below the Spokane confluence, contained a total PCB concentration of 25.2 ng/g, comparable to sediments in the Spokane. The Grand Coulee fish sample, however, had only 35.8 ng/g total PCBs, lower than the Spokane sample by a factor of 10.

Upstream of Grand Coulee there was no concordance in detection of PCBs in sediment and fish tissue. Fish from China Bend and Marcus Island, for example, had somewhat higher levels of PCBs (59.4 - 61.1 ng/g) than fish at Grand Coulee, but no PCBs were detected in the sediments. Conversely, French Point Rocks, the only site above Grand Coulee where PCBs were detected (3.8 ng/g) in Lake Roosevelt sediments, had no PCBs detected in fish.

Table 2. PCB and DDE Concentrations in Sediment Samples (ng/g, dry weight; parts per billion).

EPA Sample No. (DJ0226-)	Location	Approximate River Mile	Sampling Depth (ft.)	Percent Fines*	Percent TOC	Tetrachloro-biphenyls	Pentachloro-biphenyls	Hexachloro-biphenyls	Total PCBs	p,p'-DDE
				LAKE ROOSEVELT						
02	Northport	735	70	0	0.10	ND	ND	ND	ND	ND
05	China Bend	724	54	0	0.04	ND	ND	ND	ND	ND
07	Marcus Island	709	80	0	0.25	ND	ND	ND	ND	ND
09	French Pt. Rocks	692	86	76	2.40	1.8D	3.7	1.6D	3.7	ND
12	Hunters	661	105	88	1.08	ND	ND	ND	ND	ND
16	Grand Coulee	601	155	97	1.58	5.5	11.9	7.8	25.2	2.1
				RUFUS WOODS LAKE						
18	Bridgeport St. Park	546	120	63	1.26	1.2D	4.9	2.4D	4.9	ND
				SPOKANE RIVER						
14	Long Lake	39	72	99	2.62	4.5	10.2	6.1	20.8	ND

ND = Not detected

D = Below quantitation limit, not included in total

* $\leq 62\ \mu m$

Table 3. PCB and DDE Concentrations in Whole Largescale Sucker Samples (ng/g, wet weight; parts per billion).

EPA Sample No. (DJ0226-)	Location	Approximate River Mile	Mean Length (mm)	Mean Weight (g)	Percent Lipid	Trichloro-biphenyls	Tetrachloro-biphenyls	Pentachloro-biphenyls	Hexachloro-biphenyls	Heptachloro-biphenyls	Total PCBs	p,p'-DDE
					LAKE ROOSEVELT							
01	Northport	733	469	1,054	6.6	ND	ND	ND	ND	ND	ND	19.2
04	China Bend	722	486	1,143	5.6	ND	ND	12.2	38.2	10.7	61.1	18.9
06	Marcus Island	709	524	1,500	9.4	ND	4.8	40.9	13.7	ND	59.4	19.4
08	French Pt. Rocks	697	484	1,188	8.4	ND	ND	ND	ND	ND	ND	ND
11	Hunters	661	527	1,303	5.3	ND	ND	ND	ND	ND	ND	ND
15	Grand Coulee	600	504	1,195	10.1	ND	ND	15.5	20.4	ND	35.8	41.8
					RUFUS WOODS LAKE							
17	Bridgeport St. Park	546	505	1,560	2.4	ND	ND	ND	ND	ND	ND	27.3
					SPOKANE RIVER							
13	Long Lake	37	437	815	5.4	9.3	143	107	57.1	19.5	337	69.7

ND = Not detected

Compared to PCBs, DDE concentrations were more consistent throughout the study area. DDE was below detection limits in all sediment samples except for a trace amount (2.1 ng/g) at Grand Coulee. DDE was detected in most fish samples from Lake Roosevelt (four of six sites) and in Rufus Woods Lake and Spokane River fish. Concentrations were between 18.2 ng/g and 69.7 ng/g.

Results of this survey are consistent with U.S. Fish and Wildlife Service (USFWS) data on PCBs and chlorinated pesticides in Lake Roosevelt fish. As part of the National Contaminant Biomonitoring Program, USFWS has established a sampling station in Lake Roosevelt at Grand Coulee. The most recent data from this station show 10 - 60 ng/g DDE and 100 ng/g total PCBs in whole largescale suckers samples collected in 1984 (Schmitt *et al.*, 1990). Several other pesticides not analyzed in Ecology's samples - DDT, DDD (also a DDT metabolite), and toxaphene - were also reported by USFWS as present in these samples, but they were inconsistently detected.

PCBs and DDE are ubiquitous contaminants. Although use of PCBs and the DDE parent compound DDT was restricted or banned in the U.S. during the 1970s, they are among the most frequently detected organochlorines in fish. DDE was detected in fish samples from all 112 stations of the USFWS National Contaminant Biomonitoring Program (Schmitt *et al.*, 1990) and 98% of the 388 stations in the previously mentioned EPA National Bioaccumulation Study (Tetra Tech, Inc., 1991-draft). The detection frequency of PCBs in fish exceeded 90% in both studies.

To put results from the present survey in better perspective, PCB and DDE data from the USFWS and EPA national fish monitoring programs have been summarized in Table 4. Also included in the table are recommendations by the National Academy of Sciences for protection of fish-eating predators and human health advisory levels of the U.S. Food and Drug Administration and Canada. Comparison of these data with present survey results shows that the levels of PCBs and DDE in Lake Roosevelt, Rufus Woods Lake, and Spokane River fish samples are moderate to very low.

Although the PCB concentrations in the Spokane River fish sample is not high in comparison to the information in Table 4, PCBs may be of some concern here. Historical Ecology data on PCBs in Spokane River fish collected between 1980 and 1983 are summarized in Table 5. While these results are scattered with respect to location and species, a number of fish samples have had in excess of 500 ng/g total PCBs, with several samples over 1,000 ng/g.

SUMMARY AND RECOMMENDATIONS

Forty-four bioaccumulative organic chemicals were analyzed at parts per billion levels in sediment and fish samples from six sites in Lake Roosevelt, and one site each in Rufus Woods Lake and the Spokane River. Detection of target compounds was essentially limited to PCBs and DDE (a metabolite of DDT), which were detected in sediment and/or fish from half or more of the sampling sites, including Rufus Woods Lake and the Spokane River.

Table 4. Present Study Results Compared to Concentrations of PCBs/DDE in U.S. Fish and Relevant Recommendations/Criteria (ng/g, wet weight; parts per billion).

	Total PCBs	p,p'- DDE
Concentration range in whole fish, present study:	ND - 337	ND - 69.7
Levels in U.S. fish:		
1. USFWS National Contaminant Biomonitoring Program (Schmitt et al., 1990)		
geometric mean (n = 321)	390	190
maximum	6,700	4,740
2. EPA National Bioaccumulation Study (Tetra Tech, Inc., 1991-draft)		
background sites (mean ± SD, n = 33)	49 ± 98	44 ± 75
NASQAN sites (mean ± SD, n = 31)	146 ± 348	97 ± 135
industrial/urban sites (mean ± SD, n = 258)	2,316 ± 8,788	309 ± 1,084
agricultural sites (mean ± SD, n = 13)	93 ± 292	844 ± 1,096
Recommendations/Criteria:		
1. Residues in diets to protect fish-eating predators (NAS, 1973)	500	1,000
2. Human health		
FDA	2,000	5,000
Canada	2,000	5,000

ND = not detected

Table 5. Summary of Ecology Data on PCBs in Spokane River Fish (ng/g, wet weight; parts per billion).

Approximate River Mile	Species	Tissue	No. Samples	Year Collected	Total PCB Concentrations Mean	Total PCB Concentrations Maximum	Reference
87	Longnose Sucker	Whole	3	1983	529	986	Bailey & Singleton (1984)
87	Longnose Sucker	Whole	3	1983	133	270	Hopkins et al., (1985)
66	Largescale Sucker	Whole	2	1980,81	195	230	Hopkins et al., (1985)
66	Bridgelip Sucker	Whole	3	1982,83	455	697	Hopkins et al., (1985)
66	Bridgelip Sucker	Viscera	1	1983	---	1,464	Hopkins et al., (1985)
66	Mountain Whitefish	Various	3	1983	308	424	Hopkins et al., (1985)
66	Northern Squawfish	Whole	3	1980,82	1,220	2,300	Hopkins et al., (1985)
37	Largescale Sucker	Whole	1	1990	---	337	Present Study

The highest levels of PCBs occured in the Spokane River, where sediment and fish samples contained 20.8 ng/g and 337 ng/g total PCBs, respectively. DDE was primarily detected in fish tissue. Concentrations were generally comparable throughout the study area, ranging from 18.2 - 69.7 ng/g

Based on comparison with data reported from EPA and USFWS national surveys of U.S. fish and other criteria, the PCB and DDE concentrations observed in the present study were moderate to very low. Historical data on PCBs in Spokane River fish show some evidence of significant contamination. Because of the long term persistence of PCBs, a more thorough evaluation of these compounds should be conducted in the Spokane drainage.

AJ:kd

cc: Claude Sappinston
Carol Jolly

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