WASHINGTON STATE
DEPARTMENT OF
ECOLOGY

Analysis of Fish Tissue from Long Lake (Spokane River) for PCBs and Selected Metals

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WASHINGTON STATE DEPARTMENT OF ECOLOGY

Analysis of Fish Tissue from Long Lake (Spokane River) for PCBs and Selected Metals

by
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November 2002

Waterbody No. WA-54-9040 (QZ45UE)
Long Lake - Lower Spokane River

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Abstract

Lead and polychlorinated biphenyls (PCBs) have been found in fish from the upper Spokane River at concentrations that pose potential risks for human consumption. To evaluate aquatic conditions, over 200 fish from five species were collected from two reaches of Long Lake, Spokane County. The Washington State Department of Ecology and the Washington Department of Fish and Wildlife (WDFW) collected fish on June 18 and 19, 2001.

The species were largemouth bass, largescale suckers, mountain whitefish, smallmouth bass, and yellow perch. These fish were grouped into 28 species-specific composite samples, ranging in size from 4 to 11 fish. Composite, skin-on fillet samples were analyzed for PCB Aroclors, PCB congeners, percent lipids, zinc, cadmium, lead, and, in some instances, mercury. A few whole fish also were analyzed.

All fish were aged by WDFW personnel. Age, weight, length, and chemical composition of the fish are reported in this study. Chemical concentrations in edible fillets were compared to National Toxics Rule (NTR) thresholds. When a numeric standard was unavailable, concentrations were compared to U.S. Environmental Protection Agency risk screening numbers.

Zinc, cadmium, and mercury concentrations were substantially below regulatory or screening criteria. For PCB Aroclors, fish tissues varied between 7 and 77 times the NTR criteria. Percent lipids predicted about 74 to 82 percent of total apparent Aroclor concentrations. Fish age and length for the composites analyzed was a poor predictor of chemical concentrations. Compared to historic data in the Spokane River system, PCB levels in mountain whitefish fillets declined sharply. Samples from whole and fillet largescale suckers are slightly lower than concentrations from 1993 and 1999.

These data have been provided to the Washington State Department of Health for further evaluation of human health impacts.

Acknowledgements

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Glossary and Abbreviations

Aroclor: a former commercial mixture of polychlorinated biphenyl (PCB) congeners. The last two digits of the identity represent the chlorine content in percent.

Coelute: to merge with other peaks on the chromatograph. Results from coeluting congeners are combined with one or more other congeners, and the value is a sum of those congeners.

Congener: an individual PCB with a precise orientation of chlorine atoms.

CRM: certified reference material, a generic term.

Data qualifiers (used in laboratory reports):

C		The congener coelutes
U	-	The analyte was not detected at or above the reported value.
J	-	The analyte was positively identified. The associated numerical value is an estimate.
UJ	-	The analyte was not detected at or above the reported estimated result.
NJ	-	There is evidence that the analyte is present. The associated numerical result is an estimate.

Elute: to segregate out of a mixture at a specific time and place on a gas chromatographic column.

OPR: ongoing precision and recovery, similar to internal check standards. Used to measure instrument drift.

NAD: North American Datum.

NTR: National Toxics Rule, 40 CFR Part 131 as amended.

RM: river mile, distance upstream in statute miles from river mouth.

Segment: as used in this report, a segment is a geographic river reach, irrespective of data or statistical implications.

SRM: standard reference material, a specific commercially available CRM with a product number assigned by the commercial distributor.

Stratum (plural is Strata): a grouping of measurements to partition variance in such a way as to assign as much as possible to differences among strata. In this way, variation within strata is minimized.

TEQ: toxic equivalent quotient, a conversion of various specific congeners into standard toxic units (typically based on one compound) using established toxicity conversions.

WWTP: wastewater treatment plant.

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Introduction

In the Spokane River, upstream from river mile 58.1 at Nine-Mile Dam, elevated polychlorinated biphenyls (PCBs) have been historically detected in sediments and fish tissue (Johnson, 1994; 1997; 2000). Concentrations in fish tissue from the Spokane River above Nile-Mile Dam have been high enough to warrant an ecological risk assessment (Johnson, 2001a) and the issuance of a human health fish consumption advisory (Spokane Regional Health District, 2001). The upper Spokane River health advisory for fish is issued jointly by the Washington State Departments of Ecology (Ecology) and Health (DOH) and the Spokane Regional Health District.

Both lead and PCBs have been found in rainbow trout, mountain whitefish, and largescale suckers from the upper Spokane River at concentrations that pose a potential risk to consumers. Metals concentrations, especially lead, cadmium, and zinc, are elevated in the Spokane River system due to contributions from the Coeur d'Alene Basin mining districts in Idaho (USEPA, 1988). A fish consumption advisory has also been issued for lead in the upper Spokane River. Various PCBs are known to enter the river from industrial sites and municipal wastewater treatment plants in and around the city of Spokane (Golding, 2001).

To date, an insufficient number of fish have been analyzed from Long Lake to evaluate potential human or ecological risks from metals or PCBs below Nine-Mile dam. The objectives of this project were to:

- Describe the nature and extent of fish contaminant concentrations in Long Lake.
- Determine the fish tissue concentrations of PCB Aroclors in edible fillet tissues.
- Determine the PCB congener concentrations in edible fillet tissues.
- Determine relationships between fish ages, weights, lengths, and contaminant concentrations.
- Screen largemouth bass for mercury concentrations.
- Compare fish tissue contaminant concentrations with historic data.

This project collected data on five species of fish via 28 composite samples. The data from this study have been provided to DOH to evaluate potential Long Lake fish consumption risks. Both PCB Aroclors and PCB congeners were analyzed to aid in assessing PCB fate and potential human health and ecological risks.

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Methods

Study Design

In July 2001, the Washington Department of Fish and Wildlife (WDFW) conducted a comprehensive population survey of fish in Long Lake. Ecology's Eastern Regional Office coordinated a joint collection of fish with Ecology's Environmental Assessment Program to evaluate fish tissue PCB and metals concentrations. For the collection efforts, two lake segments were defined, and Ecology/WDFW personnel targeted the collection of 30 fish from five species from each zone. The five species chosen represent a range of feeding guilds and both pelagic and benthic species.

Because sediment PCB concentrations vary within Long Lake (Johnson, 2001b) and fish tissue concentrations also may vary, an upper and a lower lake strata were established. These two strata were defined using WDFW specified bank reaches (Figure 1). Latitudes and longitudes for the upstream and downstream points are shown in Table 1. A target of 30 fish from each species/segment was established to provide three composite samples of ten fish each. For statistical purposes, these geographic river reaches stratify the sampling efforts. The geographic strata were intended to partition some of the variance in means. The composite sizes were considered necessary to establish, with high confidence, the mean contaminant concentration in the fish tissues.

Table 1. Long Lake sampling segments (defined by a line drawn between the following starting and ending coordinates).

	Segment Length		Latitude	Longitude	Latitude	Longitude
Upper Lake	5.7 miles	Start	N47^{0}47'37.23"	W117^{0}32'6.71"	N47^{0}47'39.16"	W117^{0}31'53.0"
		End	N47^{0}49'43.91"	W117^{0}37'46.21"	N47^{0}49'57.41"	W117^{0}37'27.34
Lower Lake	6.2 miles	Start	N47^{0}50'32.46"	W117^{0}43'25.42"	N47^{0}50'0.67"	W117^{0}43'56.51"
		End	Long Lake Dam			

All coordinates, NAD 1983

The species collected include largemouth bass (*Micropterus salmoides*) large-scale sucker (*Catostomus macrocheilus*), mountain whitefish (*Prosopium williamsoni*), smallmouth bass (*Micropterus dolomieui*), and yellow perch (*Perca flavescens*). Due to habitat requirements, mountain whitefish are only found in the upper lake. Rainbow trout are one of the species of concern in the upper river, and the joint Ecology/DOH/Spokane Regional Health District health advisory addresses this species. However, rainbow trout are not found in Long Lake. Brown trout were targeted as a surrogate species, but only four brown trout were caught during the survey. Thus, a field decision was made to collect smallmouth bass instead. Based on discussions with WDFW, the species collected represent the commonly caught, edible fish in Long Lake.

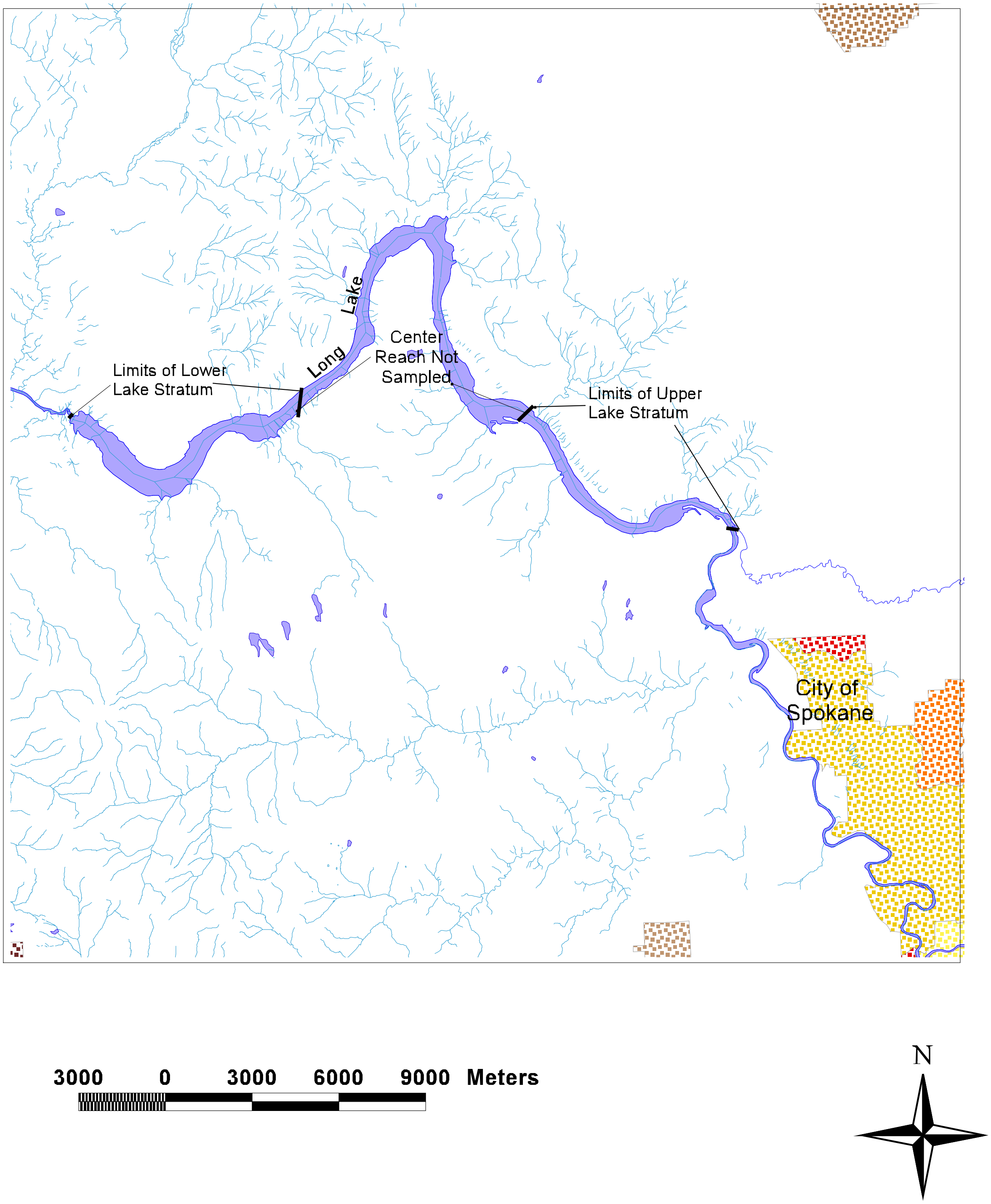

Figure 1. Long Lake sampling segments.

Thirty fish from each species were targeted. It was not possible to obtain the target for all species. Collection techniques included electroshocking, gill nets, and Fyke nets. At the end of each sampling effort, fish were weighed and their length measured. Some fish weights and lengths were not recorded, as their heads required removal to extract the fish from the gill and Fyke nets. Fish were wrapped in aluminum foil, placed in polyethylene bags, and held on ice. All fish were assigned a unique identification number and letter code, the latter corresponding to their lake strata. Fish were transported to Ecology headquarters storage facilities and frozen at -18^0 C. Composite samples were randomly created post hoc. They ranged in size from four to ten fish per composite (Table 2), with the same number of fish in each species composite. The size range of total fish within each composite is also provided in Table 2.

Table 2. Composite sizes for upper and lower Long Lake fish fillets by species, with whole sucker size ranges.

Upper lake species	Size range (mm)	Composite size	Lower lake species	Size range (mm)	Composite size
Largemouth Bass	312-473	10 fish	Largemouth Bass	289-493	4 fish
Largescale Sucker	265-561	10 fish	Largescale Sucker	259-504	9 fish
Mountain Whitefish	205-325	6 fish	--	--	--
Smallmouth Bass	223-460	5 fish	Smallmouth Bass	240-367	5 fish
Yellow Perch	198-248	10 fish	Yellow Perch	181-275	11 fish
Largescale Sucker, whole	437-485	na	Largescale Sucker, whole	449-509	na

Fish were randomly assigned to one of the three composites per strata. A random number generator and each fish's unique identifier were used. Aging structures were removed from all fish prior to tissue preparation. These include: otoliths, scales, opercles, and/or spines depending on the species. The structures were sent to WDFW for aging and their own population studies. WDFW provided an overall age assessment for each fish as a single number, despite the multiple aging structures analyzed.

Sample Preparation

Preparation of the tissue samples followed USEPA (1997 and 2000) guidance. The techniques described below were used to minimize the potential for sample contamination and cross-contamination. All resection and homogenizing used only non-corrosive stainless steel implements. Preparers wore non-talc polyethylene or nitrile gloves and worked on aluminum foil covering a polyethylene cutting board. Gloves and foil were changed between composite samples. The cutting board and knives were cleaned using Liquinox® detergent and hot tap water, followed by rinses with 10% nitric acid, deionized water, and pesticide grade methanol. Implements were air dried in a fume hood before use.

Fish were partially thawed to remove their aluminum foil wrapping and aging structures. Prior to dissection, fish were rinsed with tap water followed by deionized water. For fillets, the fish were descaled but skins left on. The entire fillet tissue was removed and mixed at least three times in a Kitchenaide meat grinder before weighing out an aliquot for compositing. Fillets minimized the inclusion of scales or bones, to avoid biasing the metals results.

Homogenized tissues were placed in glass jars with Teflon lids; precleaned to USEPA (1990a) quality assurance/quality control (QA/QC) specifications. All composite samples, by species, contained equal numbers of fish. Each composite sample contained approximately 100 grams of tissue, comprised of an equal mass from each individual fillet. For each species, the individual fish were pooled as shown in Table 2.

Two whole suckers from the lower lake strata and three whole suckers from the upper lake strata were analyzed independently and individually for lead, zinc, cadmium, and PCB Aroclors. Whole fish were homogenized in a Hobart commercial meat grinder and included all scales, bones, slime, and associated liquids. The meat grinder was cleaned between samples with Liquinox®, nitric acid, and methanol using the same procedures described above for cutting boards and knives.

Laboratory Preparation and Analysis

The fish tissues were analyzed using the methods shown in Table 3. There were no significant analytical issues reported; case narratives are included in the Appendix. Ecology's Manchester Environmental Laboratory analyzed the metals, Aroclors, and percent lipids. Axys Analytical Services of Sidney, British Columbia analyzed the PCB congeners.

Table 3. Preparation and analytical methods used for Long Lake fish tissues.

Analyte	Preparation Method	Analytical Method
Cadmium , Lead, Zinc	Microwave digestion, EPA Method 3051A (USEPA, 1994a)	ICP/MS, EPA Method 200.8 (USEPA, 1994b)
Mercury	Aqua regia + oxidizing permanganate (MEL, 2001)	CVAA, EPA Method 245.6 (USEPA, 1991)
Aroclors	Soxhlet extraction (USEPA, 1996)	GC/ECD, EPA Method 8082 (USEPA, 2000)
PCB Congeners	Soxhlet extraction (USEPA, 1996)	GC/ECD, EPA Method 1668A (USEPA, 1999)
Percent Lipids	NA	Gravimetric (USEPA, 1990b)

Data quality was evaluated using matrix spikes and spike duplicates, certified reference materials, and field duplicates.

Data Quality Assessment

The laboratory results were compared to the precision goals specified in the Quality Assurance (QA) Project Plan (Jack and Roose, 2002). Laboratory duplicates of fillets were compared to the QA Project Plan's relative standard deviation goals (RSD). These results are shown in Table 4.

Table 4. Precision estimates relative to Quality Assurance Project Plan goals.

Analyte	Precision Goal (RSD)	Number of Duplicates	Average RSD from Duplicates
Zinc	10%	4	8.5%
Cadmium	10%	4	0%[a]
Lead	10%	4	0%[a]
Mercury	10%	4	7.9%
Sum of Detected Aroclors	50%	6	14.0%
Total PCB Congeners	50%	4	6.02%
Percent lipids	10%	4	19.0%

[a] Cadmium and lead were not detected in any replicate or duplicate sample.

Precision for all the metals and the organic analytes are within the QA Project Plan goals. Unfortunately, precision for the percent lipids analysis exceeds the QA Project Plan goal. Samples may not have been completely homogeneous for lipids, which may contribute to this problem. Lipids are often differently distributed in fish, with greater concentrations near the tail and abdomen. Fortunately, duplicate results for metals, apparent Aroclors, and PCB congeners suggest that the potentially poor homogenization has not influenced their data.

For largescale suckers, lipid concentrations are similar to results from 1999 (Johnson, 2000). Other species results appear lower relative to historic concentrations. Differences may be due to bias in these or the 1999 data or due to actual variation in fish from different river segments, or from seasonal variation. Lipid data are not directly part of health risk determinations; rather they are to further our understanding of why particular samples might be particularly high or low in organic contaminants. Thus, these data are deemed useable for their intended purposes in the discussion, despite their low precision.

Bias was estimated using recoveries from matrix spikes, matrix spike duplicates, and recovery from certified reference materials (CRM). Table 5 outlines the bias estimates.

Table 5. Bias estimates relative to Quality Assurance Project Plan goals.

Analyte	QA Project Plan Goal	Average Spike Recovery	Average Recovery of Reference Tissue	Average Recovery of Spikes and Reference Material Combined	Overall Bias Estimate
Lead	±10%	107.3%	92.9%	103.7%	+3.7%
Zinc	±10%	78.5%	105.8%	85.3%	-14.7%
Cadmium	±10%	94.3%	111.0%	96.7%	-3.3%
Mercury	±10%	84.0%	83.0%	83.5%	-16.5%
Aroclors	Not estimated, see text for discussion.				
PCB Congeners	Not estimated, see text for discussion.				
% lipids	Not estimated, see text for discussion.				

Matrix spike recoveries of reference tissues for lead and cadmium suggest no systematic bias in the analysis. For zinc, the recoveries are highly variable, with the minimum of negative 31%, which is not theoretically plausible. Removing this single negative recovery value estimates bias at only +3%, which is within the QA Project Plan goals. DORM-2, a CRM of dogfish (*Squalus acanthias*) muscle supplied by the National Research Council of Canada, was analyzed concurrently with the tissue samples. Lead and zinc CRM results were within the 95 percent confidence limits specified by the supplier.

For unknown reasons, the mercury results appear biased low about 15 to 20 percent. The results of the mercury analysis on the CRM were also outside the 95 percent confidence limits. This suggests that the low bias is not due to the exceedance of the mercury holding time.

Aroclor analysis met all QC limits for surrogates, blanks, calibration standards, duplicates, matrix spikes, and lab control samples. The tissue extracts did not exactly match standard Aroclor patterns due to weathering, partitioning, chemical transformation, and preferential bioaccumulation. This suggests that analytical bias is probably low, but interpretive, pattern matching by the analyst may contribute some bias.

For PCB congeners, the initial laboratory results inadvertently failed to aggregate two coeluting congeners. Quality assurance review by Ecology recognized this error, and revised data were distributed. The concentrations determined in the mussel tissue (*Mytilus edulis*) (SRM 2977) were outside the certified values. However, the OPR and other QC parameters show that the method was in specifications, and results should not be significantly affected. An alternative SRM (Cod liver oil – 1588a) was analyzed and found to be within certified values. The deviation of SRM 2977 from the certified values is believed due to sample heterogeneity. Recoveries of congeners from SRM 1588a were between 87 and 105 percent. The quality control limits in USEPA method 1668A are 50 to 150 percent, so these SRM recoveries are well within the method limits.

Some analytes required dilution due to high concentrations exceeding the calibrated range of the instrument. Other congeners have been flagged due to an excursion of the ion abundance beyond EPA method criteria or when positive identification of the analyte cannot be made through visual inspection of the peak shape. Data flagged "NJ" should be considered with caution, because they did not meet quantification criteria. All other method parameters were within method performance criteria, and the data are considered acceptable for use.

Lipids were not detected in method blanks. However, there are no CRMs or other calibration standards for lipids, so it is not possible to estimate bias for this analyte.

Definitively segregating overall bias from random error is not possible, given the small number of matrix spikes and certified tissues analyzed. However, mercury appears to be the only analyte with moderate bias. All of the analytical data are considered useable for their intended purposes.

Results

Fish Weights and Ages

Table 6 describes the mean ages and weights of fish within each composite sample. Yellow perch and mountain whitefish were of similar size and age across all composites. The lower lake largemouth bass composites were formed from only four individuals, and this is reflected in their variable mean ages. The upper lake ten fish largemouth bass composites have more uniform mean ages. For individual fish, their ages range widely. Most perch and whitefish were three or four years old, while most largescale suckers were between 15 and 20 years old. Most largemouth bass were between five and 12 years old. Variation in ages between composites shown in Table 6 may also be related to the number of individuals within a composite.

Table 6. Fish composite samples with mean fish age, length, and weight.

Upper Lake				Lower Lake			
Species, and Composite number	Mean fish age (years)	Mean fish length (mm)	Mean fish weight (gms)	Species, and Composite number	Mean fish age (years)	Mean fish length (mm)	Mean fish weight (gms)
LMB 1	6.7	399	1002	LMB 1	9.0	436	1309
LMB 2	6.9	400	1004	LMB 2	7.5	395	1049
LMB 3	6.2	394	887	LMB 3	6.8	368	699
LRS 1	6.0	504	626	LRS 1	4.7	356	518
LRS 2	9.4	457	946	LRS 2	8.9	452	905
LRS 3	16.3	492	1208	LRS 3	10.6	479	1064
LRS Whole 1	14	475	1019	LRS Whole 1	24	449	831
LRS Whole 2	10	485	1064	LRS Whole 2	13	462	990
LRS Whole 3	7	437	868	SMB 1	4.2	308	543
MHF 1	3.5	274	181	SMB 2	3.2	285	238
MHF 2	3.5	260	161	SMB 3	3.2	264	205
MHF 3	3.3	262	165	YPC 1	3.2	219	123
SMB 1	3.2	269	294	YPC 2	3.0	221	133
SMB 2	4.0	324	545	YPC 3	3.0	229	136
SMB 3	3.4	280	349	-	-	-	-
YPC 1	3.2	238	171	-	-	-	-
YPC 2	3.2	229	151	-	-	-	-
YPC 3	3.0	234	167	-	-	-	-

LMB = Largemouth Bass
LRS = Largescale Sucker
MHF = Mountain Whitefish
SMB = Smallmouth Bass
YPC = Yellow Perch

Metals

Tables 7 and 8 illustrate the concentrations of metals within both strata for these various tissues analyzed. For both the upper and lower lake, lead and cadmium were only detected in whole suckers. For some tissues, only one sample was analyzed. Zinc was the most variable metal, with a suitable frequency of detection. Concentrations of zinc are shown by species and strata in Figure 2.

Table 7. Upper Long Lake: Minimum, maximum, and mean concentrations of metals in fish tissue by species. All concentrations are µg/Kg, wet.

Species	Number fish in composite	Mean composite or actual age	Analyte	Mean	Minimum	Maximum
Largemouth Bass, fillet	10	6.6	Zinc	7,676	7,720	7,930
			Lead	100 U	100 U	100 U
			Cadmium	100 U	100 U	100 U
			Mercury	99.7	81.1	113
Largescale Sucker, fillet	10	10.6	Zinc	14,083	10,450	15,900
			Lead	100 U	100 U	100 U
			Cadmium	100 U	100 U	100 U
Largescale Sucker, whole	1	10	Zinc	22,300	Only one sample was analyzed for metals	
			Lead	730		
			Cadmium	280		
Mountain Whitefish, fillet	6	3.3	Zinc	11,700		
			Lead	100 U		
			Cadmium	100 U		
Smallmouth Bass, fillet	5	3.2	Zinc	5,910		
			Lead	100 U		
			Cadmium	100 U		
Yellow Perch, fillet	10	3.2	Zinc	11,900		
			Lead	100 U		
			Cadmium	100 U		

U = Not detected at the detection limit shown

Table 8. Lower Long Lake: Minimum, maximum, and mean concentrations of metals in fish tissue by species. All concentrations are µg/Kg, wet.

Species	Number fish in composite	Mean composite or actual age	Analyte	Mean	Minimum	Maximum
Largemouth Bass, fillet	4	7.6	Zinc	8,663	7,430	9,460
			Lead	100 U	100 U	100 U
			Cadmium	100 U	100 U	100 U
			Mercury	86.6	65.4	108.0
Largescale Sucker, fillet	9	8.0	Zinc	11,533	9,400	15,000
			Lead	100 U	100 U	100 U
			Cadmium	100 U	100 U	100 U
Largescale Sucker, whole	1	13.0	Zinc	32,200	Only one sample was analyzed for metals	
			Lead	500		
			Cadmium	420		
Smallmouth Bass, fillet	5	3.5	Zinc	7,610		
			Lead	100 U		
			Cadmium	100 U		
Yellow Perch, fillet	11	3.1	Zinc	9,110		
			Lead	100 U		
			Cadmium	100 U		

U = Undetected at the reported concentration

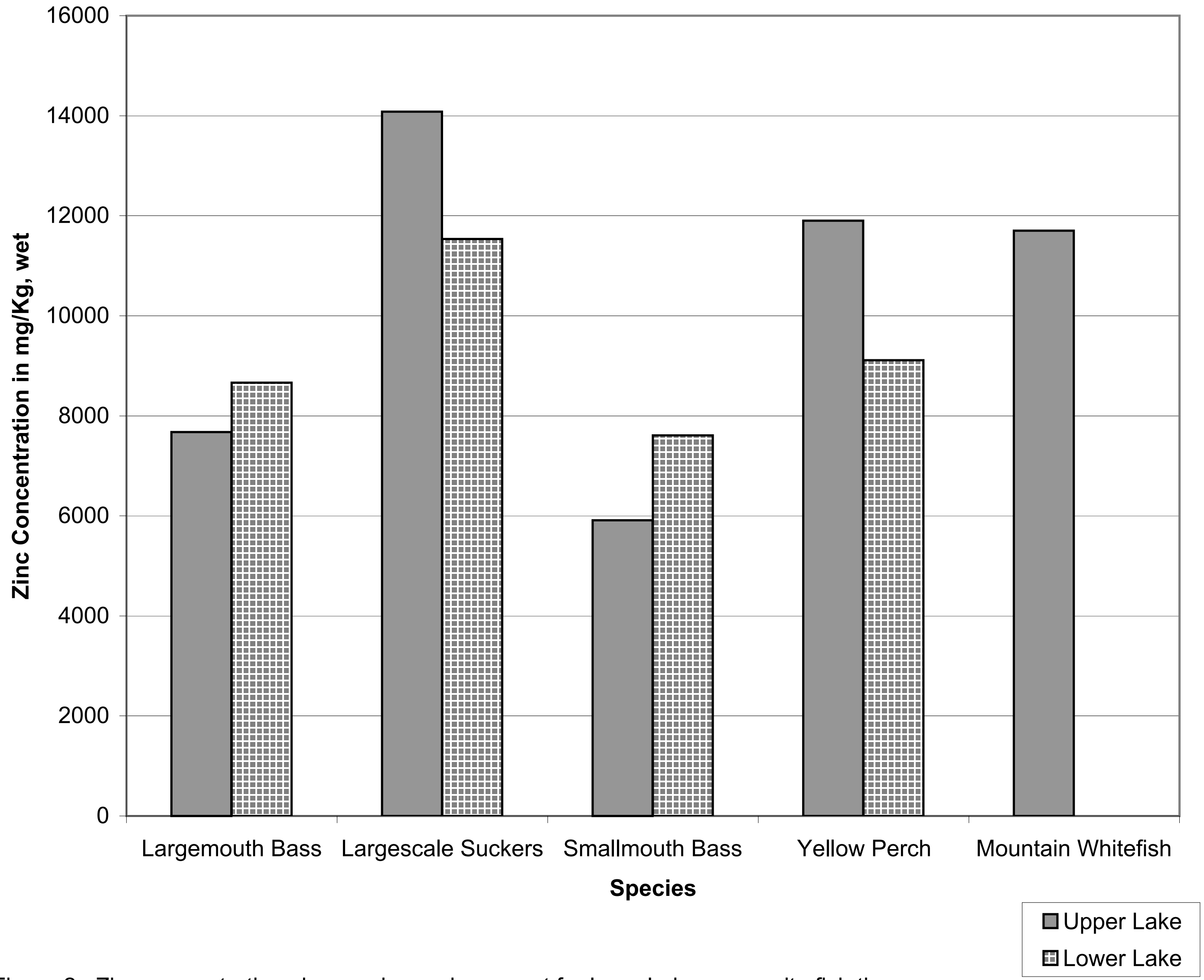

Figure 2. Zinc concentrations by species and segment for Long Lake composite fish tissue samples.

Polychlorinated Biphenyls

Total PCB congener concentrations, the sum of apparent Aroclors, and percent lipids are presented in Tables 9 and 10. When available, the sum of detected congeners is also reported. Apparent Aroclor 1016, 1221, 1232, and 1242 were not detected in any sample. Figure 3 illustrates the variation of PCB apparent Aroclor concentrations by species and strata.

Table 9. Upper Long Lake: Total PCB concentrations of apparent Aroclor 1016, 1221, 1232, 1242, 1248, 1254, and 1260 in fish tissue by species.

Species and sample number	Mean fish age (years)	Sum of detected congeners (ug/Kg)	Lipid-based concentration µg Σcongeners/ g-lipid	Sum of detected apparent Aroclors (µg/Kg wet)	Sum of apparent Aroclors with ½ detection limit used for ND (µg/Kg wet)	Percent lipids
LMB 1	6.7	89.9	118.3	72.0	94.0	0.76
LMB 2	6.9	40.3	122.1	38.8	60.8	0.33
LMB 3	6.2	43.5	71.3	39.2	59.2	0.61
LRS 1	6.0	131.0	71.6	112.0	168.0	1.83
LRS 2	9.4	152.0	80.4	132.0	154.0	1.89
LRS 3	16.3	144.0	92.9	86.0	108.0	1.55
LRS Whole 1	14	-	-	164.0	208.0	1.46
LRS Whole 2	10	-	-	336.0	438.0	4.02
LRS Whole 3	7	-	-	294.0	338.0	5.52
MHF 1	3.5	85.1	45.3	89.0	111.0	1.88
MHF 2	3.5	68.3	44.6	60.0	82.0	1.53
MHF 3	3.3	80.9	44.2	70.0	92.0	1.83
SMB 1	3.2	-	-	32.4	54.4	0.46
SMB 2	4.0	-	-	54.0	76.0	0.69
SMB 3	3.4	-	-	39.5	61.5	0.43
YPC 1	3.2	-	-	0.0	38.5	0.26
YPC 2	3.2	-	-	0.0	38.5	0.35
YPC 3	3.0	-	-	0.0	38.5	0.33

LMB = Largemouth Bass
LRS = Largescale Sucker
MHF = Mountain Whitefish
SMB = Smallmouth Bass
YPC = Yellow Perch
- = not analyzed

Table 10. Lower Long Lake: Total PCB concentrations of Aroclor 1016, 1221, 1232, 1242, 1248, 1254, and 1260 in fish tissue by species.

Species, and sample number	Mean fish age (years)	Sum of detected congeners (ug/Kg)	Lipid-based concentration µg Σcongeners/ g-lipid	Sum of detected apparent Aroclors (µg/Kg wet)	Sum of apparent Aroclors with ½ detection limit used for ND (µg/Kg wet)	Percent lipids
LMB 1	9.0	70.1	170.9	47.0	69.0	0.41
LMB 2	7.5	65.0	104.0	64.0	86.0	0.62
LMB 3	6.8	101.0	31.5	57.0	79.0	0.32
LRS 1	4.7	68.1	46.0	63.0	85.0	1.48
LRS 2	8.9	122.0	62.4	100.0	122.0	1.86
LRS 3	10.6	147.0	76.6	112.0	134.0	1.92
LRS Whole 1	24	-	-	393.0	447.0	2.54
LRS Whole 2	13	-	-	321.0	375.0	4.1
SMB 1	4.2	-	-	31.0	58.5	0.23
SMB 2	3.2	-	-	0.0	38.5	0.09
SMB 3	3.2	-	-	33.0	60.5	0.17
YPC 1	3.2	-	-	0.0	35.0	0.13
YPC 2	3.0	-	-	0.0	38.5	0.17
YPC 3	3.0	-	-	0.0	35.0	0.18

LMB = Largemouth Bass
LRS = Largescale Sucker
SMB = Smallmouth Bass
YPC = Yellow Perch
- = not analyzed

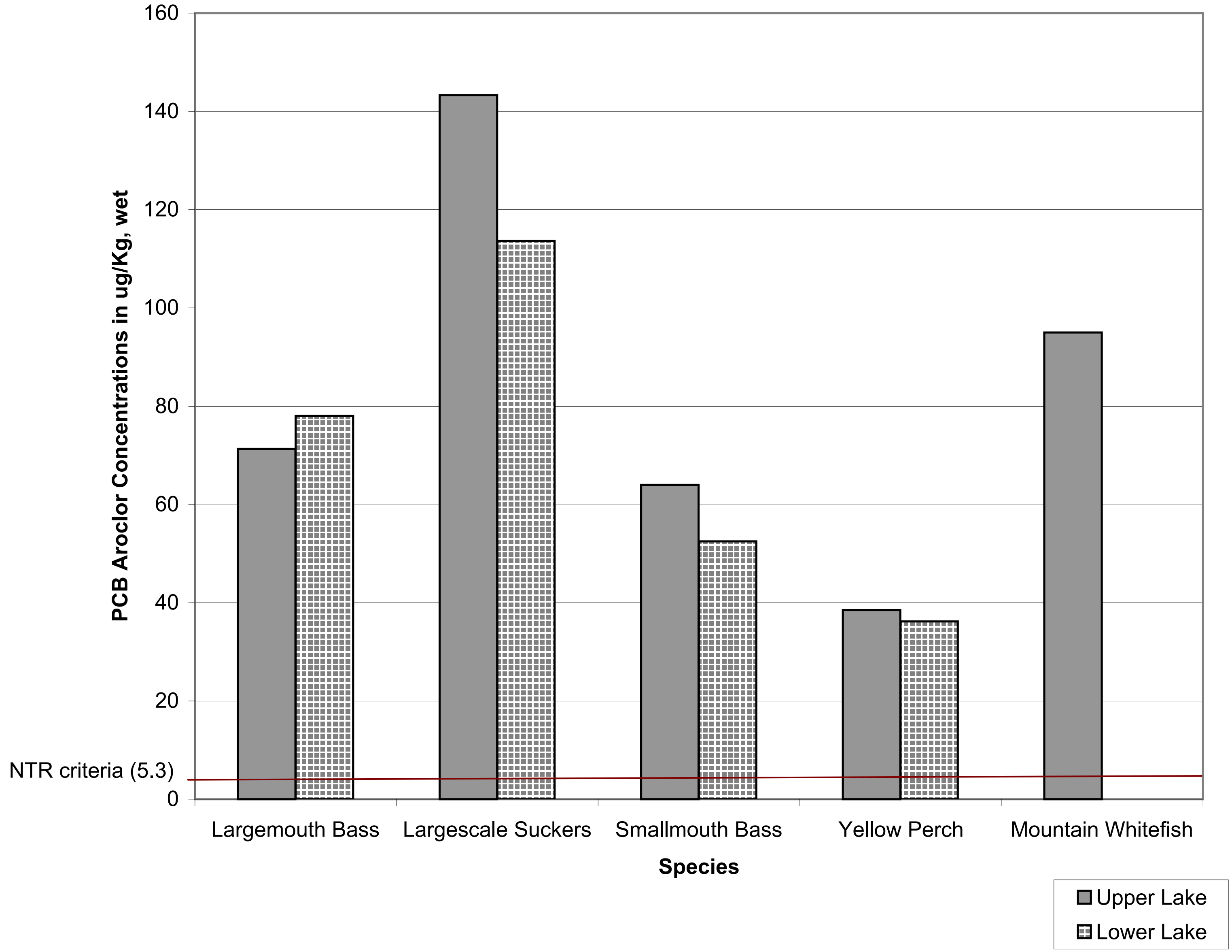

Figure 3. Mean PCB Aroclor concentrations by species and segment for Long Lake composite fish tissue samples. One-half detection limit used for non-detect Aroclors.

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Discussion

Metals

Comparison to Criteria

Several screening or regulatory thresholds are available for comparison with the Long Lake fish tissue data. These thresholds have been established by the USEPA for either regulatory purposes or as conservative risk screening values. While these thresholds do not specify the site-specific human health risk, they are used herein for comparative purposes. The National Toxics Rule (NTR) (40 CFR Part 131.36) establishes surface water criteria that are used in developing National Pollutant Discharge Elimination System (NPDES) permit limits. Concentrations applicable to fish tissue can be derived from these values by using USEPA's bioaccumulation factors. These derived concentrations are used by Ecology to define impaired waterbodies under the 303d listing process of the federal Clean Water Act.

Cadmium, lead, and zinc are not listed in the NTR; however, various USEPA regions have developed tissue screening levels for some of these constituents. Region III has a set of widely used risk based screening concentrations (RBCs) which includes values for cadmium (1,400 µg/Kg) and zinc (410,000 µg/Kg) in tissues. All samples collected from Long Lake were below these thresholds by several orders of magnitude.

There are no regulatory or risk based threshold values for lead in tissue. However, the World Health Organization (2002) has a draft value of 200 µg/Kg for lead in fish muscle tissue. None of the fillet samples exceeded this value.

For mercury, the calculated NTR criterion is 825 µg/Kg. The maximum mercury concentration detected in largemouth bass fillets was 113 ug/Kg. All samples were at least seven times below the NTR criterion of 825 µg/Kg. The mercury analysis was significantly over the holding time of 28 days, but these data were collected for screening purposes only. The low levels detected suggest that future analyses need not focus on this analyte.

In summary, none of the composite samples exceeded available tissue guidelines for metals.

Polychlorinated Biphenyls (PCBs)

Upper Lake

Table 9 illustrates the good agreement between total detected congeners and the sum of detected apparent Aroclors. The use of one-half the detection limit when summing apparent Aroclors produced slightly to moderately conservative sums when compared to the total congeners results which did not use one-half of their detection limits. The use of the total apparent Aroclors with one-half the detection limit is consistent with established risk assessment guidelines (USEPA, 1991), and these values have been used for comparisons with NTR limits below.

The USEPA has published revised standards for PCBs in the NTR (Federal Register, 1999) for total PCBs. The derived NTR value for fish tissues is 5.3 µg/Kg. The bioconcentration factor used to derive this value is 31,200 L/Kg as described in the USEPA NTR PCB rulemaking (Federal Register, 1999). All samples from the upper lake strata exceeded NTR PCB standards (Table 11).

Table 11. Upper Long Lake: Total apparent Aroclors in fillets relative to National Toxics Rule (NTR) tissue criteria by composite. One-half the detection limit was used when summing non-detected apparent Aroclors.

Species and sample number	Sum of apparent Aroclors with ½ detection limit used for ND (µg/Kg wet)	NTR criterion (µg/Kg wet)	Sum:NTR Aroclor criterion ratio	Sum:NTR mean ratio
LMB 1	94.0	5.3	17.7	13.4
LMB 2	60.8	5.3	11.5	
LMB 3	59.2	5.3	11.2	
LRS 1	168.0	5.3	31.7	27.0
LRS 2	154.0	5.3	29.0	
LRS 3	108.0	5.3	20.4	
LRS Whole 1	208.0	5.3	39.2	61.8
LRS Whole 2	438.0	5.3	82.6	
LRS Whole 3	338.0	5.3	63.7	
MHF 1	111.0	5.3	20.9	17.9
MHF 2	82.0	5.3	15.5	
MHF 3	92.0	5.3	17.3	
SMB 1	54.4	5.3	10.3	12.1
SMB 2	76.0	5.3	14.3	
SMB 3	61.5	5.3	11.6	
YPC 1	38.5*	5.3	7.3	7.3
YPC 2	38.5*	5.3	7.3	
YPC 3	38.5*	5.3	7.3	

* = apparent Aroclors not detected

Apparent Aroclors 1016, 1221, 1232, and 1242 were not detected, but detection limits were elevated above derived NTR criteria. The detected apparent Aroclors required diluting the extractions, elevating the detection limits of the remaining Aroclors. Apparent Aroclors were not detected in yellow perch, but detection limits were elevated above derived NTR criteria for unknown reasons.

For the purposes of the following discussion, r^2 values greater than 0.5 to 0.6, depending on the parameter, have been considered to illustrate potentially valuable relationships. When r^2 values are below 0.5, the predictiveness of the relationship has been considered not worthy of substantial additional investigation or discussion.

Increasing lipids content is a useful predictor of an increase in the total apparent Aroclors (Figure 4) (r^2=0.74). While insufficient data points are available to evaluate this relationship by species, the general trend appears the same across species. The trend is most discernable in species with higher apparent Aroclor concentrations. When the sum of detected congeners is converted to a fish lipid basis (µg-PCB/g-lipid)(Table 9), the concentrations are within a factor of 3 and relatively consistent within and between species. This suggests similar PCB bioaccumulation processes regardless of habitat, trophic level, or species guild.

Mean age of the composite is a less useful predictor of total apparent Aroclor concentrations (Figure 5) (r^2=0.21). An insufficient number of individual fish were analyzed to further evaluate this relationship. The relationship between mean fish age within a composite and percent lipid content was positive as expected, although not particularly predictive. In upper Long Lake, the r^2 value was only 0.27. Fish length, one of the most easily discernable measures to a fisherman, was not clearly related to total apparent Aroclor concentrations across species (Figure 6). Length may be a useful predictor within a species, as suggested by the smallmouth bass samples (Figure 6); however, more extensive sampling would be needed to adequately describe any such relationship.

Lower Lake

As in the upper lake, there is good agreement between total detected congeners and the sum of detected apparent Aroclors (Table 10). The use of one-half the detection limit when summing apparent Aroclors produced slightly to moderately conservative sums when compared to the total congeners results which did not use one-half of their detection limits. The use of the total apparent Aroclors with one-half the detection limit is consistent with established risk assessment guidelines (USEPA, 1991), and these values have been used for comparisons with NTR limits below. In the lower lake strata, whole suckers had the highest apparent Aroclor:criteria quotients (>71) (Table 12).

As in the upper lake strata, apparent Aroclors 1016, 1221, 1232, 1242 were not detected, but detection limits were elevated above NTR criteria. The detected apparent Aroclors required diluting the extractions, elevating the detection limits of the remaining apparent Aroclors. Aroclors were not detected in yellow perch.

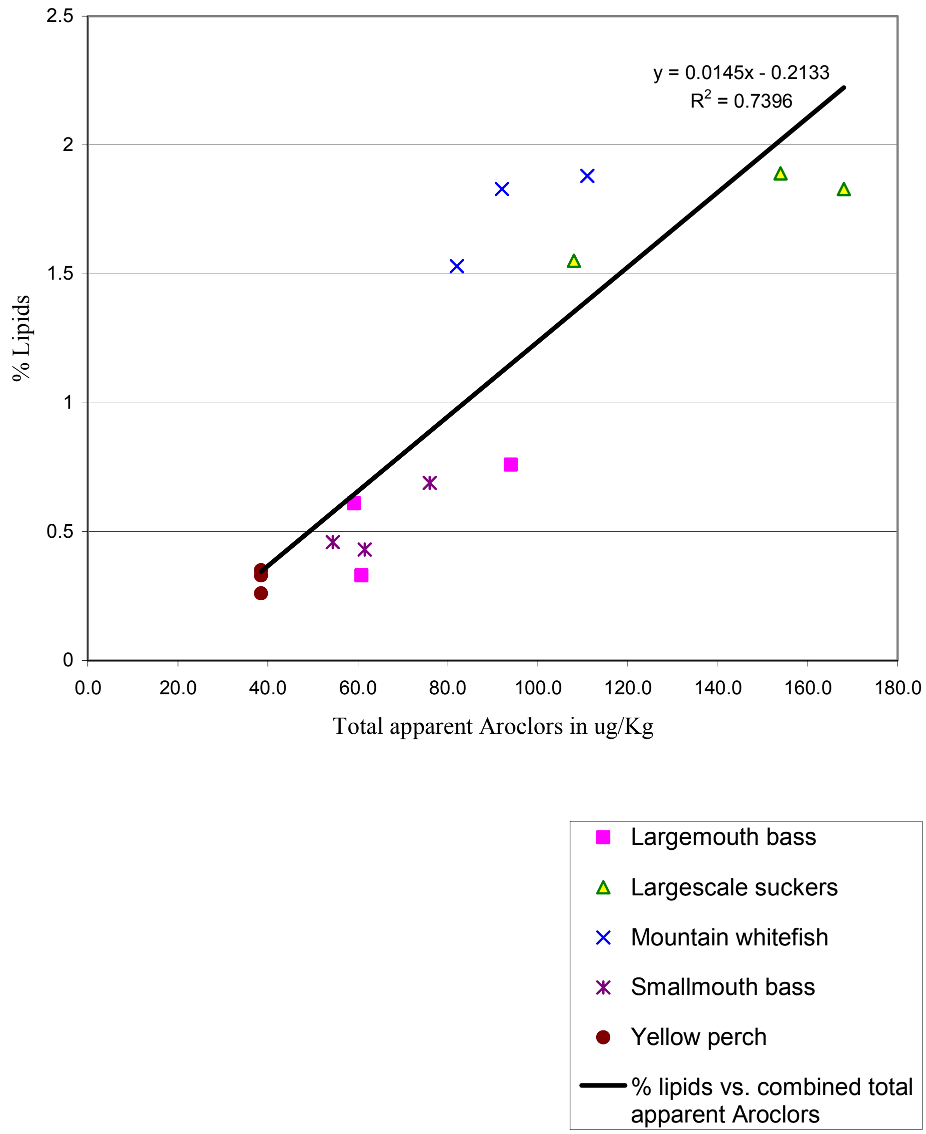

Figure 4. Percent lipids vs. total apparent Aroclors in upper Long Lake composite fish tissue samples.

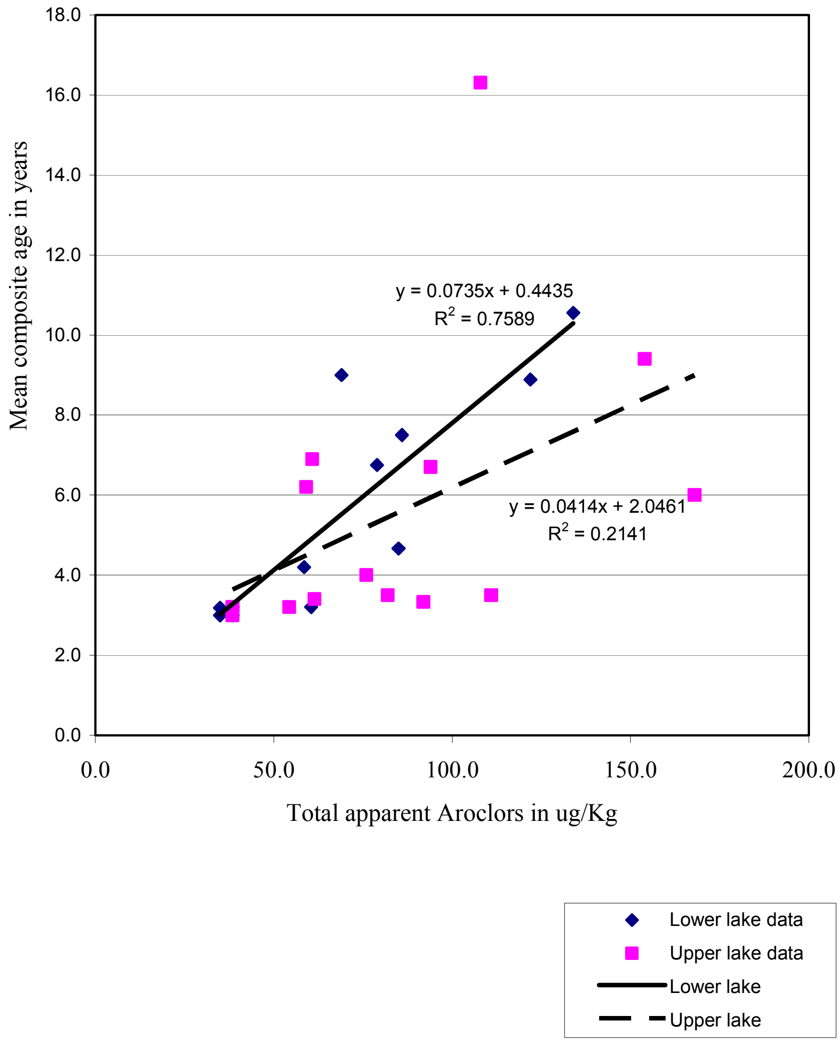

Figure 5. Mean composite age vs. total apparent Aroclors in Long Lake composite fish tissue samples.

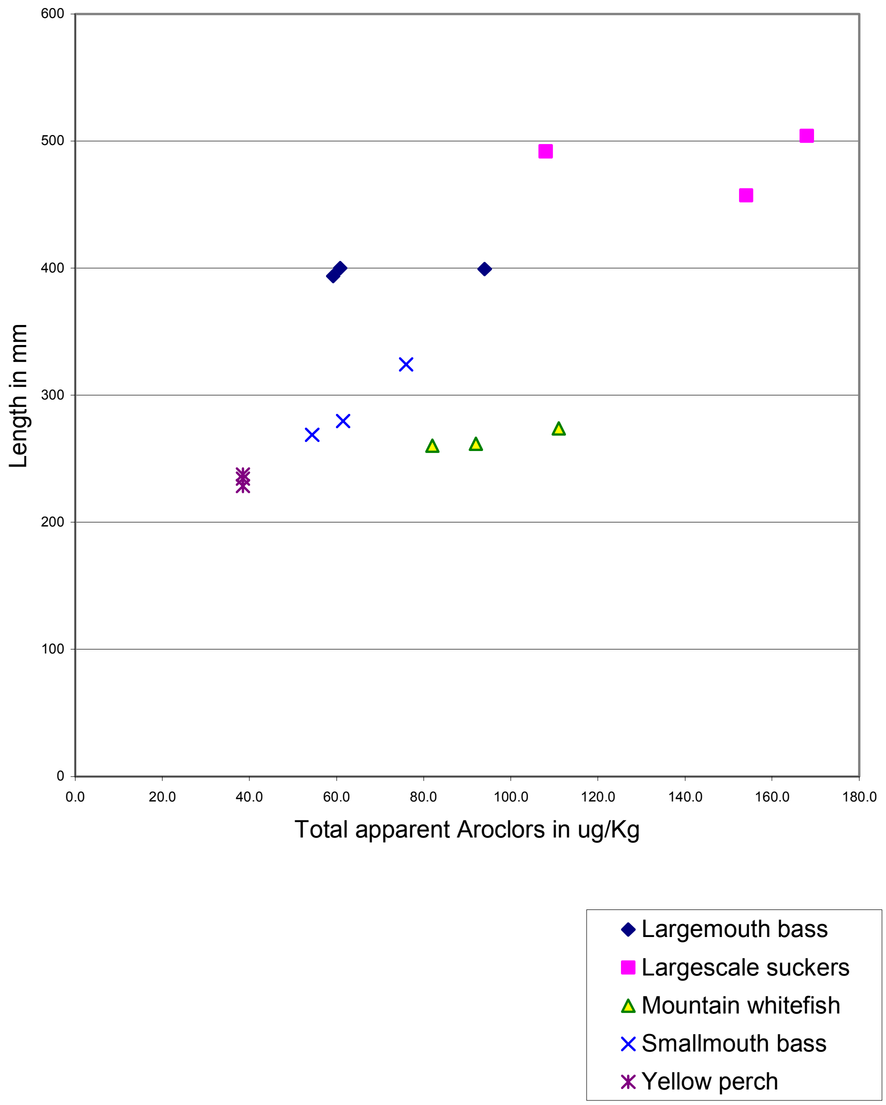

Figure 6. Total apparent Aroclor concentration by species compared to arithmetic mean fish length for upper Long Lake composite fish tissue samples.

Table 12. Lower Long Lake: Total apparent Aroclors in fillets relative to National Toxics Rule (NTR) tissue criteria by composite. One-half the detection limit was used when summing non-detected apparent Aroclors.

Species, and sample number	Sum of apparent Aroclors with ½ detection limit used for ND (µg/Kg wet)	NTR criterion (µg/Kg wet)	Sum:NTR Aroclor criterion ratio	Sum:NTR mean ratio
LMB 1	69.0	5.3	13.0	14.7
LMB 2	86.0	5.3	16.2	
LMB 3	79.0	5.3	14.9	
LRS 1	85.0	5.3	16.0	21.4
LRS 2	122.0	5.3	23.0	
LRS 3	134.0	5.3	25.3	
LRS Whole 1	447.0	5.3	84.3	77.5
LRS Whole 2	375.0	5.3	70.7	
SMB 1	58.5	5.3	11.0	9.9
SMB 2	38.5*	5.3	7.3	
SMB 3	60.5	5.3	11.4	
YPC 1	35.0*	5.3	6.6	6.8
YPC 2	38.5*	5.3	7.3	
YPC 3	35.0*	5.3	6.6	

* = apparent Aroclors not detected

Increasing lipids content reliably predicts the increase in total apparent Aroclor concentrations (r^2=0.82) (Figure 7). The r-squared value is slightly higher than in the upper lake strata, probably due to random variation in associated analytical results. The lack of precision in the percent lipids data (Table 3) may explain the variability in this relationship.

When the sum of detected congeners is converted to a fish lipid basis (µg-PCB/g-lipid) (Table 10), the concentrations vary by a factor of 5, with the principal variation within the largemouth bass composites. This may be a function of the more variable lipid data. Examination of the Aroclor and congener data relative to the lipid data suggests the lipid normalized contaminant fraction is more highly variable (Table 10).

Since the lower lake composites had four largemouth bass per composite compared to the ten per composite in the upper lake (Table 2), the change in sample size probably explains the variability in this species PCB results. For largescale suckers, the fish lipid-based PCB concentrations were still highly variable relative to the upper lake. These were composites of nine fish relative to the ten fish composites from the upper lake. This suggests differences in biotic PCB processes within lower lake largescale suckers instead of changes in ample size. These differences might be due to patchy contamination, fish habitat preferences, or some other environmental or chemical variable.

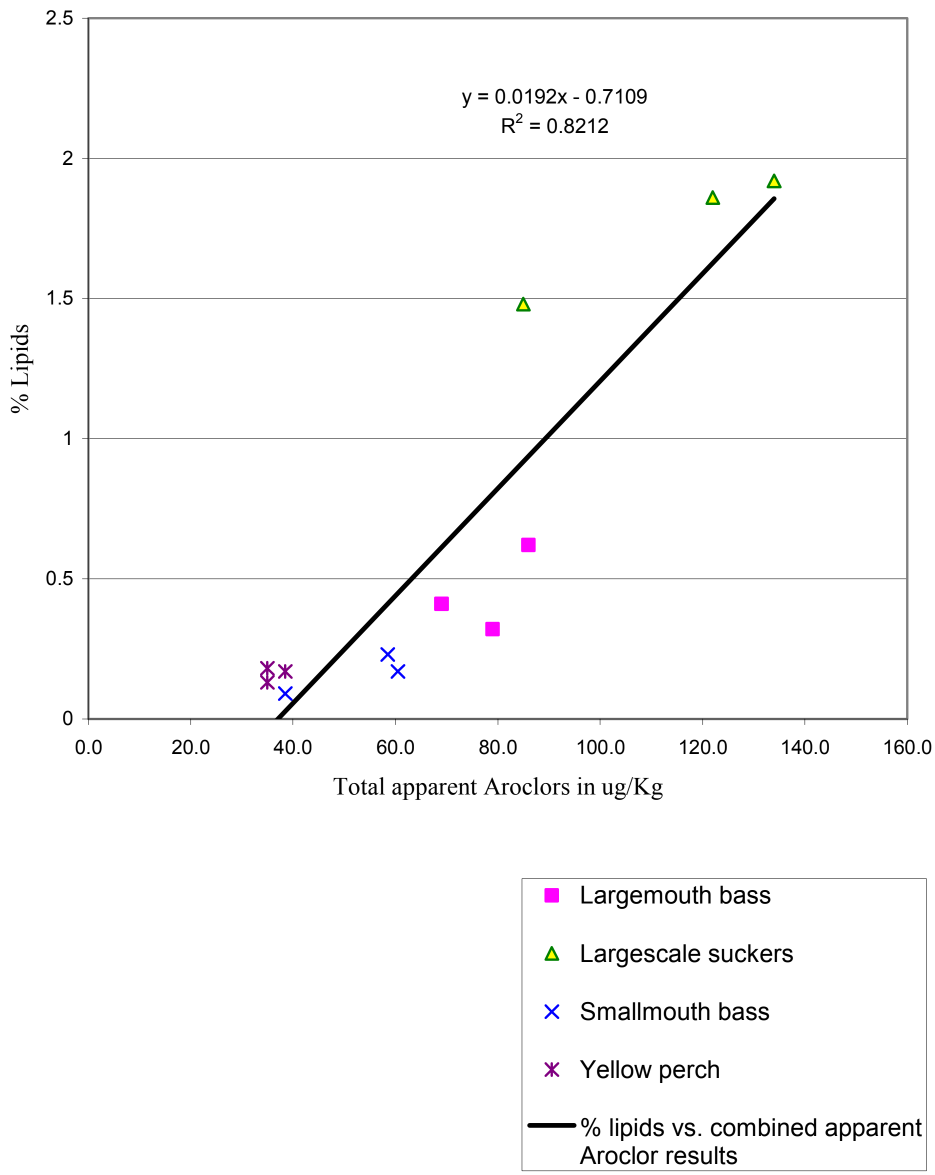

Figure 7. Percent lipids vs. total apparent Aroclors in lower Long Lake composite fish tissue samples.

For the lower lake, the relationship of age to total apparent Aroclor concentrations yielded an r-squared value of 0.76 (Figure 5). This r^2 value is higher than that observed in the upper lake (r^2=0.21). The relationship between mean fish age within a composite and percent lipid content was positive as expected, although not especially predictive (r^2 = 0.49). Fish length was possibly related to total apparent Aroclor concentrations (Figure 8) in largescale suckers. The upper lake data suggested a relationship between smallmouth bass and apparent Aroclor concentrations.

The length-apparent Aroclor relationships are not consistent by species between strata. Additionally, these potential relationships are currently suggested by only three data points. Thus, extensive use of these relationships is currently not appropriate. If future studies wished to further evaluate the use of length as a surrogate for apparent Aroclor concentrations, more information on individual fish should be collected. The absence of a clear relationship, in either lake strata, by species may be due to the physical averaging of results through compositing. If length is of interest in guiding possible consumption advisories or other monitoring efforts, individual fish should be analyzed to accurately determine its predictiveness first.

Historic Data

For metals, historic 1993 Long Lake data are available from Johnson et al. (1994). Only one composite of five whole largescale suckers was analyzed, and lead was estimated at 740 µg/Kg. This value compares to the 730 µg/Kg and 500 µg/Kg detected in the upper and lower lake strata, respectively, during this study. The other historic largemouth bass and mountain whitefish five fillet composite results for lead were nondetect. This is probably a function of the very limited number of whole fish which have been analyzed, both in this study and historically. Additionally, metals such as lead are known to partition into tissues other than muscle, and into mucus (Wiener, 1982).

For cadmium, the concentration in one whole sucker was estimated at 110 µg/Kg in 1993. This datum was qualified as limited due to low matrix spike recovery. This study analyzed one whole sucker from the upper lake and another from the lower lake segments for cadmium. The results were 280 and 420 µg/Kg respectively. Given the qualifiers on the previous data point, any significance to this comparison cannot be adequately established.

Mercury has previously been detected at a concentration of 82 µg/Kg in Long Lake largemouth bass fillet tissue (Johnson, 1994). Only one largemouth bass composite of five fillets was analyzed. This study detected mercury in largemouth bass fillet composites at higher concentrations. For the lower lake strata, composites ranged from 65.4 to 108.0 µg/Kg; while in the upper lake, concentrations ranged from 81.1 to 113.0 µg/Kg. The values detected in these composites appear to be analytically biased low, based on the QA review (Table 5).

The total detected concentrations of apparent Aroclors in both upper and lower Long Lake strata were compared to nearby historic concentrations from the 1999 and 1993. Johnson (2000) reports concentrations of apparent Aroclors in mountain whitefish and largescale suckers from the 7-Mile Bridge reach of the upper Spokane River. The 7-mile Bridge river reach is the closest upstream data to Long Lake and is about five river miles upstream from the upper end of Long Lake. From this study (Johnson, 2000), individual largescale sucker and mountain

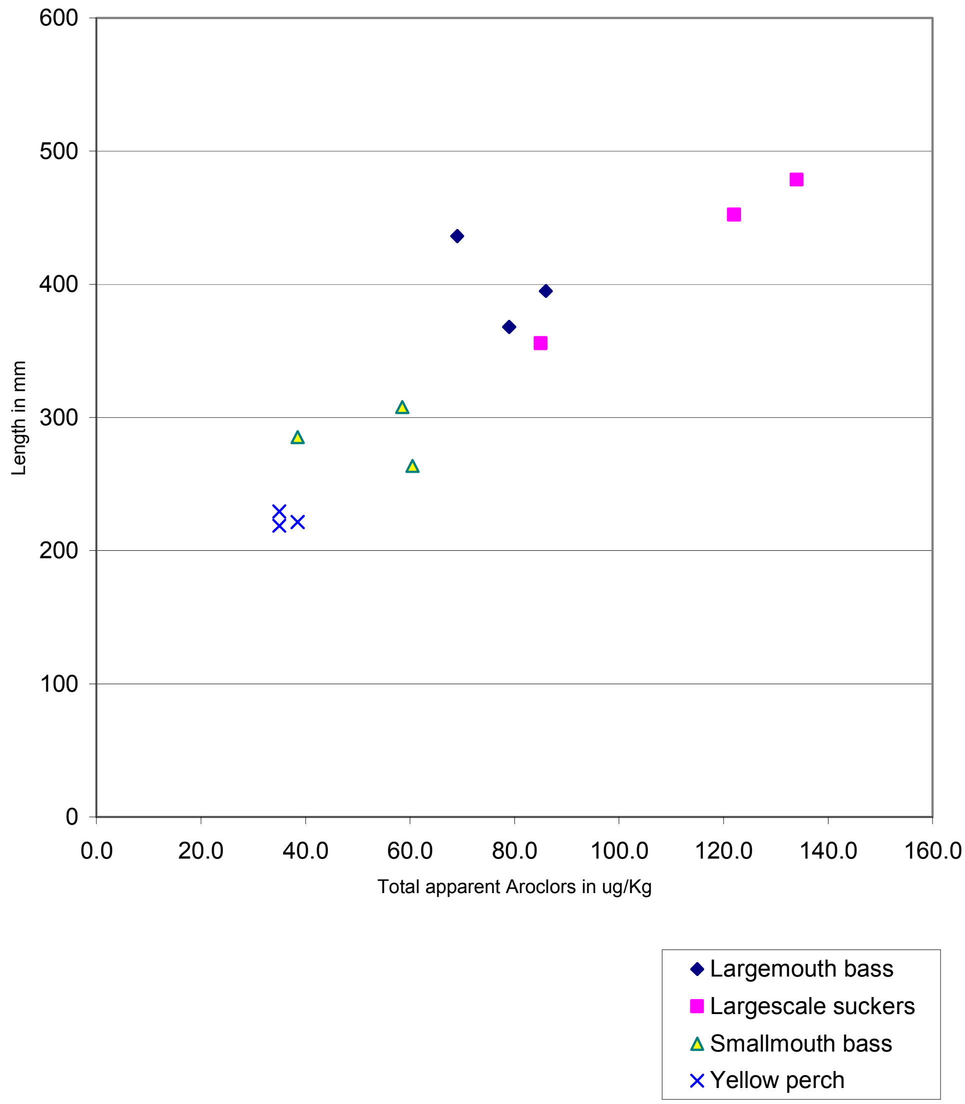

Figure 8. Total apparent Aroclor concentration by species compared to arithmetic mean fish length for lower Long Lake composite fish tissue samples.

whitefish fillets are arithmetically averaged for use in Table 10. The mean age of these fish was 8.2 years for largescale suckers and 4.0 years for mountain whitefish. The 1999 study analyzed five whole suckers. Age estimates for these fish ranged from 4+ to 8+ years with one fish having regenerated scales. These ages are generally consistent with the ages of fish in both upper and lower lake composite samples in this study. However, the age distribution in suckers does not appear to be normal. The distribution might fit a log-normal pattern, although more individual fish would be required to statistically test this hypothesis.

For fillets, minor differences in age are not suspected to change muscle burdens of PCBs significantly. This is because half-lives of PCBs in trout fillets have been estimated at <5 to 127 days (Niimi and Oliver, 1983). This is consistent with the poor relationships between age and total apparent Aroclors encountered (Figure 5). For whole fish, differences in age are probably significant when comparing results to previous studies. Niimi and Oliver (1983) found whole fish PCB half-lives to be >1000 days for whole trout. Coristine et al. (1996) found elimination rates from 0.004 to 0.012 µg/Kg/day in whole trout, but it is not known if the excreted congeners might be bioavailable for uptake by other fish.

In addition to probable variation in elimination rates interacting with fish age, other factors might also interact to vary PCB concentrations. These include issues such as dietary preferences of different species and age classes within each species, as well as issues such as home range and territoriality. In general, the largescale suckers were extremely difficult to determine sex. It is not known whether females might eliminate significant PCBs via eggs during spawning, although this is yet another interaction to potentially consider. Via the lipid fraction of gametes, female marine copepods eliminate PCBs about twice as fast as males (McManus et al, 1983), so this interaction might be significant.

Fillets and whole fish were generally similar to sizes from Johnson (2000). Insufficient numbers of fish are available to reliably test for differences. Variation in size is known to interact with metals concentrations in organs (Evans et al. 1993).

Johnson et al. (1994) report concentrations from samples in lower Long Lake. The sampling area was in the vicinity of the upstream end of the lower lake strata used in this report. Five mountain whitefish fillet were analyzed as a single composite. Their mean total length was 309 mm, and their mean weight was 242 grams. For the whole largescale sucker composite, their mean total length was 468.8 mm, and their mean weight was 966.8 grams. These weights and lengths are similar to those found in the current study. The most apparent difference is the mountain whitefish, the historical sample appears to have been comprised of larger fish. The Johnson et al. (1994) total apparent Aroclor concentrations in mountain whitefish and largescale suckers are shown on Figure 9.

In general, concentrations are lower in the current study by about a factor of 10 compared to both historic data points for mountain whitefish fillets. This may be partially due to changes in fish sizes, although the compositing appears to conceal any potentially relevant relationships between age, length, weight, and apparent Aroclor concentrations. Historic largescale sucker data are limited to three samples. The 1993 whole sucker concentrations appear higher than the maximum detected in this study. Sucker fillet concentrations between the 7-mile samples and

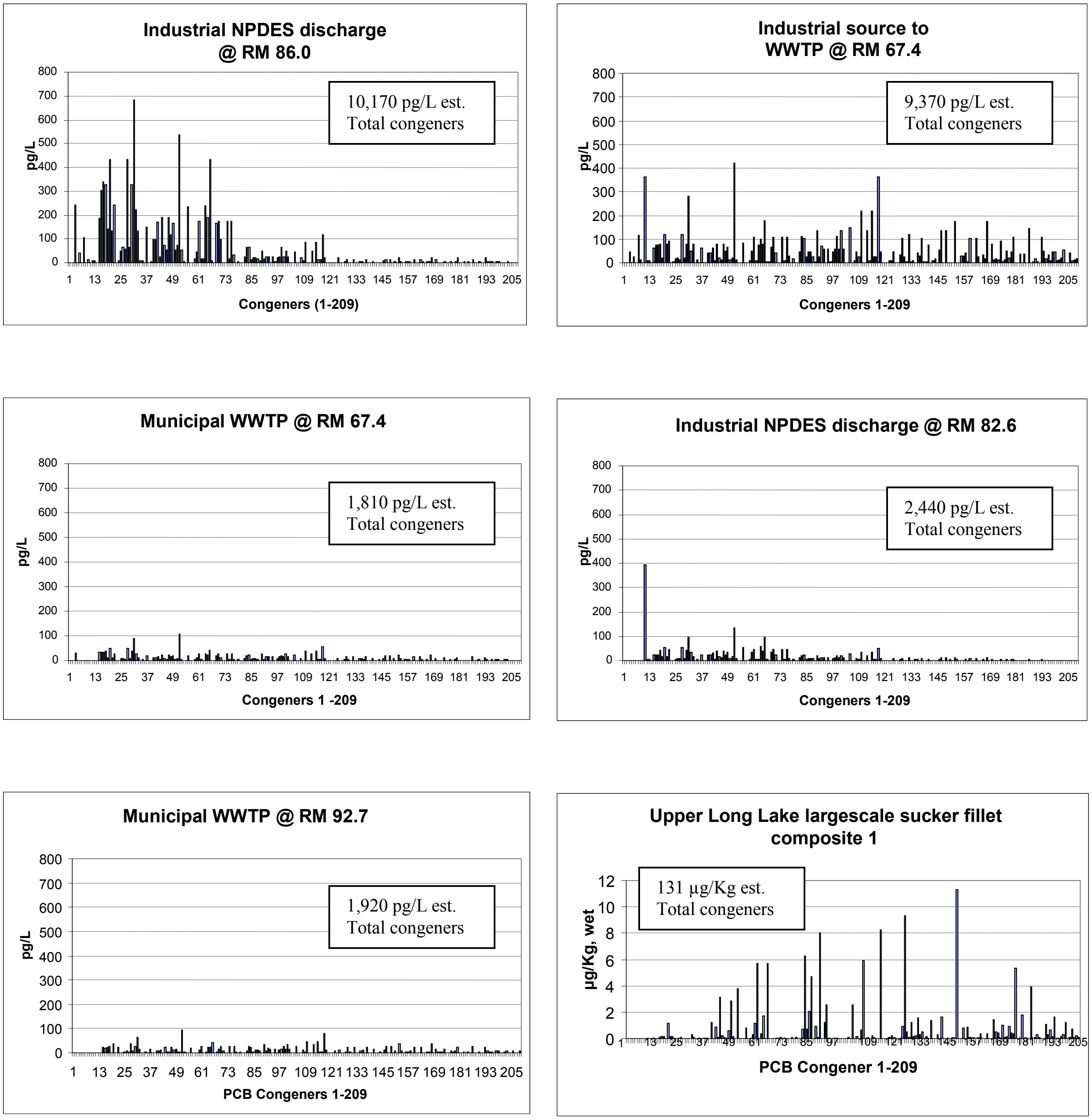

Figure 9. PCB congener distributions in effluent wastewater, and a typical composite fish fillet sample. Water results from Golding (2002).

the current study appear similar. For future data collections, largescale suckers greater than 500 mm should be used for trend monitoring, as these larger fish are the most conservative (Figures 6 and 8).

PCB congener results were compared between historic water samples and fish fillet composites. The fish fillet composite results shown in Figure 9 are typical. Figure 9 illustrates that fish congener patterns do not clearly match any known water discharge. This result is not unexpected, as chlorination patterns in PCB congeners have a very strong influence on their bioaccumulation rates and half-lives in the tissue (Evans et al. 1993; Coristine et al. 1996). Any future attempts at pattern matching should incorporate information on the octanol-water partitioning coefficients (K_{ow}) of the relevant congeners, and data on their relative metabolism and excretion rates. In general, more chlorinated congeners are more resistant to metabolism and excretion. Chlorine location on the congener also plays a significant role. Congener 153 is found in abundance within most of the fish fillet tissues. This congener could serve as a possible indicator compound for other more chlorinated compounds in future investigations.

Primary literature suggests that less than 20% of congener 153 would ever be excreted (Mathews and Anderson, 1975) because it is particularly resistant to metabolic action. This suggests that the more chlorinated congeners found in fish tissues during the current study are a product of both higher bioaccumulation rates and their metabolic stability.

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Conclusions

Insufficient numbers of historic tissue samples are available from within Long Lake proper to fully statistically evaluate changes in concentrations which may have occurred over the past decade. When visually comparing (Figure 10) historic results from upstream of Long Lake with the current data set, it appears that PCB concentrations in whole largescale suckers have declined slightly in Long Lake since 1999. Comparisons with whole suckers from Long Lake collected in 1993 also suggest slight declines.

As shown in Figure 3, the utility of dividing the lake into strata for Aroclor analysis varies by species. For largemouth bass, smallmouth bass, and yellow perch, the segregation was probably not warranted. Other non-geographic factors such as size, age, and lipid content probably account for the variation seen in these composites. For largescale suckers, the segregation of the lake into strata allows for illustration of the higher concentrations in the upper lake vs. the lower lake (Figure 3). This slight decrease in mean composite concentrations is consistent with both dilution and reduction in bioavailability.

The change in fish lipid-based concentrations supports a hypothesis of changing uptake modes or concentrations between the upper and lower lake segments. Robison (1998) found reductions in PCB bioavailability with increasing downstream distances from riverbank sources/inputs in Kentucky streams. The upper lake strata also had more variable whole sucker concentrations which may be a product of patchy sediment contamination relative to the lower lake. Composite sizes were higher in the upper lake, suggesting that variation in composite size is not responsible for the apparent concentration decline.

Upper Long Lake is believed to be receiving uncontaminated sediment from the Latah Creek drainage (SCCD, 2002). The various particulate sources to upper Long Lake may produce PCB gradients in sediments. In lower Long Lake, particulates may be more completely mixed, leading to more homogenous sediment PCB concentrations and, therefore, less variable fish tissue concentrations.

For metals, no geographic patterns are apparent as a result of separating the lake into strata. For zinc, cadmium, and mercury, concentrations also are below the derived NTR and/or human consumption screening criteria. For these metals, fish tissue concentrations suggest Long Lake is not impaired for these constituents. For lead, the absence of established screening or regulatory criteria may necessitate an evaluation by the Washington State Department of Health (DOH) in conjunction with organics data.

For PCB constituents, Long Lake continues to be impaired, as described by the Ecology 1998 303(d) listing. DOH is being provided these results to permit further evaluation of apparent Aroclor and congener data, to determine the potential impacts of this impairment on human health.

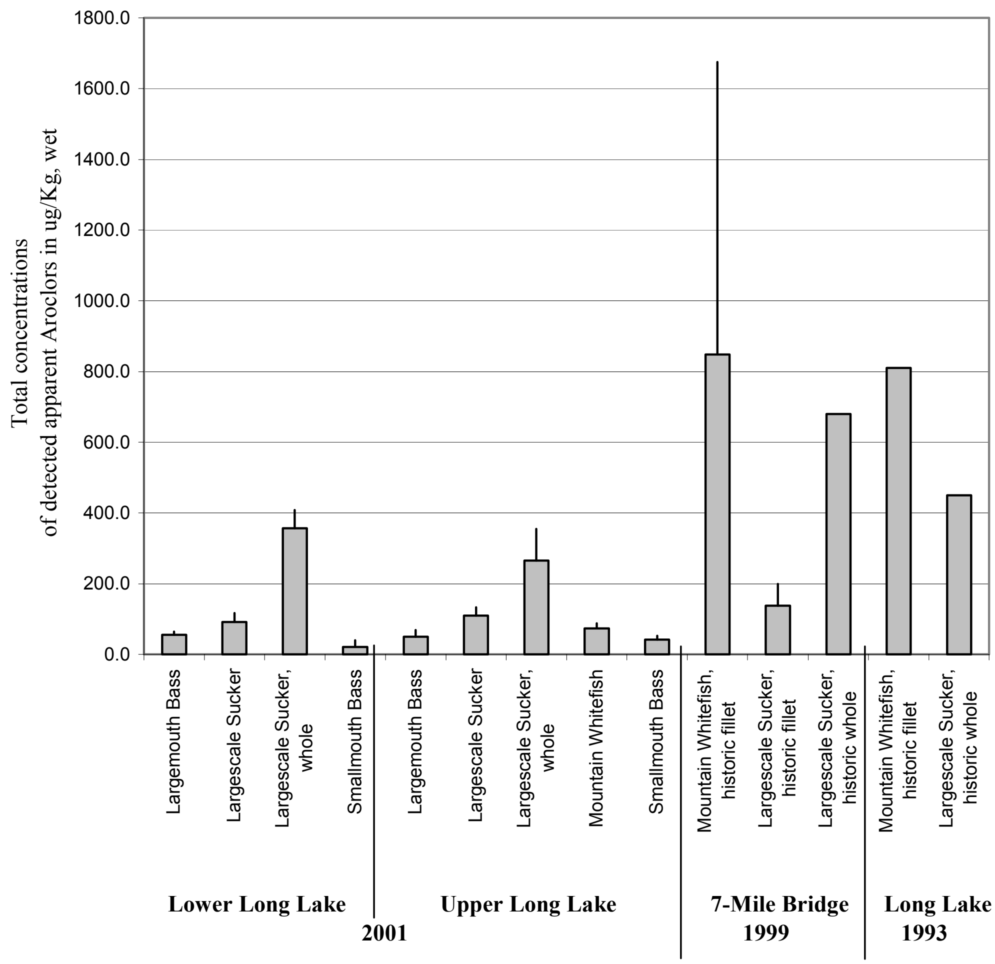

Figure 10. Comparison of current results with historic largescale sucker and mountain whitefish results from 7-Mile Bridge and Long Lake (Johnson et al. 1994; Johnson, 2000). Bars are standard errors.

Recommendations

For future investigations, the following recommendations are suggested:

- Investigate sediment congener characteristics and patterns to evaluate the role of sediment in observed fish concentrations.
- Continue to segregate Long Lake into multiple strata until the factors influencing apparent downstream declines in largescale sucker Aroclor concentrations are determined.
- Use suckers greater than 500 mm when conducting future trend monitoring studies, as this species and size is most conservative.
- Avoid spending extensive resources on metals tissue analyses for human consumption studies, as metals do not appear to be of concern.
- Avoid spending extensive resources on large or smallmouth bass PCB congener analysis, as these species are not conservative.
- Continue to list Long Lake as an impaired waterbody via the 303(d) listing process.
- Investigate the utility of other parameters that might influence PCB concentrations in fish, such as dissolved organic carbon, age, sex, and size. This investigation should use individual fillet samples.

PCB concentrations and metals were significantly higher in whole largescale suckers. If whole sucker consumption occurs, future studies should evaluate whole suckers in more detail, including additional fish and replicates, as well as possibly liver, bile, and/or adipose tissue sampling.

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